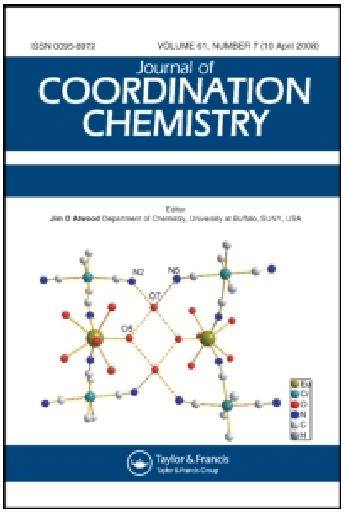

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Lead(II) and cadmium(II) complexes of 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-*p,p'*-disulfonate with high thermal stability

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Lead(II) and cadmium(II) complexes of 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-*p*,*p*′-disulfonate with high thermal stability

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Pb(II) and Cd(II) complexes, $\{[Pb_2(PDTS)_2(CH_3OH)((H_2O)_2]\cdot H_2O\}_n$ (**1**) and $[Cd(PDTS)(H_2O)_4]\cdot 2H_2O$ (**2**) ($PDTS^{2-}$ = 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-*p*,*p*′-disulfonate), are synthesized and characterized by elemental analysis, infrared (IR), ^{1}H-NMR spectroscopy, and thermal analysis. The single-crystal structure of **1** shows that the complex forms a 2-D polymeric network containing two types of Pb^{2+} with coordination number eight (PbN_2O_6). Both possess hemidirected coordination geometries. The single-crystal structure of **2** shows distorted octahedral geometry for cadmium(II), CdN_2O_4. These compounds are the first complexes of "$PDTS^{2-}$". The supramolecular features in these complexes are guided/controlled by hydrogen bonding and noncovalent intermolecular interactions.

Keywords: Lead(II); Cadmium(II); 1,2,4-Triazine; Hydrogen bonding

1. Introduction

The self-assembly approach to construction of supramolecular or extended frameworks based on coordination complexes is a major current research area in inorganic chemistry [1]. In the vast majority of such compounds, the molecular building blocks are held together by strong metal–ligand–metal bonding interactions or weaker forces such as hydrogen bonding between ligands coordinated to different metal units. The use of bridging ligands for controlled self-assembly of 1-, 2-, or 3-D metallosupramolecular species have been the subject of enormous study in recent years [2]. Generally, extended high dimensional networks can be obtained by assembly of lower dimensional coordination polymers *via* noncovalent intermolecular forces such as hydrogen-bonding and π–π stacking interactions [3–5]; 1,2,4-triazine ligands are excellent candidates for the construction of extended supramolecular architectures [6]. In this article, we report

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the synthesis of the first Pb(II) and Cd(II) complexes with 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-*p*,*p*′-disulfonate($PDTS^{2-}$) and characterization by X-ray structural analysis.

2. Experimental

2.1. *Physical measurements*

Infrared (IR) spectra were recorded as KBr pellets using Perkin Elmer 597 and Nicolet 510P spectrophotometers. Elemental analyses were performed with a Heraeus CHN–O–rapid analyzer. Melting points were measured using an Electrothermal 9100 apparatus and are uncorrected. ^{1}H-NMR spectra were recorded on a BRUKER DRX-500 AVANCE spectrometer at 500 MHz using dimethylsulfoxide (DMSO)-d_6.

2.2. *Preparation of* $\{[Pb_2(PDTS)_2(CH_3OH)(H_2O)_2]\cdot H_2O\}_n$ *(1)*

3-(2-Pyridyl)-5,6-diphenyl-1,2,4-triazine-*p*,*p*′-disulfonic acid, disodium salt hydrate "Na_2PDTS", (0.514 g, 1 mmol) was placed in one of the arms of a branched tube and $Pb(OOCCH_3)_2\cdot 3H_2O$ (0.379 g, 1 mmol) was placed in the other arm [7]. Methanol and water (ratio 50 : 50) were carefully added to fill both arms, the tube sealed and the ligand-containing arm immersed in a bath at 60°C whereas, the other was maintained at ambient temperature. After 10 days, crystals deposited in the cooler arm were filtered, washed with ether, and dried in air. Anal. Calcd for $C_{41}H_{33}N_8O_{16}Pb_2S_4$: C: 34.25%, H: 2.30%, and N: 7.80%. Found: C: 34.12%, H: 2.20%, and N: 8.10%. IR (KBr) ν 3150–3450(m), 3024(w), 2996(w), 1580(s), 1569(s), 1423(s), 1285(m), 1124(w), 780(w), 630(s) cm^{-1}. ^{1}H-NMR (500 MHz, DMSO-d_6) δ (ppm) 8.00 (m, 4H, 2 benzyl), 8.30 (m, 4H, 2 benzyl), 7.85 (dd, 1H, pyridyl), 8.35 (dd, 1H, pyridyl), 8.55 (d, 1H, pyridyl) and 8.85 (d, 1H, pyridyl).

2.3. *Preparation of* $[Cd(PDTS)(H_2O)_4]\cdot 2H_2O_2$ *(2)*

"Na_2PDTS" (0.514 g, 1 mmol) was placed in one of the arms of a branched tube and $Cd(OOCCH_3)_2\cdot 3H_2O$ (0.308 g, 1 mmol) was placed in the other arm. Methanol and water (ratio 50 : 50) were carefully added to fill both arms, the tube sealed and the ligand-containing arm immersed in a bath at 60°C while the other was maintained at ambient temperature. After 10 days, crystals that deposited in the cooler arm were filtered, washed with ether, and dried in air. Anal. Calcd for $C_{40}H_{44}Cd_2N_8O_{22}S_4$: C: 35.77%, H: 3.28%, and N: 8.35%. Found: C: 35.40%, H: 3.10%, and N: 8.30%. IR (KBr) ν 3150–3450(m), 3062(w), 1554(m), 1500(s), 11480(s), 1445(s), 1270(m), 1026(m), 850(w), 622(m) cm^{-1}. ^{1}H-NMR (500 MHz, DMSO-d_6) δ (ppm) 8.95 (m, 4H, 2 benzyl), 8.40 (m, 4H, 2 benzyl), 7.75 (dd, 1H, pyridyl), 8.20 (dd, 1H, pyridyl), 8.60 (d, 1H, pyridyl), and 8.80 (d, 1H, pyridyl).

Table 1. Crystal data of **1** and **2**.

Identification code	**1**	**2**
Empirical formula	$C_{41}H_{33}N_8O_{16}Pb_2S_4$	$C_{20}H_{22}CdN_4O_{11}S_2$
Formula weight	1436.37	670.94
Temperature (K)	100.0(1)	100.0(1)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$P2(1)/n$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	16.1583(3)	10.0576(2)
b	12.0884(2)	10.6272(3)
c	23.3780(4)	12.6382(3)
α	90.00	71.888(1)
β	100.113(1)	75.543(1)
γ	90.00	88.754(1)
Volume (Å^3), Z	4495.43(14), 4	1240.94(5), 2
Calculated density (g cm^{-3})	2.122	1.796
Absorption coefficient (mm^{-1})	7.750	1.116
$F(000)$	2764	676
Crystal size (mm^3)	$0.45 \times 0.30 \times 0.20$	$0.34 \times 0.26 \times 0.13$
θ range for data collection (°)	1.42–35.00	1.75–30.00
Index ranges	$-26 \le h \le 25$; $-19 \le k \le 19$; $-37 \le l \le 37$	$-14 \le h \le 14$; $-14 \le k \le 14$; $-17 \le l \le 17$
Reflections collected	111,130	36,275
Independent reflections	19,712 [R(int) = 0.0401]	7188 [R(int) = 0.0266]
Completeness to $\theta = 25.02$ (%)	99.6	99.3
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	19,712/0/640	7188/0/413
Goodness-of-fit on F^2	1.070	1.048
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0466$, $wR_2 = 0.1061$	$R_1 = 0.0303$, $wR_2 = 0.0796$
R indices (all data)	$R_1 = 0.0621$, $wR_2 = 0.1131$	$R_1 = 0.0433$, $wR_2 = 0.0828$
Largest difference peak and hole (e Å^{-3})	5.574 and −4.845	0.899 and −1.256

2.4. *Crystallography*

Crystallographic data were collected at 100 K with an Oxford Cyrosystem Cobra low temperature attachment. The data were collected using a Bruker Apex2 CCD diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at a detector distance of 5 cm with APEX2 software [8]. The collected data were reduced using SAINT [8] and empirical absorption corrections were performed using SADABS [8]. The structures were solved using direct methods and refined using least-squares from the SHELXTL software package [9]. Materials for publication were prepared using SHELXTL and ORTEP III [9, 10]. The oxygens attached to S2 and Cd1 in **2** were disordered over two positions with refined site-occupancies of 0.732 (4): 0.268 (4) and 0.600 (10): 0.400 (10), respectively. The large difference residues (highest peak, 0.71 Å from Pb1 and deepest hole, 0.59 Å from Pb2) were near heavy atoms. Initially, rigid bond (DELU and SAME) and similarity (SIMU) restraints were used in the refinement using SHEXTL. In the final refinement, all these restraints were removed. Hydrogens bound to oxygen were located from difference Fourier maps and refined as riding on the parent oxygen with $U_{iso}(H) = 1.5U_{eq}(O)$. The remaining hydrogens were positioned geometrically and refined using a riding model, with C–H = 0.93–0.93 Å and $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$.

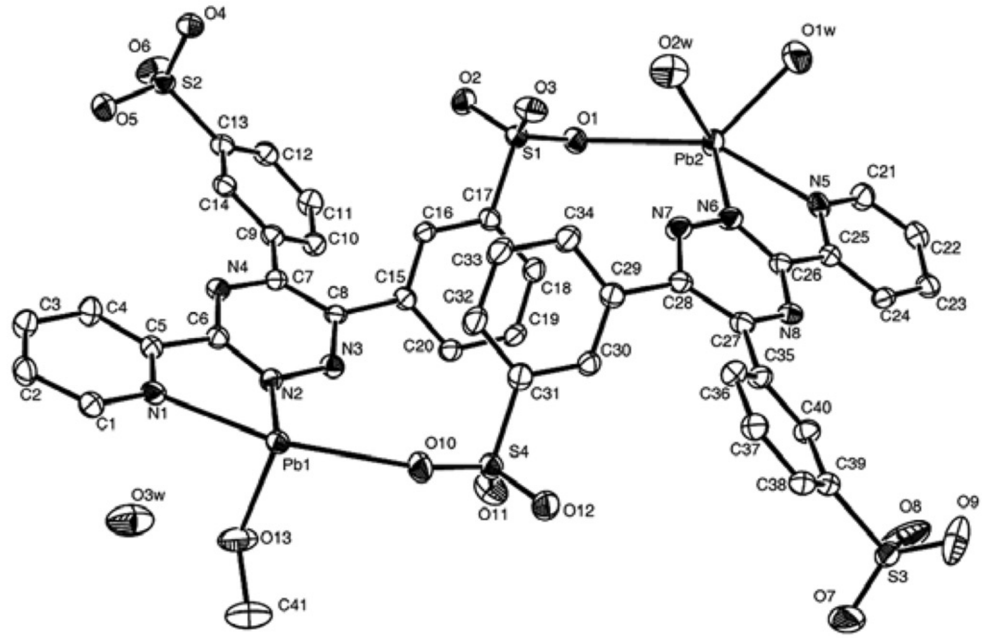

Figure 1. ORTEP diagram of the asymmetric $\{[Pb_2(PDTS)_2(CH_3OH)((H_2O)_2] \cdot H_2O\}_n$ unit. Only non-hydrogen atoms are shown.

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

1				**2**	
Pb1–O13	2.388(4)	Pb2–O1W	2.485(4)	Cd1–O8A	2.153(8)
Pb1–N1	2.546(4)	Pb2–N5	2.517(4)	Cd1–O7A	2.277(10)
Pb1–N2	2.565(3)	Pb2–N6	2.575(3)	Cd1–O10A	2.309(3)
Pb1–O10	2.702(3)	Pb2–O8$^{\mathrm{i}}$	2.592(6)	Cd1–O9A	2.318(16)
Pb1–O4$^{\mathrm{i}}$	2.749(3)	Pb2–O12$^{\mathrm{ii}}$	2.617(4)	Cd1–N1	2.3486(17)
Pb1–O2	2.761(4)	Pb2–O1	2.838(4)	Cd1–N2	2.3779(12)
Pb1–O1	2.846(4)	Pb2–O2W	2.985(5)	O8A–Cd1–O7A	92.7(4)
Pb1–O5$^{\mathrm{i}}$	3.057(4)	Pb2–O10	3.028(4)	O8A–Cd1–O10A	110.1(4)
O13–Pb1–N1	81.61(13)	O13–Pb1–N2	80.71(13)	O8A–Cd1–O9A	96.9(4)
O13–Pb1–O1	85.19(13)	O13–Pb1–O2	83.01(15)	O7A–Cd1–O10A	92.0(2)
O2–Pb1–N1	80.24(11)	O1–Pb1–N1	130.60(11)	O7A–Cd1–O9A	167.7(3)
N1–Pb1–N2	64.15(11)	O13–Pb1–O10	94.64(14)	O10A–Cd1–O9A	77.6(3)
N1–Pb1–O10	153.92(11)	N2–Pb1–O10	89.78(11)	O8A–Cd1–N1	87.9(4)
O13–Pb1–O4$^{\mathrm{i}}$	142.29(12)	N1–Pb1–O4$^{\mathrm{i}}$	74.53(10)	O7A–Cd1–N1	87.4(2)
N2–Pb1–O4$^{\mathrm{i}}$	62.77(10)	O10–Pb1–O4$^{\mathrm{i}}$	94.31(10)	O10A–Cd1–N1	162.0(2)
O1W–Pb2–N5	76.42(14)	O1W–Pb2–N6	97.16(14)	O9A–Cd1–N1	100.5(4)
N5–Pb2–N6	64.04(11)	O1W–Pb2–O8$^{\mathrm{i}}$	142.64(17)	O8A–Cd1–N2	157.8(4)
O1W–Pb2–O2W	70.88(14)	O1W–Pb2–O12$^{\mathrm{ii}}$	77.70(16)	O7A–Cd1–N2	87.3(3)
O1–Pb2–N6	92.91(11)	O1–Pb2–N5	149.13(11)	O10A–Cd1–N2	92.1(2)
N5–Pb2–O8$^{\mathrm{i}}$	75.66(15)	N6–Pb2–O8$^{\mathrm{i}}$	92.45(15)	O9A–Cd1–N2	86.7(4)
O1W–Pb2–O12$^{\mathrm{ii}}$	77.70(16)	N5–Pb2–O12$^{\mathrm{ii}}$	79.45(11)	N1–Cd1–N2	69.92(6)
N6–Pb2–O12$^{\mathrm{ii}}$	143.18(11)	O8$^{\mathrm{i}}$–Pb2–O12$^{\mathrm{ii}}$	73.19(13)		

$^{\mathrm{i}}3/2-x,\ -1/2+y,\ 1/2-z$; $^{\mathrm{ii}}x,\ 1+y,\ z$.

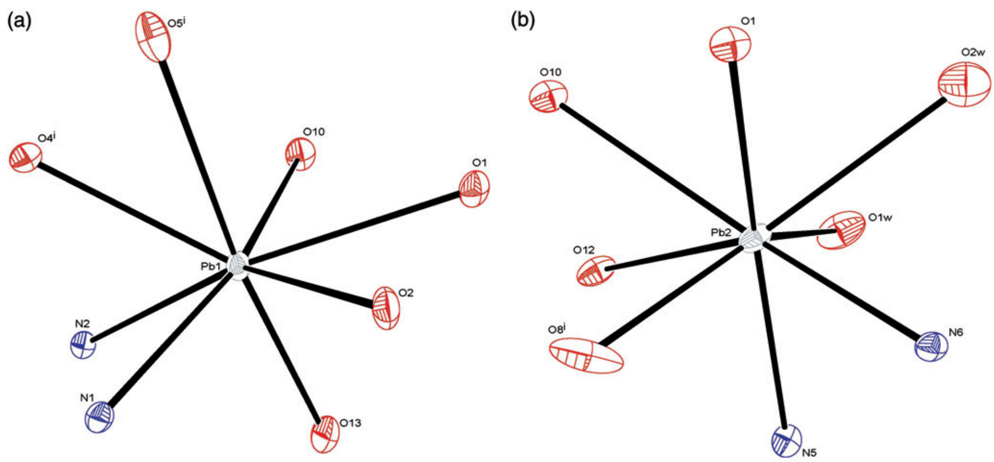

Figure 2. Schematic representation of two different Pb(II) environments.

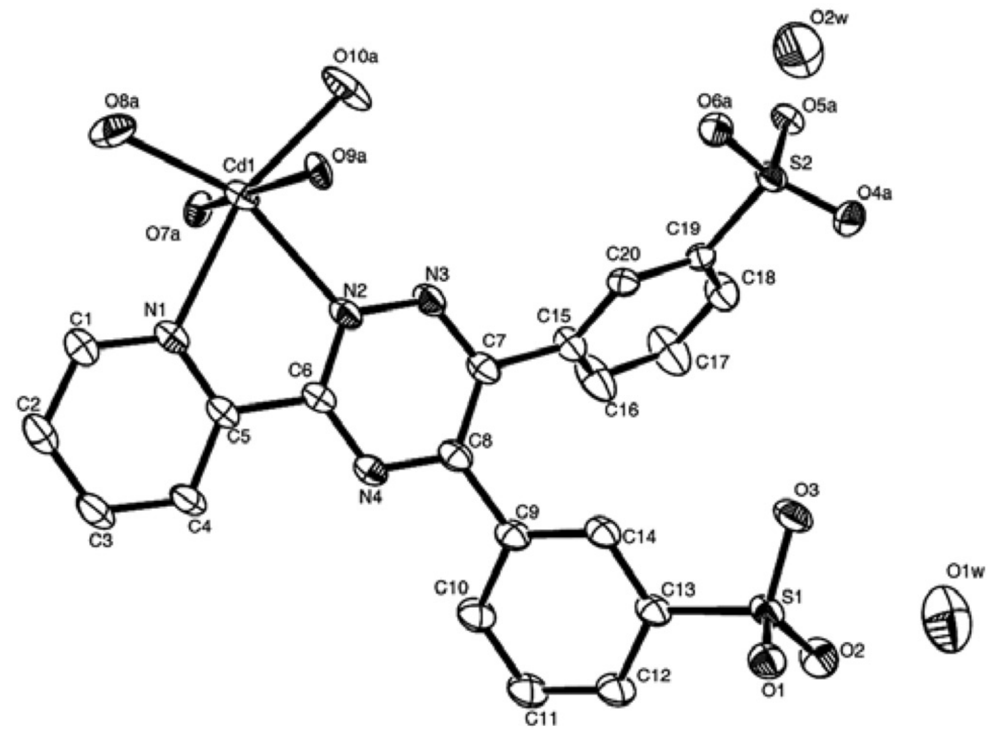

Figure 3. ORTEP diagram of $[Cd(PDTS)(H_2O)_4]\cdot 2H_2O$, **2**.
Only major disorder component was shown.

3. Results and discussion

Reaction between lead(II) acetate and cadmium(II) acetate with 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-*p*,*p*′-disulfonic acid, disodium salt hydrate in methanol provided

Table 3. Hydrogen bonding and directional intermolecular interactions in **1** and **2**.

B–H···A	H···A (Å)	B···A (Å)	B–H···A (°)
1			
O1W–H1WB···O2W($2-x, 4-y, -z$)	2.0400	2.8107(7)	151.00
O3W–H1W3···O13	1.7800	2.608(7)	166.00
O3W–H2W3···O11($1-x, 2-y, -z$)	1.9700	2.807(6)	166.00
C1–H1A···O2($x, -1+y, z$)	2.4600	3.211(6)	137.00
C2–H2A···O4($x, -1+y, z$)	2.4900	3.411(6)	171.00
C18–H18A···O7($3/2-x, 1/2+y, -1/2-z$)	2.3700	3.138(6)	139.00
C21–H21A···O12($x, 1+y, z$)	2.4000	3.093(6)	131.00
C22–H22A···O7($3/2-x, 1/2+y, -1/2-z$)	2.2400	3.160(7)	172.00
C36–H36A···O3($2-x, 3-y, -z$)	2.5900	3.088(6)	114.00
2			
C1–H1A···O8A	2.5900	3.182(12)	122.00
C1–H1A···O2W($1-x, 1-y, 1-z$)	2.2800	3.111(8)	149.00
C2–H2A···O4A($1+x, 1+y, -1+z$)	2.5100	3.375(4)	155.00
C4–H4A···O1($-x, 1-y, -z$)	2.5800	3.368(3)	143.00
O9A–H209···O3($-x, 1-y, 1-z$)	1.9000	2.693(1)	154.00
O7A–H207···O1($1+x, y, z$)	1.8000	2.734(11)	164.00
O8A–H208···O2($1+x, 1+y, z$)	1.9100	2.725(12)	161.00
O10A–H1O1···O3($-x, 1-y, 1-z$)	2.4000	2.927(6)	120.00
O10A–H2O1···N3($1-x, 1-y, 1-z$)	2.2100	2.970(5)	145.00
O1W–H1W1···O2($-x, 1-y, 1-z$)	2.0500	2.668(6)	129.00
O1W–H2W1···O2($1+x, 1+y, z$)	2.2100	2.889(6)	137.00

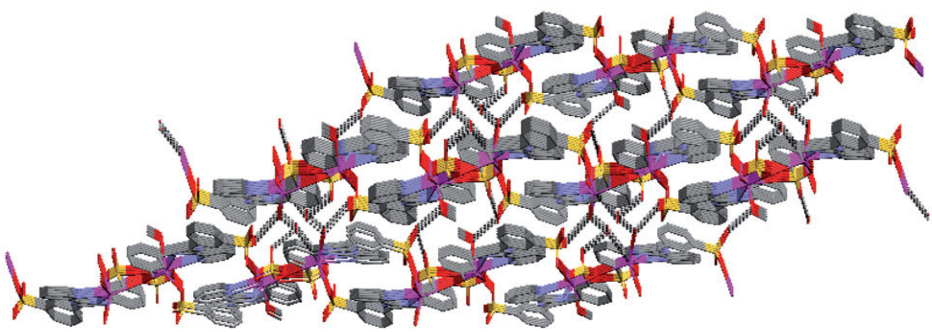

Figure 4. Packing of **1** down the *b*-axis, formation of 3-D networks from 2-D channels *via* hydrogen bonding.

the crystalline materials $\{[Pb_2(PDTS)_2(CH_3OH)((H_2O)_2]\cdot H_2O\}_n$ (**1**) and $[Cd(PDTS)(H_2O)_4]\cdot 2H_2O$ (**2**), respectively. The IR spectrum of these compounds show absorption bands resulting from skeletal vibrations of aromatic rings from 1400 to 1580 cm^{-1}. Absorptions in the region 1000–1285 cm^{-1} are typical for the sulfonate group. Strong absorption bands at 630 and 622 cm^{-1} for **1** and **2**, respectively, are due to $\nu_{S\text{–}O}$ [11]. The ^{1}H-NMR spectra (DMSO-d_6) of **1** and **2** display absorption bands at 6.85–8.87 ppm assigned to the aromatic protons of "$PDTS^{2-}$" ligand; bands at 7.66, 8.20, 8.55, and 8.87 ppm are assigned to pyridyl protons of "$PDTS^{2-}$". Thermogravimetric analyses (TGA) for these complexes performed on polycrystalline samples under nitrogen follow a two-step mechanism. The first stage from 75 to 110°C corresponds to the loss of coordinated and uncoordinated water and methanol (in **1**, Calcd 5.98%, found 6.5% and in **2** Calcd 14.00%, found 16.50%). The next loss at 502°C corresponds

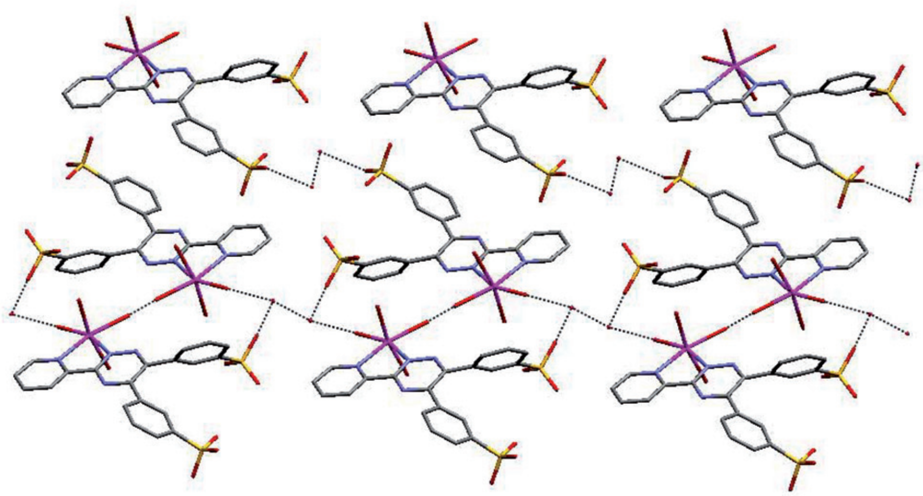

Figure 5. Packing of **2** down the *b*-axis, formation of 3-D networks *via* hydrogen bonding. Irrelevant hydrogen bonds omitted for clarity.

to the beginning of decomposition (exothermic) and showed that the compounds have good thermal stability. The solid residues formed at the end of the decomposition at 600°C are suggested to be PbO and CdO.

According to the single crystal structure analysis (table 1), **1** crystallized in the monoclinic $P21/n$ space group and forms a 2-D coordination polymer. The ORTEP diagram of the asymmetric $\{[Pb_2(PDTS)_2(CH_3OH)((H_2O)_2)] \cdot H_2O\}_n$ unit is shown in figure 1 and selected bond lengths and angles are given in table 2. In the structure of **1**, there are two different Pb's (figure 2). Pb1 is eight coordinate, chelated by two nitrogens of "$PDTS^{2-}$", one oxygen of methanol, and four oxygens of the three different sulfunate groups of "$PDTS^{2-}$". The coordination number in Pb2 is also eight, chelated by two nitrogens of "$PDTS^{2-}$", two oxygens of water, and four oxygens of the three different sulfonates of "$PDTS^{2-}$". In **1**, there is an uncoordinated water molecule close to Pb1 and the Pb $\cdots$ Pb distance through sulfonate bridges are 4.571 Å. Three bond lengths Pb1–O5^{i} 3.057(4), Pb2–O2W 2.985(4), and Pb2–O10 3.028(4) Å are significantly longer than the sum of the ionic radii, but shorter than the sum of the van der Waals radii (3.54 Å) [12]. These longer Pb–O bonds were largely overlooked in previous articles [13]. These long distances are interpreted as a consequence of the position of the oxygen atoms close to the sterically active Pb(II) lone pair [14–17]. The leads are bridged by "$PDTS^{2-}$" forming a 2-D coordination polymer.

Compound **2** crystallized in triclinic $P\bar{1}$ space group (table 1) with only one Cd(II) in the asymmetric unit; **2** is a mononuclear structure. As shown in figure 3, each Cd(II) is coordinated by two nitrogens from a chelating "$PDTS^{2-}$" (Cd–N = 2.3486(17) and 2.3779(12) Å) and four oxygens from four disordered waters (Cd–O = 2.153(8)–2.318(16) Å) in a distorted octahedral geometry. The CdN_2O_4 coordination environment is normal and comparable to that reported by Xie *et al*. [18]. Selected bond and angle parameters of **2** are displayed in table 2.

A search was made for weak directional intermolecular interactions in the crystal structures **1** and **2**. Existence of coordinated and uncoordinated water and oxygens of sulfonates help to form supramolecular networks *via* strong hydrogen bonding. In **1**, there are strong OH···O hydrogen bonds and CH···O directed intermolecular interactions (table 3) [19]. The formation of 3-D network is governed by these intermolecular interactions (figure 4). In **2**, the deviation from ideal octahedral geometry is probably due to the large steric hindrance of "$PDTS^{2-}$," and intramolecular hydrogen bonds between "$PDTS^{2-}$" ligand and water. An inspection of **2** for weak directional intermolecular interactions shows that there are interesting CH···O and OH···O hydrogen bonds (figure 5) [20]. Thus, the supramolecular features in these complexes are guided/controlled by hydrogen bonding and non-covalent intermolecular interactions.

Supplementary material

Crystallographic data for the structures reported in the article have been deposited in the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-749017 for **1** and CCDC-749016 for **2**. Copies of this information can be obtained for free from The Director, CCDC, 12 Union Road, Cambridge, CB2 IEZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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