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Syntheses and structures of two one-dimensional double-stranded lead polymers of dicyanamide with unusual coordination mode

Yu-Jun Shi [a,1], Li-Hong Li [a], Yi-Zhi Li [a], Xue-Tai Chen [a,*], Ziling Xue [b], Xiao-Zeng You [a,*]

[a] *State Key Laboratory of Coordination Chemistry, Coordination Chemistry Institute, Nanjing University, Nanjing 210093, People's Republic of China*
[b] *Department of Chemistry, The University of Tennessee, Knoxville, TN 37996-1600, USA*

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Abstract

The combined use of lead nitrate, sodium dicyanamide (Nadca) and 1,10-phenanthroline or 2,2-bipyridine has led to the preparation of two one-dimensional coordination polymers Pb(dca)(NO_3)(1,10-phen) (**1**) and Pb(dca)$_2$(2,2′-bipy)(H_2O) (**2**). X-ray crystallography shows that both **1** and **2** have one-dimensional chains with dca in a novel 1,1,5-μ_3-coordination mode, which results in one-dimensional double-stranded chains. This coordination mode has not been observed in the reported complexes of dca, further indicating the versatility of dca in bonding to metal ions.

Keywords: Lead; Dicyanamide; Coordination polymer; Coordination mode; Double-stranded; One-dimensional

1. Introduction

Coordination polymers constitute one of the most important class of organic–inorganic hybrid materials which have attracted great research interests [1,2]. Our interest in this field has focused on the construction of novel PbX$_2$-based organic–inorganic hybrids using lead(II) ion as the building block due to the belief that its versatile coordination number and varied coordination geometries would give rise to novel coordination polymeric networks [3]. PbX$_2$-based coordination polymers with nitrogen-containing Lewis bases such as 2,2′-bipyridine, 4,4-bipyridine have been studied and found to exhibit one- or two-dimensional structures [3], where PbX$_2$ chain or layer perseveres. Furthermore, coordination polymers based on lead pseudohalide Pb(SCN)$_2$ have also been prepared and structurally characterized [3a,4].

The coordination chemistry of the pseudohalide ligand dicyanamide(dca) has been explored for many years. Köhler [5], Hvastijova [6] and their co-workers have published a series of papers describing transition metal complexes with dca, but without X-ray single crystal structures. Transition metal compounds with dca have recently received renewed interest initiated by the novel magnetic properties of binary complexes M(dca)$_2$ (M = Cr, Mn, Fe, Co, Ni, Cu) [7]. Many coordination polymers of transition metals of dca with nitrogen-containing coligands such as 2,2′-bipyridine, 4,4-bipridine and pyrazine have subsequently appeared in the literatures [8]. However, the coordination chemistry of main group elements of dca is scarcely reported [9–11,15–17]. Pb(dca)$_2$ was prepared in 1922, but its structural characterization was not given [9]. More recently, we [16] and Schnick et al. [17] have independently determined the crystal structure of Pb(dca)$_2$, which is isotypic with Ba(dca)$_2$ [17]. From the viewpoint of the crystal engineering of supramolecular coordination network, it is the first novel (4,5,9)-connected 3D polymeric network [16].

Five coordination modes of **I**–**V** of dca have been substantiated by X-ray crystallography (Scheme 1). The most common coordination mode is 1,5-μ_2 bridging, **II**,

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* Corresponding authors. Fax: +86-25-331-4502.
E-mail address: xtchen@netra.nju.edu.cn (X.-T. Chen).

[1] Permanent address: Department of Chemistry, Nantong Teacher's College, Nantong 226007 P.R. China

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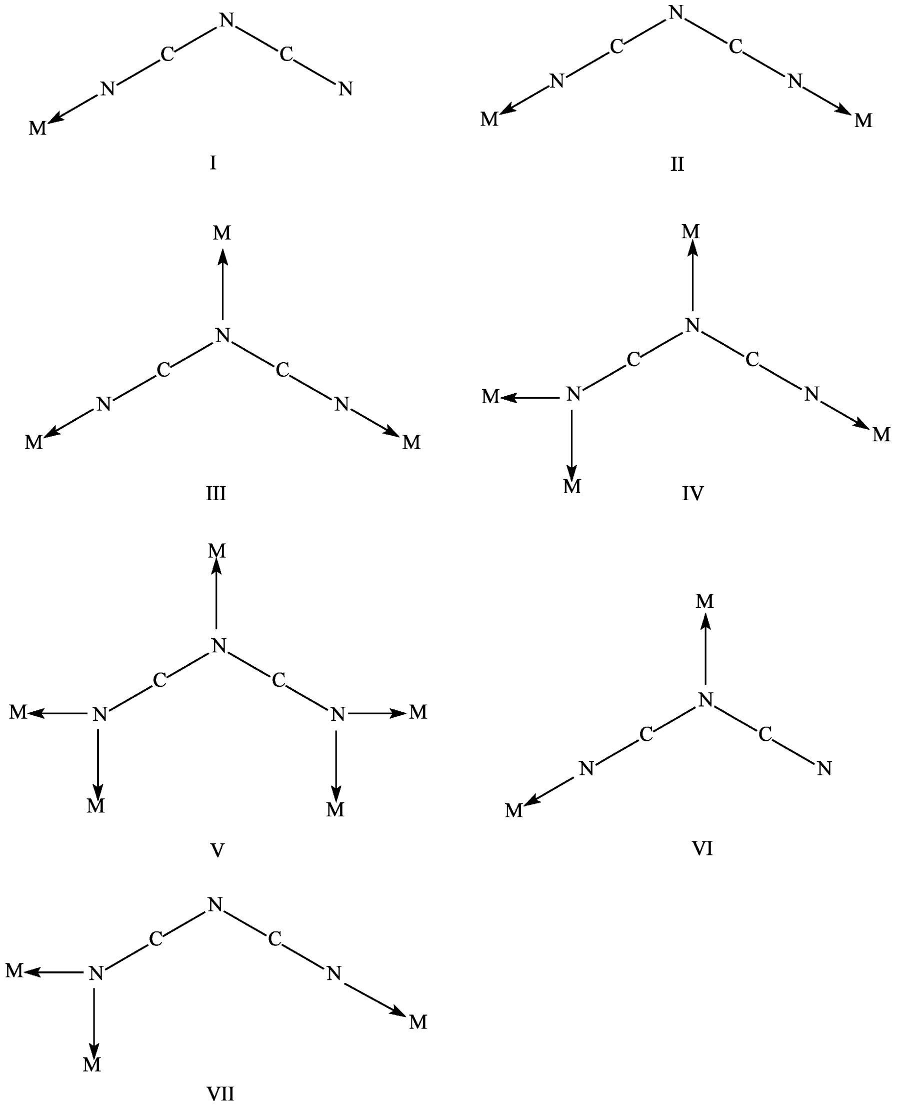

Scheme 1.

which has been found in many transition metal compounds and some main group metal compounds. The terminal coordination mode **I** occurs in some mononuclear complexes for example [Cu(1,10-phen)$_2$(dca)$_2$] [12], [Cu^{II}(1,10-phen)$_2${dca}$_2$][C(CN)$_3$] [13] and Mn(NITpPy)$_2$(dca)$_2$(H$_2$O)$_2$ [14]. The three-coordinate mode **III** is observed in the rutile-like material M(dca)$_2$, M = Cr, Mn, Fe, Co, Ni. The unusual coordination mode **IV** is observed in (CH$_3$)$_2$Tl(dca) [15] and Pb(dca)$_2$ [16,17]. Five-connecting coordination mode **V** was newly found in Pb(dca)$_2$ 16[17]. The possible two-connecting mode **VI** has yet to be confirmed, although it is postulated for one isomer of [Cu^{II}(dca)$_2$(imidazole)] [6b].

Following our research on PbX$_2$-based organic-inorganic hybrids [3h–j,16], we combined dca and 1,10-phenanthroline or 2,2-bipyridine with the expectation that novel polymeric structure and even new coordination mode of dca would be found by extending from halide to pseudohalide, taking advantage of the versatibility of both lead and dca in coordination chemistry. Here we report two one-dimensional coordination polymers Pb(dca)(NO$_3$)(1,10-phen) (**1**) and Pb(dca)$_2$(2,2′-bipy)(H$_2$O) (**2**) displaying a new three-connecting coordination mode **VII** which has not been observed before.

2. Experimental

All chemicals were used as received without further purification. Elemental analyses were carried out on a Perkin–Elmer 240C elemental analyzer. IR spectra were recorded on a Perkin–Elmer 1600 FT-IR spectrometer as KBr pellets in the 4000 to ~400 cm^{-1} range.

2.1. Synthesis

2.1.1. Pb(dca)(NO_3)(1,10-phen) (1)

A 5-ml ethanol solution containing 0.089 g (1.0 mmol) Nadca and 0.090 g (0.5 mmol) 1,10-phen was added dropwise to 5 ml aqueous solution of $Pb(NO_3)_2$ (0.17 g, 0.5 mmol) without stirring. The resulting mixture was left at room temperature (r.t.), and single crystals suitable for X-ray single-crystal diffraction appeared after several weeks. Yield 0.19 g (72% based on $Pb(NO_3)_2$). *Anal.* Found: C, 32.83; H, 1.57; N, 16.19. Calc. for $C_{14}H_8N_6O_3Pb$: C, 32.62; H, 1.56; N, 16.30%. IR (KBr pellets, cm^{-1}): 513(s), 551 (w), 639(m), 722(vs), 845(s), 912 (w), 1032(s), 1099(s), 1147(s), 1282(vs), 1325 (m), 1357(m), 1383(m), 1428(m), 1514 (s), 1578(w), 1588 (w), 1620 (m), 1730 (w), 2131 (vs), 2210(s), 2272 (s).

2.1.2. $Pb(dca)_2$(2,2′-bipy)(H_2O) (2)

A similar procedure to **1** using 0.089 g (1.0 mmol) Nadca, 0.16 g (1.0 mmol) 2,2′-bipy and (0.17 g, 0. 5 mmol) $Pb(NO_3)_2$ gave **2**. Yield 0.18 g (69% based on $Pb(NO_3)_2$). *Anal.* Found: C, 32.80; H, 1.84; N, 22.02. Calc. for $C_{14}H_{10}N_8OPb$: C, 32.75; H, 1.96; N, 21.81%. IR (KBr pellets, cm^{-1}): 524(s), 645(m), 733(m), 771(s), 913(s), 1008(s), 1060(w), 1104(w), 1167(m), 1243(w), 1323(vs), 1435(s), 1473(w), 1591(s), 1633(w), 2143(vs), 2213(s), 2260(vs).

2.2. Crystal structure determination

Intensity measurements for **1** and **2** were made at 293 K on a Bruker SMART Apex CCD diffractometer and a FR590 CAD4 four-circle diffractometer, respectively, using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) with a $\theta/2\theta$ scan method. Crystal data, data collection parameters, and refinement statistics for **1** and **2** are listed in Table 1. Relevant bond distances and bond angles for **1** and **2** are collected in Tables 2 and 3. The structures were solved by direct methods using SHELXS-97 [18] and refined by full-matrix least-squares calculation on F^2 with SHELXL-97 [19]. All non-hydrogen atoms were refined anisotropically, whereas the hydrogen atoms were generated geometrically.

Table 1
Crystal data and structural refinement of **1** and **2**

	1	**2**
Crystal size (mm)	0.30 × 0.20 × 0.20	0.12 × 0.11 × 0.09
Formula	$C_{14}H_8N_6O_3Pb$	$C_{14}H_{10}N_8OPb$
M	515.45	513.49
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions		
a (Å)	8.702(1)	8.165(1)
b (Å)	9.337(1)	9.937(1)
c (Å)	10.255(1)	10.594(1)
α (°)	67.88(1)	73.004(1)
β (°)	87.60(1)	77.531(1)
γ (°)	74.53(1)	81.031(1)
U (Å^3)	742.33(1)	798.6(2)
Z	2	2
T (K)	293	293
λ (Å)	0.71073	0.71073
D_{calc} (g cm^{-3})	2.306	2.131
Unique data	2567	2745
Observed data	2354	2448
R (wR)	0.037(0.092)	0.030(0.077)
GOF	1.006	1.036

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

Bond lengths			
Pb(1)–N(2)	2.497(7)	Pb(1)–N(1)	2.522(6)
Pb(1)–O(1)	2.692(7)	Pb(1)–N(4)	2.707(7)
Pb(1)–N(6b)	2.792(7)	Pb(1)–O(2)	2.797(6)
Pb(1)–N(4a)	2.856(7)		
Bond angles			
N(2)–Pb(1)–N(1)	65.2(2)	N(2)–Pb(1)–O(1)	68.7(2)
N(1)–Pb(1)–O(1)	72.8(2)	N(2)–Pb(1)–N(4)	86.1(2)
N(1)–Pb(1)–N(4)	74.1(2)	O(1)–Pb(1)–N(4)	144.5(2)
N(2)–Pb(1)–N(6b)	95.1(3)	N(1)–Pb(1)–N(6b)	143.2(3)
O(1)–Pb(1)–N(6b)	71.0(2)	N(4)–Pb(1)–N(6b)	138.3(2)
N(2)–Pb(1)–O(2)	114.2(3)	N(1)–Pb(1)–O(2)	86.5(3)
O(1)–Pb(1)–O(2)	45.8(3)	N(4)–Pb(1)–O(2)	143.6(3)
N(6b)–Pb(1)–O(2)	72.3(4)	N(2)–Pb(1)–N(4a)	81.7(3)
N(1)–Pb(1)–N(4a)	132.3(3)	O(1)–Pb(1)–N(4a)	127.3(3)
N(4)–Pb(1)–N(4a)	69.0(4)	N(6b)–Pb(1)–N(4a)	70.3(3)
O(2)–Pb(1)–N(4a)	140.2(4)		

Symmetry codes, a: $1-x$, $2-y$, $1-z$; b: x, $-1+y$, z.

Table 3
Selected bond lengths (Å) and bond angles (°) for **2**

Bond lengths			
Pb(1)–N(1)	2.571(6)	Pb(1)–N(2)	2.569(6)
Pb(1)–N(3)	2.576(6)	Pb(1)–O(1W)	2.600(5)
Pb(1)–N(5a)	2.784(7)	Pb(1)–N(5b)	2.828(6)
Pb(1)–N(6c)	2.970(7)	Pb(1)–N(8d)	3.004(8)
Bond angles			
N(2)–Pb(1)–N(1)	63.1(2)	N(2)–Pb(1)–N(3)	72.6(2)
N(1)–Pb(1)–N(3)	104.4(2)	N(2)–Pb(1)–O(1W)	113.9(2)
N(1)–Pb(1)–O(1W)	75.1(2)	N(3)–Pb(1)–O(1W)	71.3(2)
N(2)–Pb(1)–N(5a)	75.5(2)	N(1)–Pb(1)–N(5a)	81.1(2)
N(3)–Pb(1)–N(5a)	140.2(2)	O(1W)–Pb(1)–N(5a)	145.3(2)
N(2)–Pb(1)–N(5b)	81.6(2)	N(1)–Pb(1)–N(5b)	140.0(2)
N(3)–Pb(1)–N(5b)	80.5(2)	O(1W)–Pb(1)–N(5b)	140.7(2)
N(5a)–Pb(1)–N(5b)	71.8(2)	N(2)–Pb(1)–N(6c)	142.2(2)
N(1)–Pb(1)–N(6c)	146.2(2)	N(3)–Pb(1)–N(6c)	75.4(2)
O(1W)–Pb(1)–N(6c)	73.0(2)	N(5a)–Pb(1)–N(6c)	121.1(2)
N(5b)–Pb(1)–N(6c)	73.8(2)	N(2)–Pb(1)–N(8d)	134.7(2)
N(1)–Pb(1)–N(8d)	79.0(2)	N(3)–Pb(1)–N(8d)	144.9(2)
O(1W)–Pb(1)–N(8d)	76.1(2)	N(5a)–Pb(1)–N(8d)	74.8(2)
N(5b)–Pb(1)–N(8d)	119.7(2)	N(6c)–Pb(1)–N(8d)	82.9(2)

Symmetry codes: a: $1+x$, y, z; b: $1-x$, $2-y$, $-z$; c: x, y, $-1+z$; d: $1+x$, y, $-1+z$.

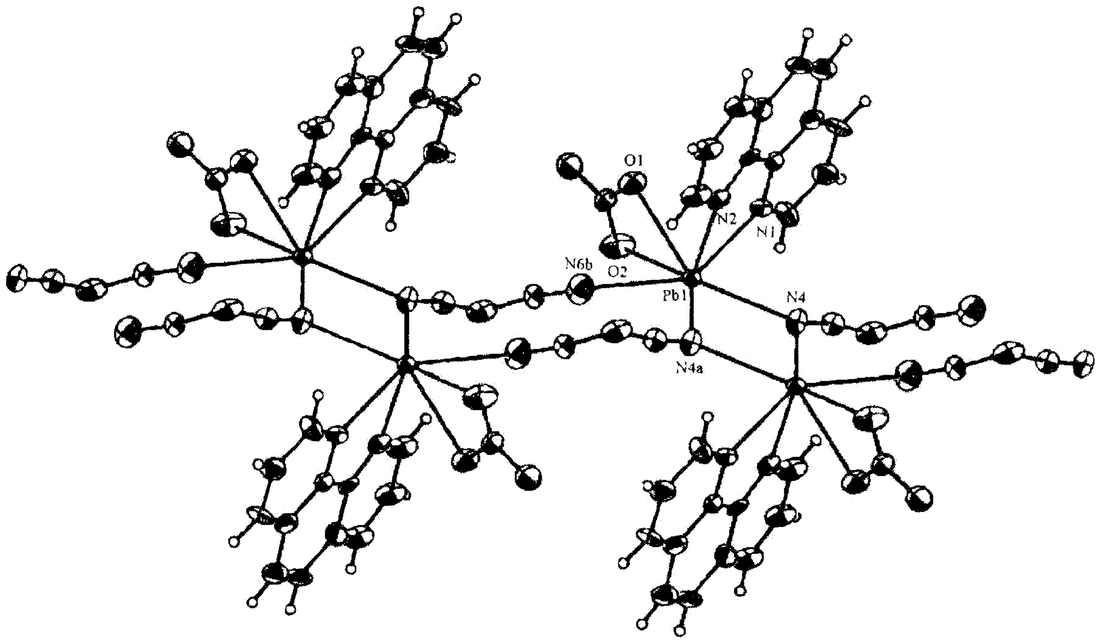

Fig. 1. The coordination environment of Pb(II) and the double-stranded structure of **1**.

3. Results and discussions

The coordination environment of Pb(II) in Pb(dca)(NO_3)(phen) (**1**) is shown in Fig. 1. Each lead atom is seven-coordinated with two oxygen atoms from NO_3^- group, two nitrogen atoms from 1,10-phen, and three nitrogen atoms from bridging dca ligands. The coordination geometry is far from regular. The structure consists of a one-dimensional chain bridged by dca ligands in 1,1,5-μ_3 coordination mode, **VII**, which has not been observed in the literature. The pairwise bridging between adjacent chains gives rise to a novel double-stranded chain with the formation of a Pb_2N_2 rhomb, see Fig. 1.

The selected bond distances and bond angles for **1** are listed in Table 2. The average distance of Pb–N(phen) is 2.509 Å, which is in good agreement with the reported values [4b]. The coordination of NO_3^- is unsymmetrical with the Pb–O distances of 2.692(7) and 2.797(6) Å, respectively, which have been similarly observed in $Pb_2(NO_2)(NO_3)(SeO_3)$ [20]. There are three different Pb–N bond distances for Pb–N(dca). The shorter bond is 2.707(7) Å occurring for Pb–N(4), and the value of 2.792(7) Å for Pb–N(6b) within the single chain. The

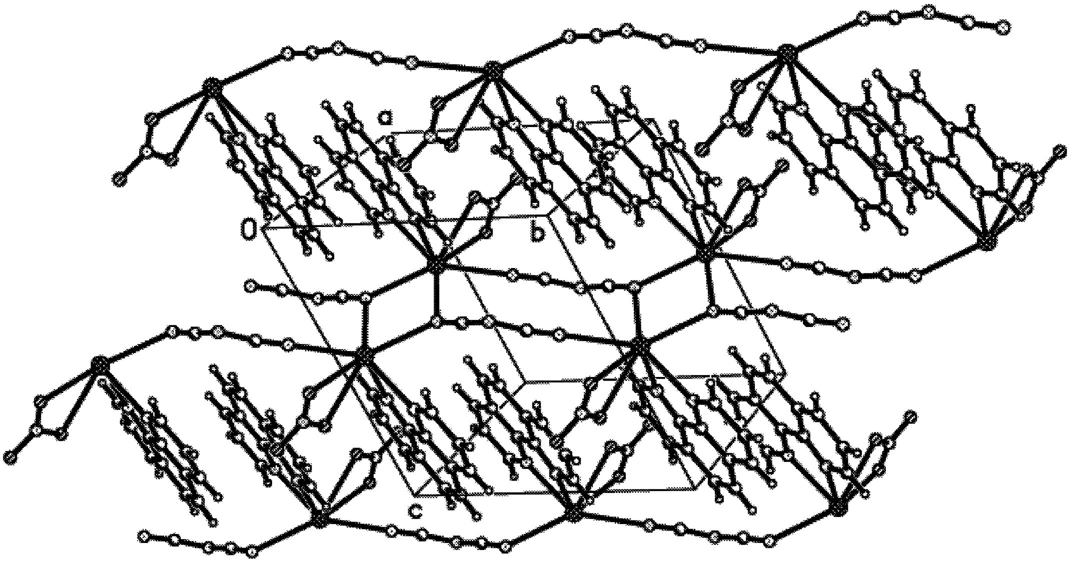

Fig. 2. The crystal packing of **1** showing aromatic ring interactions.

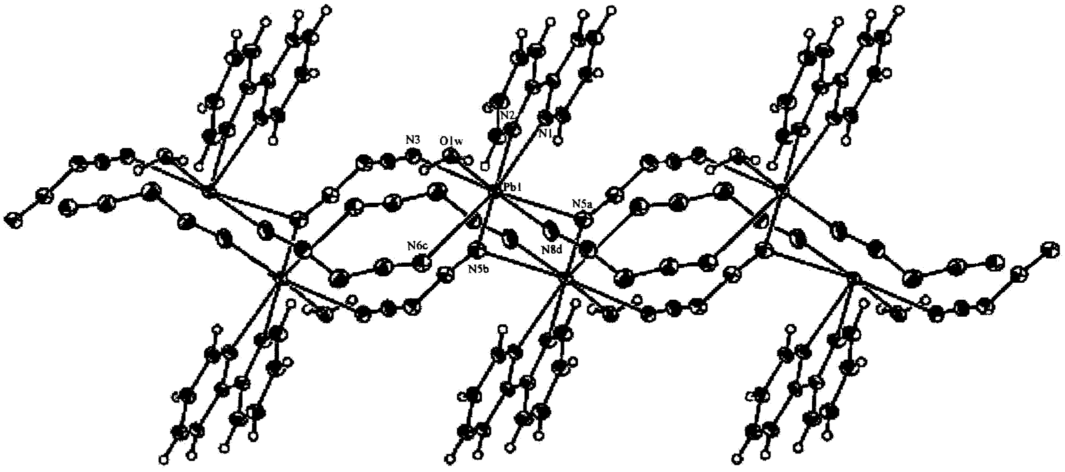

Fig. 3. The coordination environment of Pb(II) and the double-stranded structure of **2**.

distance of 2.850(6) Å was observed for the inter-chain bridging bond Pb(1)–N(4a) leading to the double-stranded chain. Even though this distance is relatively long, it can be marginally viewed as weak bonding. Comparable Pb–N bond distances have been reported, for example, 2.827 Å in {HC(3,5-$Me_2Pz)_3$}Pb{HB(3,5-$Me_2Pz)_3$}(BF_4) [21] and 2.655–2.956 Å in [Pb(phen)$_4$(O-ClO_3)](ClO_4) [22]. The Pb···Pb separation is 4.570 Å in the Pb_2N_2 planar unit.

The crystal structure of **1** is shown in Fig. 2. There are weak interactions between adjacent double-stranded chains. The aromatic rings of 1,10-phen ligands interdigitate each other within the adjacent double-stranded chain with pairwise π–π stacking at about 3.47 Å.

The coordination environment of Pb(II) in Pb(dca)$_2$(2,2′-bipy)(H_2O) (**2**) is shown in Fig. 3. Similar to **1**, the structure of **2** consists of double-stranded chains with bridging dca in the unusual 1,1,5-μ_3 mode, mode **VI**. However, in contrast with **1**, there is also one 1,5-μ_2 bridging dca in mode **II** with relatively long Pb–N distances (Pb(1)–N(6c), 2.970(7) Å; Pb(1)–N(8d), 3.004(8) Å), indicating the weak bonding. Each lead atom can be considered eight-coordinated with one aqua oxygen atom, two nitrogen atoms of 2,2-bipyridine, four-nitrogen atoms from bridging dca ligands. The coordination polyhedron is also far from regular. The mean Pb–N(bipy) bond distance is 2.569 Å, comparable to the reported values [3a]. There are three different Pb–N(dca) bond distances occurring in Pb–1,1,5-μ_3-dca. Pb–N(3) and Pb–N(5a) bonds are 2.576(6) and 2.784(7) Å while Pb–N(5b) bond is 2.828(6) Å, comparable to those in **1**. The Pb···Pb separation is 4.546(5) Å in the

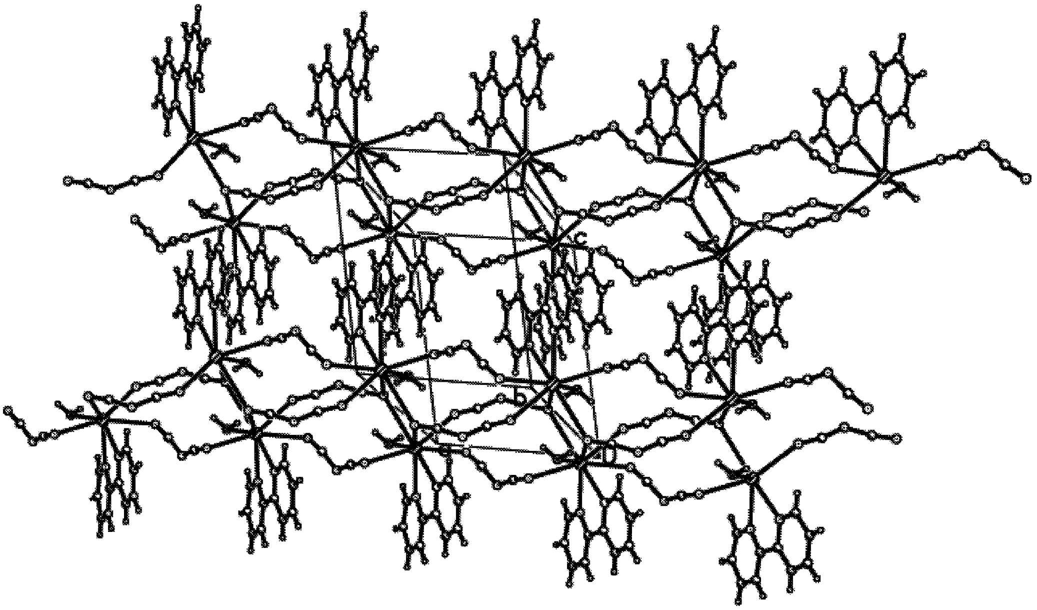

Fig. 4. The crystal packing of **2** showing aromatic ring interactions.

Pb_2N_2 planar unit. Again, there is π–π stacking between the aromatic rings of 2,2-bipyridine at 3.57 Å, see Fig. 4.

The most interesting structural feacture of **1** and **2** is that they have novel one-dimensional double-stranded chains resulting from the presence of the novel coordination mode 1,1,5-μ_3 of dca. Secondly, lead atoms are coordinated by both strong and weak Pb–N bonds. Thirdly, there are π–π stacking between the aromatic rings. The chain feature of **1** and **2** resemble those of the manganese complexes [Mn(dca)(H_2O)(terpy)](dca) and [Mn(dca)(NO_3)(terpy)] [8g], where there is no Mn–N bonding between adjacent chains and dca exhibits the common 1,5-μ_2 mode. The present observation further indicates the novel characteristics of the coordination chemistry of lead and the versatile coordination modes of dca.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 188823 and 188824 for compounds **1** and **2**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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