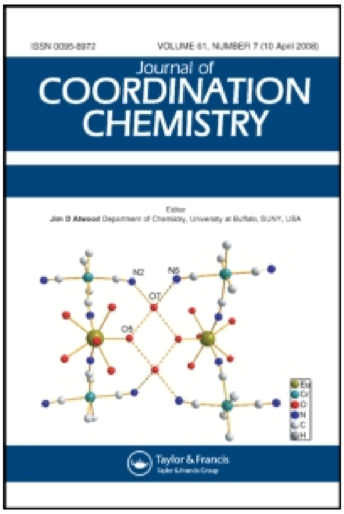

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Syntheses, crystal structures and luminescence properties of cadmium(II) *O,O'*-dialkyldithiophosphates with nitrogen donors

Tao Li[ab]; Zhi-Hua Li[a]; Sheng-Min Hu[a]; Shao-Wu Du[a]
[a] State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian, 350002, China [b] College of Life Sciences, Fujian Agriculture and Forestry University, Fuzhou, Fujian 350002, China

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Syntheses, crystal structures and luminescence properties of cadmium(II) *O*,*O*′-dialkyldithiophosphates with nitrogen donors

TAO LI†‡, ZHI-HUA LI†, SHENG-MIN HU† and SHAO-WU DU*†

†State Key Laboratory of Structural Chemistry, Fujian Institute of Research on
the Structure of Matter, Chinese Academy of Sciences, Fuzhou,
Fujian, 350002, China
‡College of Life Sciences, Fujian Agriculture and Forestry University,
Fuzhou, Fujian 350002, China

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The polymeric compounds $\{Cd(bix)[(CH_3O)_2PS_2]_2\}_n$ (**1**) and $\{Cd(bix)[(C_2H_5O)_2PS_2]_2\}_n$ (**2**) [bix = 1,4-bis(imidazole-l-ylmethyl)benzene; $(CH_3O)_2PS_2 = O,O'$-dimethyldithiophosphate; $(C_2H_5O)_2PS_2 = O,O'$-diethyldithiophosphate] have been synthesized and characterized by single-crystal structure determinations, elemental analyses, and IR spectra. X-ray diffraction studies revealed that they exhibit one-dimensional polymeric structures with a zigzag chain for **1** and a linear chain for **2**, connected by π–π stacking interactions to form 2D networks. Their solid-state luminescent properties were also studied. Crystal data for **1** at −100°C: monoclinic, space group $C2/c$, $a = 18.5196(7)$, $b = 10.2337(3)$, $c = 14.0241(11)$ Å, $\beta = 90.190(2)°$, $Z = 4$, $R_1 = 0.0362$; for **2** at −100°C: triclinic, space group $P\bar{1}$, $a = 8.9837(10)$, $b = 9.5038(0)$, $c = 10.4893(0)$ Å, $\alpha = 65.706(9)$, $\beta = 77.856(10)$, $\gamma = 73.184(10)°$, $Z = 1$, $R_1 = 0.0316$.

Keywords: Cadmium(II); 1,4-Bis(imidazole-l-ylmethyl)benzene; *O*,*O*′-Dialkyldithiophosphate; Coordination polymers; One-dimensional chains; Crystal structure; Luminescence

1. Introduction

O,*O*′-dialkyldithiophosphate (dtp) complexes of transition metals have attracted much interest because of their versatile coordination modes, varied molecular topologies and extensive commercial applications due to their high antioxidant activity and antiwear properties [1–9]. There is a great deal of literature available detailing the syntheses, chemistry and structural studies of such complexes, of which adducts containing diamagnetic metal atoms are useful in a number of ways [10]. In addition, amines, which can act as Lewis bases, may bond to the metal centres and modify the structure [11–14]. With this in mind, 1,4-bis(imidazole-l-ylmethyl)benzene (bix),

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*Corresponding author. Email: swdu@fjirsm.ac.cn

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Table 1. Crystallographic data for compounds **1** and **2**.

Compound	**1**	**2**
Empirical formula	$C_{18}H_{26}CdN_4O_4P_2S_4$	$C_{22}H_{34}CdN_4O_4P_2S_4$
Formula weight	665.01	721.11
Temperature (K)	173(2)	173(2)
Crystal system	Monoclinic	Triclinic,
Space group	$C2/c$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	18.5196(7)	8.9837(10)
b	10.2337(3)	9.5038(0)
c	14.0241(11)	10.4893(0)
α		65.706(9)
β	90.190(18)	77.856(10)
γ	0	73.184(10)
Volume (Å^3)	2657.9(2)	777.065(9)
Z	4	1
D_c (g cm^{-3})	1.662	1.541
μ (mm^{-1})	1.288	1.108
F (000)	1344	368
θ range (°)	3.64–27.48	3.03–27.48
Reflections collected	3034	3506
Independent reflections	2822	3126
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	3034/0/150	3506/0/169
Goodness-of-fit on F^2	1.001	1.002
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0362$, $wR_2 = 0.0651$	$R_1 = 0.0316$, $wR_2 = 0.0572$
R indices (all data)	$R_1 = 0.0402$, $wR_2 = 0.072$	$R_1 = 0.0378$, $wR_2 = 0.0602$
Largest diff. peak and hole (e Å^{-3})	0.507 and −0.324	0.397 and −0.354

a versatile N-donor ligand [15, 16], has been chosen to further explore the system. In this article, we report the syntheses and luminescence of two complexes with different one-dimensional chain structures $\{Cd(bix)[(CH_3O)_2PS_2]_2\}_n$ (**1**) and $\{Cd(bix)[(C_2H_5O)_2PS_2]_2\}_n$ (**2**).

2. Experimental

All general chemicals were commercially available and used without further purification. Bix was prepared as described in the literature [15]. Elemental analyses were carried out using a Vario ELIII instrument. IR spectra (KBr pellets) were recorded on a Perkin-Elmer Spectrum One FTIR spectrophotometer in the range of 4000–400 cm^{-1}. Fluorescence measurements were performed on an Edinburgh Instruments FLS920 system.

2.1. *Syntheses*

$\{Cd(bix)[(CH_3O)_2PS_2]_2\}_n$ (**1**): $Cd[(CH_3O)_2S_2P]_2$ (0.213 g, 0.5 mmol) and bix dihydrate (0.138 g, 0.5 mmol) were dissolved in 10 cm^3 of CH_2Cl_2. The mixture was stirred for 15 min and filtered. The filtrate was added to 10 cm^3 of CH_3CN and allowed to stand in air at room temperature for several days. Colourless blocky crystals of **1** were obtained. Yield 86%. Anal. Calcd for $C_{18}H_{26}CdN_4O_4P_2S_4$ (%): C, 32.44; H, 3.93; N, 8.41. Found: C, 32.66; H, 4.05; N, 8.56. IR (cm^{-1}): 3449 (s), 1636 (m),

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

Cd(1)–N(1)	2.313(2)	N(2)–C(3)	1.346(4)
Cd(1)–N(1)#1	2.313(2)	N(2)–C(5)	1.362(4)
Cd(1)–S(2)#1	2.7387(8)	N(2)–C(6)	1.470(4)
Cd(1)–S(2)	2.7387(8)	C(1)–O(1)	1.442(4)
Cd(1)–S(1)	2.7681(8)	C(2)–O(2)	1.430(4)
Cd(1)–S(1)#1	2.7681(8)	C(4)–C(5)	1.345(4)
P(1)–O(1)	1.587(2)	C(6)–C(7)	1.512(4)
P(1)–O(2)	1.601(2)	C(7)–C(8)	1.356(4)
P(1)–S(1)	1.9735(10)	C(7)–C(9)	1.375(4)
P(1)–S(2)	1.9764(11)	C(8)–C(9)#2	1.384(5)
N(1)–C(3)	1.319(4)	C(9)–C(8)#2	1.384(5)
N(1)–C(4)	1.368(4)		
N(1)–Cd(1)–N(1)#1	86.92(11)	S(1)–P(1)–S(2)	114.44(5)
N(1)–Cd(1)–S(2)#1	163.18(6)	P(1)–S(1)–Cd(1)	84.88(4)
N(1)#1–Cd(1)–S(2)#1	93.07(6)	P(1)–S(2)–Cd(1)	85.64(3)
N(1)–Cd(1)–S(2)	93.07(6)	C(3)–N(1)–C(4)	105.3(2)
N(1)#1–Cd(1)–S(2)	163.18(6)	C(3)–N(1)–Cd(1)	126.6(2)
S(2)#1–Cd(1)–S(2)	91.74(4)	C(4)–N(1)–Cd(1)	127.86(19)
N(1)–Cd(1)–S(1)	93.41(6)	C(3)–N(2)–C(5)	106.8(2)
N(1)#1–Cd(1)–S(1)	89.03(6)	C(3)–N(2)–C(6)	126.4(3)
S(2)#1–Cd(1)–S(1)	103.41(3)	C(5)–N(2)–C(6)	126.8(3)
S(2)–Cd(1)–S(1)	74.18(2)	N(1)–C(3)–N(2)	111.4(3)
N(1)–Cd(1)–S(1)#1	89.03(6)	C(5)–C(4)–N(1)	109.9(3)
N(1)#1–Cd(1)–S(1)#1	93.41(6)	C(4)–C(5)–N(2)	106.6(3)
S(2)#1–Cd(1)–S(1)#1	74.18(2)	N(2)–C(6)–C(7)	112.9(2)
S(2)–Cd(1)–S(1)#1	103.41(3)	C(8)–C(7)–C(9)	118.0(3)
S(1)–Cd(1)–S(1)#1	176.65(4)	C(8)–C(7)–C(6)	120.5(3)
O(1)–P(1)–O(2)	98.79(11)	C(9)–C(7)–C(6)	121.5(3)
O(1)–P(1)–S(1)	112.51(9)	C(7)–C(8)–C(9)#2	121.3(3)
O(2)–P(1)–S(1)	111.42(9)	C(7)–C(9)–C(8)#2	120.8(3)
O(1)–P(1)–S(2)	107.58(9)	C(1)–O(1)–P(1)	120.7(2)
O(2)–P(1)–S(2)	110.97(9)	C(2)–O(2)–P(1)	120.49(19)

Symmetry codes: #1 = $-x+1, y, -z+3/2$; #2 = $-x+3/2, -y+1/2, -z+1$.

1515 (m), 1435 (s), 1384 (w), 1235 (m), 1178 (w), 1113 (w), 1095 (m), 1047 (m), 1022 (w), 934 (w), 858 (w), 791 (m), 769 (s), 665 (s) and 527 (w). $\{Cd(bix)[(C_2H_5O)_2S_2P]_2\}_n$ (**2**): $Cd[(C_2H_5O)_2S_2P]_2$ (0.241 g, 0.5 mmol) and bix dihydrate (0.138 g, 0.5 mmol) were dissolved in 10 cm^3 of CH_2Cl_2 and 2 cm^3 of CH_3OH. The mixture was stirred for 15 min and filtered. The filtrate was kept in air at room temperature. After a few days colourless blocky crystals of **2** were obtained in 78% yield. Anal. Calcd for $C_{22}H_{34}CdN_4O_4P_2S_4$ (%): C, 36.57; H, 4.75; N, 7.76. Found: C, 36.44; H, 4.65; N, 7.61. IR (cm^{-1}): 3445 (s), 1652 (m), 1517 (m), 1433 (w), 1385 (w), 1239 (m), 1156 (w), 1106 (m), 1084 (m), 1027 (w), 952 (s), 826 (w), 788 (m), 745 (m), 681 (w) and 576 (w).

2.2. *X-ray crystallography*

Colorless crystals of **1** ($0.35 \times 0.20 \times 0.20\,mm^3$) and **2** ($0.20 \times 0.20 \times 0.10\,mm^3$) were used for data collection on a Rigaku mercury CCD diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Empirical absorption corrections were applied using SADABS [17]. The structures were solved by direct methods for **1** and Patterson methods for **2**, and refined by full-matrix least-squares methods. All hydrogen atoms were calculated at the ideal positions and refined isotropically; all non-hydrogen atoms were refined anisotropically. Programs used

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

Cd(1)–N(1)	2.3404(19)	N(1)–C(2)	1.378(3)
Cd(1)–N(1)#1	2.3404(19)	N(2)–C(1)	1.341(3)
Cd(1)–S(1)#1	2.7051(7)	N(2)–C(3)	1.369(3)
Cd(1)–S(1)	2.7051(7)	N(2)–C(4)	1.464(3)
Cd(1)–S(2)#1	2.7992(8)	C(2)–C(3)	1.349(4)
Cd(1)–S(2)	2.7992(8)	C(4)–C(5)	1.511(3)
S(1)–P(1)	1.9812(10)	C(5)–C(6)	1.379(3)
S(2)–P(1)	1.9789(10)	C(5)–C(7)	1.386(3)
P(1)–O(2)	1.5858(19)	C(6)–C(7)#2	1.386(3)
P(1)–O(1)	1.5934(19)	C(7)–C(6)#2	1.386(3)
O(1)–C(10)	1.457(4)	C(8)–C(9)	1.463(4)
O(2)–C(8)	1.440(4)	C(10)–C(11)	1.434(5)
N(1)–C(1)	1.317(3)		
N(1)#1–Cd(1)–N(1)	180.00(9)	O(1)–P(1)–S(1)	105.08(9)
N(1)#1–Cd(1)–S(1)	90.06(5)	S(2)–P(1)–S(1)	115.81(4)
N(1)–Cd(1)–S(1)	89.94(5)	C(10)–O(1)–P(1)	120.2(2)
N(1)#1–Cd(1)–S(1)#1	89.94(5)	C(8)–O(2)–P(1)	120.6(2)
N(1)–Cd(1)–S(1)#1	90.06(5)	C(1)–N(1)–C(2)	105.0(2)
S(1)–Cd(1)–S(1)#1	180.00(3)	C(1)–N(1)–Cd(1)	125.84(16)
N(1)#1–Cd(1)–S(2)	89.33(5)	C(2)–N(1)–Cd(1)	129.07(16)
N(1)–Cd(1)–S(2)	90.67(5)	C(1)–N(2)–C(3)	106.7(2)
S(1)–Cd(1)–S(2)	75.09(2)	C(1)–N(2)–C(4)	126.5(2)
S(1)#1–Cd(1)–S(2)	104.91(2)	C(3)–N(2)–C(4)	126.8(2)
N(1)#1–Cd(1)–S(2)#1	90.67(5)	N(1)–C(1)–N(2)	112.0(2)
N(1)–Cd(1)–S(2)#1	89.33(5)	C(3)–C(2)–N(1)	109.7(2)
S(1)–Cd(1)–S(2)#1	104.91(2)	C(2)–C(3)–N(2)	106.5(2)
S(1)#1–Cd(1)–S(2)#1	75.09(2)	N(2)–C(4)–C(5)	113.3(2)
S(2)–Cd(1)–S(2)#1	180.0	C(6)–C(5)–C(7)	118.3(2)
P(1)–S(1)–Cd(1)	85.74(3)	C(6)–C(5)–C(4)	120.3(2)
P(1)–S(2)–Cd(1)	83.24(3)	C(7)–C(5)–C(4)	121.4(2)
O(2)–P(1)–O(1)	105.28(11)	C(5)–C(6)–C(7)#2	120.9(2)
O(2)–P(1)–S(2)	105.79(9)	C(6)#2–C(7)–C(5)	120.9(2)
O(1)–P(1)–S(2)	111.90(10)	O(2)–C(8)–C(9)	110.1(3)
O(2)–P(1)–S(1)	112.59(9)	C(11)–C(10)–O(1)	111.4(3)

Symmetry codes: #1 = $-x+1$, $-y$, $-z+1$; #2 = $-x$, $-y$, $-z$.

for structure solution and refinement were SHELXS97 [18] and SHELXL97 [19], respectively.

3. Results and discussion

3.1. *Crystal structures*

The crystal structures of the complexes consist of dtp units linked by bix groups forming one-dimensional infinite chains, in which the bix ligands adopted the *trans* conformation. Crystallographic data are summarized in table 1 and selected bond distances and angles are given in tables 2 and 3.

As shown in figure 1(a), compound **1** consists of alternating $Cd[(CH_3O)_2S_2P]_2$ groups and bix ligands, with cadmium on the C_2 axis. Each Cd(II) ion is coordinated to two nitrogen atoms from two bridging bix ligands and four sulfur atoms from two bidentate dimethyldithiophosphates, forming distorted octahedral geometry. The two nitrogen atoms of the two bix ligands on each Cd(II) centre lie *cis* to each other with Cd–N 2.313(2) Å and N1–Cd1–N1A 86.9(1)°. The imidazole ring and Cd form a plane,

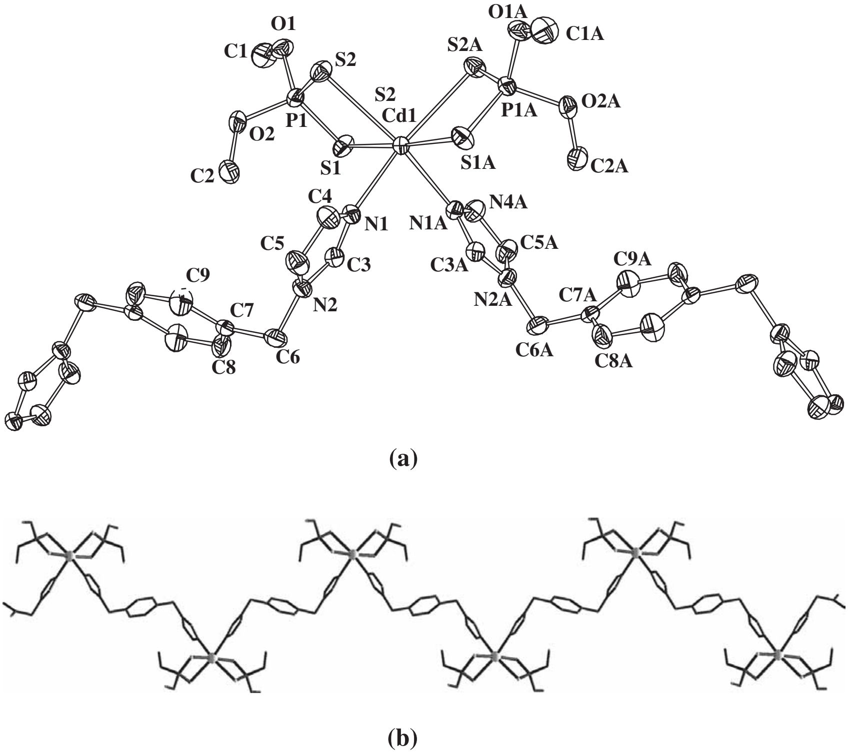


Figure 1. (a) Section of the crystal structure of **1**. Displacement ellipsoids are plotted at the 30% probability level. Hydrogen atoms are omitted for clarity. Symmetry labels: $A = -x + 1, \ y, \ -z + 3/2$; (b) The zigzag chain in **1**.

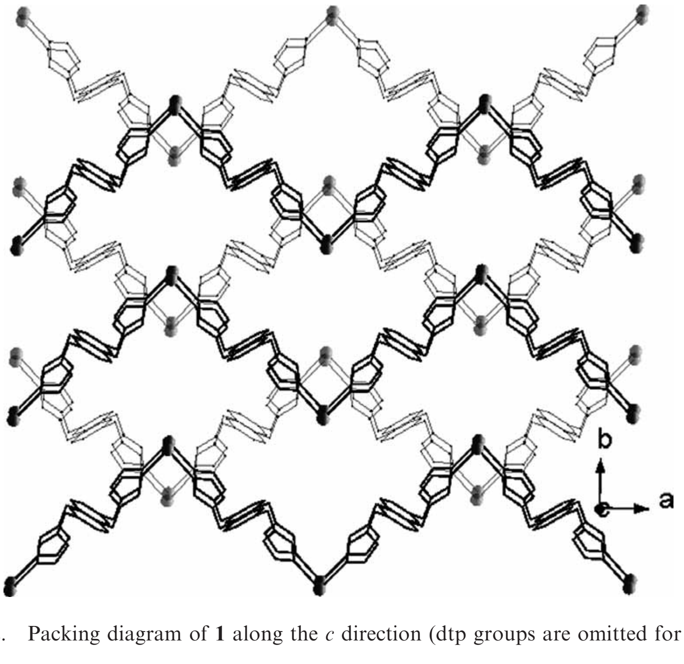
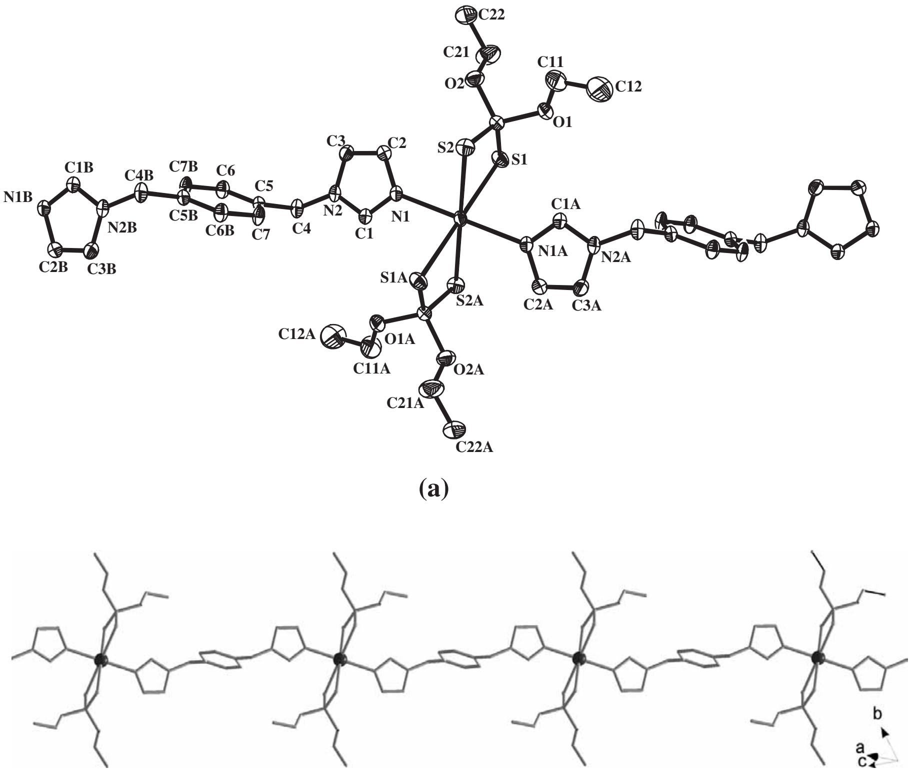


Figure 2. Packing diagram of **1** along the *c* direction (dtp groups are omitted for clarity).



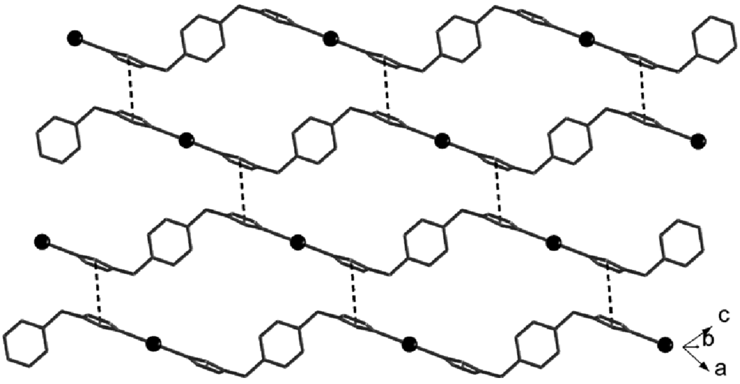
(a)

(b)

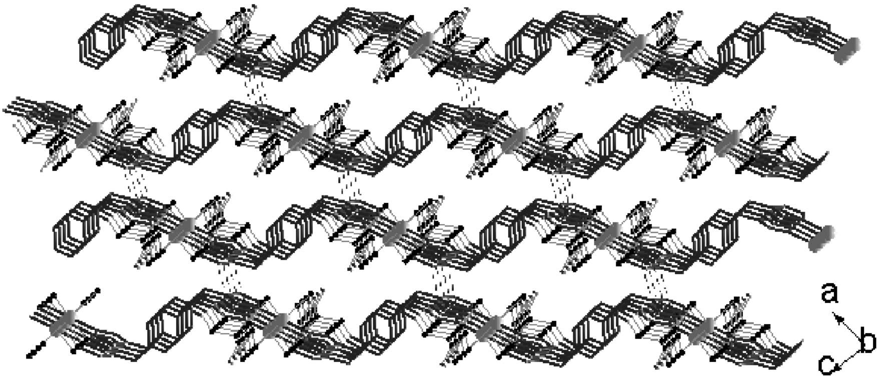
Figure 3. (a) Section of the crystal structure of **2**. Displacement ellipsoids are plotted at the 30% probability level. Hydrogen atoms are omitted for clarity. Symmetry labels: A = −*x*, −*y*, 1 − *z*; B = 2 − *x*, −*y*, −*z*; (b) The linear chain in **2**.

as does the four-membered ring formed by Cd1, S1, S2 and P1; mean deviations from best-fit planes are 0.0309 and 0.0631 Å, respectively. The two imidazole rings coordinated to the Cd(II) centre make a dihedral angle of 64.3°. The *cis*-linking mode gives an infinite zigzag chain (figure 1b). The zigzag chains are arranged parallel to form a layer with layers stacking in a {ABAB} pattern. A perspective view of the packing is shown in figure 2.

In **2** each Cd(II) ion lies on a crystallographic centre of inversion and is coordinated to two bridging bix ligands and two bidentate dtp ligands. Unlike the case in **1**, compound **2** possesses a *trans*-linking mode. As shown in figure 3(a), in each CdN_2S_4 octahedron four sulfur atoms of the chelating diethyldithiophosphates ligands lie in the equatorial plane, with an average Cd–S bond length of 2.751(9) Å. N atoms of bridging bix ligand lie in apical positions with Cd–N equal to 2.340(2) Å. The two imidazole rings and the central cadmium ion are almost co-planar, and nearly perpendicular to the plane formed by S1, S2, S1a and S2a (dihedral angle 88.1°). Thus, compound **2** forms an infinite linear chain with a linear array of cadmium ions (figure 3b). The difference in structure between **1** and **2** may be attributed to the different steric requirements of the dialkyldithiophosphate ligands. It is noted that the infinite linear chains of **2** form two-dimensional layers by a face-to-face



(a)



(b)

Figure 4. (a) A two-dimensional supramolecular layer constructed by π–π interactions (dashed line) in **2**. (b) Layers described in (a) are stacked parallel, showing large channels along the *b*-axis.

π–π stacking interaction of parallel aromatic rings of adjacent chains with a separation distance of ca 3.5 Å (figure 4a). As shown in figure 4(b), these 2 supramolecular layers are parallel with large channels along the *b*-axis. The space in the channels is not occupied by dtp ligands. Instead, the dtp ligands lie between the 2D layers.

3.2. *Luminescence properties*

Solid state emission spectra of **1** and **2** at room temperature have been studied. With excitation at $\lambda = 338$ and 350 nm, **1** and **2** show intense emission at 416 and 423 nm, respectively, with a slight red shift from **1** to **2**. Since this is similar to the emission of the free bix ligand ($\lambda_{ex} = 350$, $\lambda_{em} = 392$), we assume that the origin of emission in **1** and **2** is neither metal-to-ligand charge transfer nor ligand-to-metal charge transfer in nature and can be attributed to intraligand emission states [20, 21]. The clear red shift of the emission of **2** may be associated with the aromatic π–π stacking interactions present in the solid state.

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC 270600 and 270601 for **1** and **2**, respectively. Copies of this information may be obtained from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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