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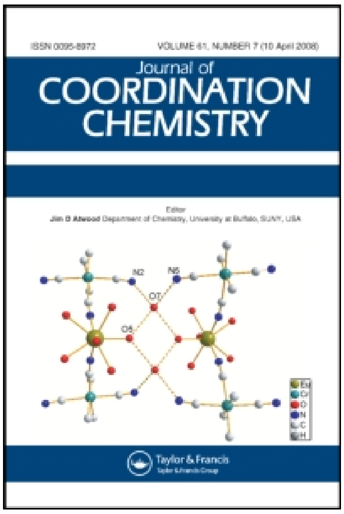

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Cadmium(II) diallyldithiocarbamato complexes with 2,2'-bipyridine and 1,10-phenanthroline: spectroscopic and crystal structure analysis

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Cadmium(II) diallyldithiocarbamato complexes with 2,2′-bipyridine and 1,10-phenanthroline: spectroscopic and crystal structure analysis

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Complexes of Cd(II) with diallyldithiocarbamato (hereafter denoted aldtc) and 2,2′-bipyridine (bipy) and 1,10-phenanthroline (phen) are discussed. Derivatives of general formula [Cd(aldtc)$_2$(NN)] [NN = bipy, **1** and phen, **2**] have been obtained by direct reaction between Cd(NO$_3$)$_2$ and a 2 : 1 molar ratio of aldtc and NN. The new complexes have been characterized by IR, ^{1}H, and ^{13}C NMR spectroscopy. Their single crystal structures were also determined. Compounds **1** and **2** have severely distorted octahedral coordination around cadmium, defined by an N$_2$S$_4$ donor set. The structure of **1** is isomorphous with the recently reported zinc analogue. The crystal packing of **1** shows different non-classical intermolecular interactions represented in both hydrophilic (π)C–H $\cdots$ S and hydrophobic (allyl)C–H $\cdots$ C(π) intermolecular interactions. Such interactions result in a chain arrangement of molecules along the crystallographic *c*-axis. These chains are further connected via $\pi \cdots \pi$ stacking along with (π)C–H $\cdots$ S parallel to *b* leading to an overall crystal packing that can be regarded as layers of complexes along the *bc* plane. Molecules in the crystal structure of **2** are arranged into infinite chains, down the *b*-axis, that are connected by aryl $\cdots$ aryl stacking. The chains are further connected to each other in *a* and *c* directions via (allyl)C–H $\cdots$ S interactions.

Keywords: Cadmium; Diallyldithiocarbamate; *N*-heterocyclic; Crystal structure; C–H $\cdots$ S and C–H $\cdots$ C(π) interactions

1. Introduction

Complexes of dithiocarbamate and its substituted derivatives have various catalytic and biological properties such as their use as accelerators in vulcanization processes, high-pressure lubricant agents and as fungicides and pesticides [1–3]. Dithiocarbamato complexes have also been considered as promising species to provide single-source materials for bulk metal sulfides [4–8]. Structures of dithiocarbamatocadmium(II) complexes containing *N*-heterocyclic ligands have been reported [9–12]. However, their

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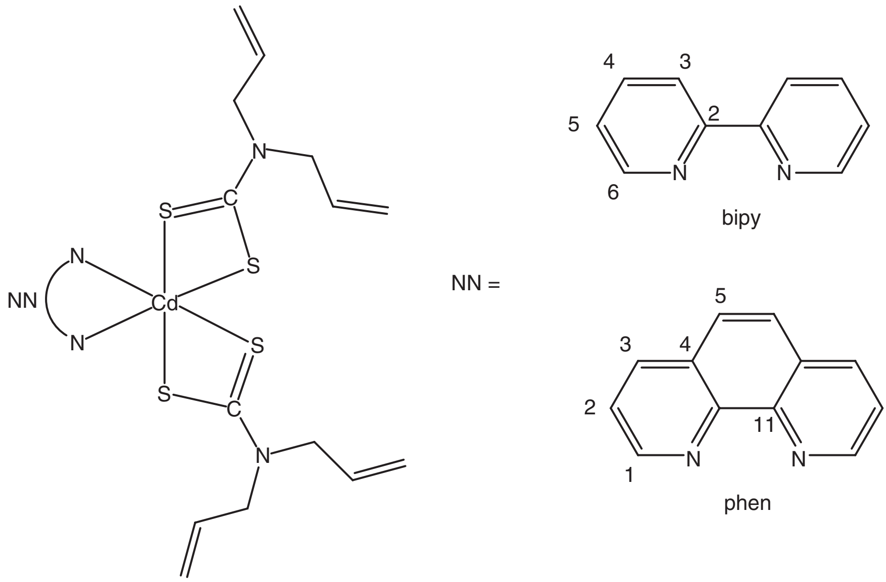

Scheme 1. Schematic representation of the prepared Cd(II) complexes showing the numbering system. Atoms without numbers are symmetry related ones.

structures were not investigated in terms of crystal packing and possible aryl ··· aryl, (allyl)C–H ··· S, and (allyl)C–H ··· aryl intermolecular interactions. Little is known about complexes containing diallyldithiocarbamato (denoted as aldtc) derivatives along with *N*-heterocyclic ligands.

Recently, we reported the crystal structure of $[Zn(aldtc)_2(bipy)]$ (bipy = 2,2′-bipyridine) with its crystal supramolecularity [13]. In the present work, we report the spectral and crystal structure of Cd(II) aldtc complexes containing *N*-heterocyclic ligands (2,2′-bipyridine (bipy) and 1,10-phenanthroline (phen); scheme 1).

2. Experimental

2.1. *Materials and instrumentation*

All manipulations were performed under aerobic conditions using solvents as received. The reagents used in the preparation of the diallyldithiocarbamate [13], $Cd(NO_3)_2$, bipy, and phen were purchased from Aldrich. Melting points were determined on an electrothermal melting point apparatus (Electrothermal 9001) and are uncorrected. Infrared (IR) spectra (4000–600 cm^{-1}; KBr pellets) were recorded on a Nicolet FT-IR spectrometer. ^1H- and ^{13}C-NMR spectra were recorded at 25°C in DMSO-d_6 on a 300 MHz Bruker AMX spectrometer using TMS as an internal standard.

2.2. *General synthesis of $[Cd(aldtc)_2(NN)]$, NN = bipy, 1; phen, 2*

To a stirred mixture of aldtc (2.0 mmol) and the corresponding NN ligand (1.0 mmol) dissolved in EtOH (40 mL) was added slowly a solution of $Cd(NO_3)_2 \cdot 4H_2O$ (1.0 mmol)

Table 1. Crystal data and structure refinement for **1** and **2**.

Identification code	**1**	**2**
CCDC No.	696807	696806
Empirical formula	$C_{24}H_{28}CdN_4S_4$	$C_{26}H_{28}CdN_4S_4$
Formula weight	613.19	637.16
T(K)	293(2)	293(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Orthorhombic	Triclinic
Space group	*Pccn*	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	24.178(7)	9.883(4)
b	6.6754(17)	10.637(5)
c	17.091(5)	15.341(6)
α	90.00	100.707(3)
β	90.00	99.541(6)
γ	90.00	112.102(7)
Volume (Å^3)	2758.5(13)	1418.2(10)
Z	4	2
D_{Calcd} (Mg m^{-3})	1.477	1.492
μ (mm^{-1})	1.11	1.09
F(000)	1248	648
θ Range for data collection (°)	2.53 to 25.02	2.77 to 25.02
Index ranges	$-8 \leq h \leq 9$, $-13 \leq k \leq 14$, $-26 \leq l \leq 25$	$-11 \leq h \leq 11$, $-12 \leq k \leq 12$, $-17 \leq l \leq 18$
Reflections collected	25,050	15,458
Independent reflections	2424 [R(int) = 0.092]	4830 [R(int) = 0.0392]
Completeness to $\theta = 25.02°$ (%)	99.5	98.3
Maximum and minimum transmission	0.946 and 0.731	1.000 and 0.6597
Data/restraints/parameters	2424/0/150	4830/0/361
Goodness-of-fit on F^2	1.434	1.035
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0984$, $wR_2 = 0.1204$	$R_1 = 0.0374$, $wR_2 = 0.0788$
R indices (all data)	$R_1 = 0.1126$, $wR_2 = 0.1143$	$R_1 = 0.0499$, $wR_2 = 0.0857$
Largest difference peak and hole (e Å^{-3})	0.384 and −0.354	0.442 and −0.481

in EtOH (20 mL). The resulting mixture was stirred for 30 min whereupon colorless solid precipitated. The solid was filtered off, washed several times with EtOH and dried. The solid was further purified by recrystallization from hot CH_3CN. The suitable crystals for single crystal crystallography obtained for **1** and **2** grew during a few days from the recrystallization process.

2.3. *Crystal structure determination*

Crystallographic data for **1** and **2** are listed in table 1. The data were collected on a Rigaku MSC Mercury CCD diffractometer in omega scans mode (Mo-Kα radiation; $\lambda = 0.71073$ Å). Cell parameters were retrieved and refined with *Crystal Clear* software [14] on all observed reflections. Data reduction and correction for Lp and decay were also performed by the *Crystal Clear* software [14]. The structures were solved by direct methods using *SHELXS-97* [15] and refined [16] by least squares on F^2. All non-hydrogen atoms were refined anisotropically; H atoms were initially located by difference Fourier map and refined with fixed individual displacement parameters using a riding model with C–H distances of 0.93 and 0.97 Å and $U_{\mathrm{iso}}(\mathrm{H}) = 1.2_{\mathrm{eq}}(\mathrm{C})$ for H atoms of allylic groups; C–H = 0.93 Å, $U_{\mathrm{iso}}(\mathrm{H}) = 1.2_{\mathrm{eq}}(\mathrm{C})$ for H atoms of aryl rings. No decomposition was observed during data collection. Details of the data collection and refinement are given in table 1. Further details are provided in the 'Supplementary material'. The R-factor and goodness-of-fit on F^2 of **1** are high, perhaps due to poor quality of the crystal.

3. Results and discussion

Reactions of $Cd(NO_3)_2$ with mixtures of ligands (2 : 1; diallyldithiocarbamate : *N*-heterocyclic) gave high yields of $[Cd(aldtc)_2(NN)]$. Various physical measurements are in accord with the formulation of the complexes (tables 2–4). The complexes are stable in air and soluble in most organic solvents.

3.1. *Spectroscopic analysis*

3.1.1. IR spectra. The principal IR bands are listed in table 2. For **1** and **2**, $\nu_{(CN)}$ is higher than that of the free ligand, consistent with reports [17–21]. A decrease in $\nu_{(CS)}$ is observed and an isolated band near $1000\,cm^{-1}$ indicates that aldtc is bidentate [17–21]. An additional characteristic band is the allylic $\nu_{(C–H)}$ at $2940\,cm^{-1}$ which further supports the presence of the diallyl derivative.

Infrared spectra of both complexes show absorption bands from skeletal vibrations of aromatic rings. The IR bands of NN were assigned on the basis of earlier studies made on bipy and phen complexes [22, and references therein].

3.1.2. ^{1}H-NMR spectra. Assignments of the peaks of aldtc and NN ligands (table 3) were made on the basis of earlier studies on complexes containing dialkyldithiocarbamato ligands [23] and complexes containing bipy and phen ligands [24–27]. Peaks in the range 4.43–5.88 ppm are due to allylic protons from coordinated aldtc, while the aromatic protons in the range 7.59–9.29 are characteristic of the NN ligands. The proton ratio of aldtc to NN (20 : 8) is in good agreement with the 2 : 1 molar ratio.

3.1.3. ^{13}C-NMR spectra. Detailed assignments of spectra (table 4) have been made on the basis of previously reported data [23–27]. Aldtc has four different types of carbon atoms while bipy and phen have five and six, respectively. These results are in good agreement with the structures.

3.2. *Molecular geometry*

The structures of **1** and **2** contain one-half molecule of **1** and one independent molecule of **2**, respectively, in the asymmetric unit (figure 1). Cd has a severely distorted octahedral arrangement in both complexes, defined by an N_2S_4 donor set (figure 1). Distortion about Cd can be illustrated by the bond lengths and angles of the complexes (table 5). In **1**, the Cd–N distances are similar [2.375(5) Å] and Cd–S distances are 2.684(2) and 2.6673(19) Å, whereas in **2**, the Cd–N distances are 2.394(3) and 2.423(3) Å while the Cd–S distances are in the range 2.6582(13)–2.6842(13) Å. These lengths are within the range of distances previously reported for similar compounds [9–12].

Table 2. Analytical and physical data with important IR stretching frequencies.

			IR frequencies of aldtc (cm^{-1})			IR frequencies of NN (cm^{-1})	
Compound	Yield (%)	m.p. (°C)	ν(CH)	ν(CN)	ν(CS)	ν(C=C) and ν(C=N)	γ(C–H)
[Cd(aldtc)$_2$(bipy)]	91	289–291	2945	1495	1002	bipy: 1638 m, 1591 s, 1564 m, 1543 w, 1465 s, 1431 s	765 s, 740 wsh
[Cd(aldtc)$_2$(phen)]	89	253–255	2941	1507	986	phen: 1649 m, 1619 m, 1602 w, 1464 s, 1391 s	850 s, 842 sh, 737 m, 725 s

m: medium; s: strong; w: weak; sh: shoulder; wsh: weak shoulder.

Table 3. ^{1}H-NMR spectra in DMSO-d$_6$.

Compound	Assignments (δ in ppm)
[Cd(aldtc)$_2$(bipy)]	allylic protons: 4.459 (d, 4H, $\underline{H}_2$C–N), 5.214 (d, 4H, $\underline{H}_2$C=CH–), 5.884 (m, 2H, $H_2C=C\underline{H}$–) bipy protons: 8.4919 (d, 2H, H_3 type), 8.076 (t, 2H, H_4 type),7.596 (t, 2H, H_5 type), 8.80 (d, 2H, H_6 type)
[Cd(aldtc)$_2$(phen)]	allylic protons: 4.47 (d, 4H, $\underline{H}_2$C–N), 5.088 (d, 4H, $\underline{H}_2$C=CH–), 5.91 (m, 2H, $H_2C=C\underline{H}$–) phen protons: 8.801 (d, 2H, H_3 type), 8.21 (dd, 2H, H_2 type), 9.44 (d, 2H, H_1 type), 8.09 (s, 2H, H_5 type)

Table 4. ^{13}C-NMR spectra in DMSO-d$_6$.

Compound	Assignments (δ in ppm)
[Cd(aldtc)$_2$(bipy)]	allylic carbons: 57.484 ($CH_2=CH–\underline{C}H_2N$), 118.682 ($\underline{C}H_2=CH–CH_2N$), 138.890 ($CH_2=\underline{C}H–CH_2N$), 207.407 ($N–\underline{C}S_2$) bipy carbons: 152.910 (C_2), 121.693 (C_3), 132.052 (C_4), 125.485 (C_5), 149.606 (C_6)
[Cd(aldtc)$_2$(phen)]	allylic carbons: 57.652 ($CH_2=CH–\underline{C}H_2N$), 118.378 ($\underline{C}H_2=CH–CH_2N$), 131.77 ($CH_2=\underline{C}H–CH_2N$), 208.421 ($N–\underline{C}S_2$) phen carbons: 149.41 ($C_1$), 129.988 ($C_2$), 138.65 ($C_3$), 124.91 ($C_5$), 126.58 ($C_6$), 142.118 ($C_{11}$)

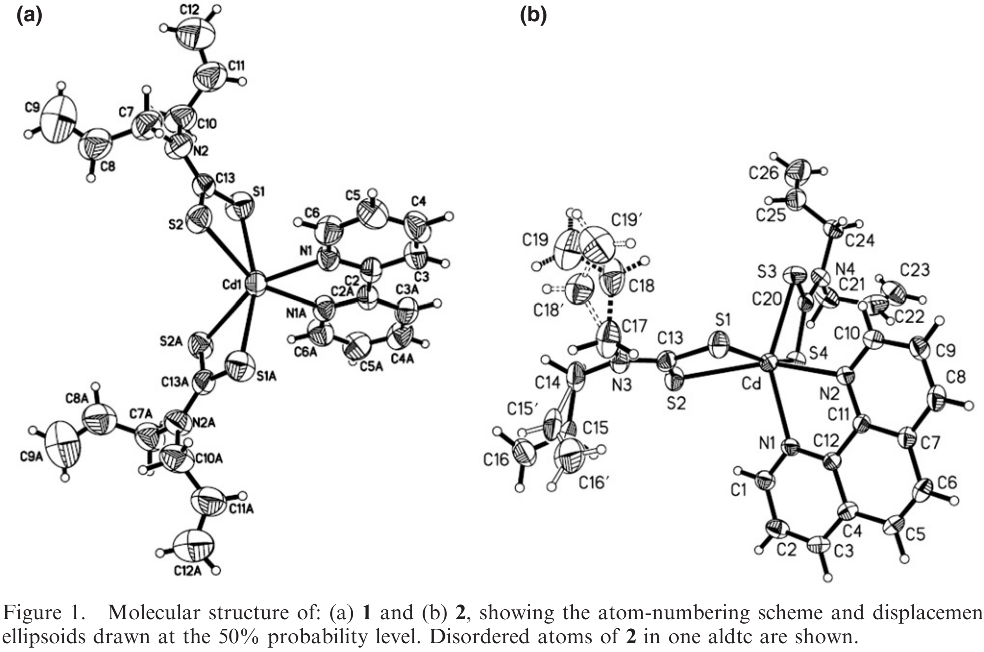

Figure 1. Molecular structure of: (a) **1** and (b) **2**, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. Disordered atoms of **2** in one aldtc are shown.

3.3. *Crystal supramolecularity and intermolecular interactions*

There are both hydrophilic and hydrophobic intermolecular interactions in the structure of **1**, table 6. The former interactions [$(\pi)C–H\cdots S$; 2.96 Å] are hydrogen bonding as a result of bipy protons bonded to S atoms from adjacent molecules, along *c*.

Table 5. Selected geometric parameters (Å, °) for **1** and **2**.

1		**2**	
Cd1–N1	2.375(5)	Cd–N1	2.394(3)
Cd1–N1^{a}	2.375(5)	Cd–N2	2.423(3)
Cd1–S2	2.6673(19)	Cd–S2	2.6582(12)
Cd1–S2^{a}	2.6673(19)	Cd–S3	2.6582(13)
Cd1–S1	2.684(2)	Cd–S1	2.6625(15)
Cd1–S1^{a}	2.684(2)	Cd–S4	2.6842(13)
N1–Cd1–S2	93.32(14)	N1–Cd–N2	68.60(9)
N1–Cd1–S1	98.92(14)	N1–Cd–S2	94.71(7)
S2–Cd1–S1	67.37(6)	N2–Cd–S2	147.86(7)
S1^{a}–Cd1–S1	160.71(9)	S2–Cd–S3	113.80(4)
N1^{a}–Cd1–N1	69.2(3)	N1–Cd–S1	105.45(7)
N1^{a}–Cd1–S2	155.01(13)	S2–Cd–S1	67.40(4)
N1–Cd1–S2^{a}	155.01(13)	N1–Cd–S4	88.97(7)
S2–Cd1–S2^{a}	108.30(9)	S2–Cd–S4	102.66(5)
N1–Cd1–S1^{a}	96.94(14)	S1–Cd–S4	162.77(3)
S2–Cd1–S1^{a}	100.86(7)	S3–Cd–S1	103.00(4)

Symmetry codes: $^{a}-x+1/2,\ -y+3/2,\ z$.

Table 6. Hydrogen bond parameters (Å, °) for **1** and **2**.

D–H $\cdots$ A	D–H	H $\cdots$ A	$D\cdots A$	D–H $\cdots$ A
Complex **1**				
C(3)–H(3A) $\cdots$ S(2)^{a}	0.93	2.96	3.367(7)	134
C(6)–H(6A) $\cdots$ S(1)^{b}	0.93	3.07	3.856(8)	144
C(8)–H(8A) $\cdots$ C(3)^{c}	0.93	2.96	3.653(12)	132
Complex **2**				
C(23)–H(23A) $\cdots$ S(1)^{d}	0.93	2.89	3.688(5)	145
C(9)–H(9A) $\cdots$ S(3)^{e}	0.93	3.06	3.847(4)	144
C(14)–H(14B) $\cdots$ S(4)^{g}	0.93	3.08	3.683(11)	125
C(16)–H(16B) $\cdots$ S(4)^{f}	0.97	3.04	3.936(4)	153

Symmetry codes: $^{a}-x+1/2,\ y,\ z+1/2$; $^{b}x,\ y-1,\ z$; $^{c}x, -y+3/2,\ z-1/2$; $^{d}x+1,\ y+1,\ z$; $^{e}-x,\ -y+1,\ -z$; $^{f}x,\ y-1,\ z$; $^{g}-x+1,\ -y+1,\ -z+1$.

The latter interactions result when the allylic protons are attracted to the bipy rings [(allyl)C–H $\cdots$ C(π); 2.96 Å], where the allylic protons are oriented in a vertex-to-face motif. These interactions result in a chain arrangement of molecules along the crystallographic *c*-axis (figure 2). The resulting chains then come together via both hydrophilic [(allyl)C–H $\cdots$ S; 3.07 Å] and hydrophobic interactions ($\pi\cdots\pi$ stacking) along the *b*-axis. These interactions are defined by distances between ring centroids: X1A [plane N1/C2/C3/C4/C5/C6 (x, y, z)] $\cdots$ X1A ($1/2-x$, $1/2-y$, z) is 3.78(9) Å and X1A ($1/2-x$, $3/2-y$, z) $\cdots$ X1A (x, $1+y$, z) being 3.92(6) Å. The overall crystal packing (figure 2) of the complex can be regarded as layers of complexes in the *bc* plane, held together through both hydrophobic and hydrophilic interactions.

The packing of **2** can be regarded as chains of molecules down the *b*-axis assembled via aryl $\cdots$ aryl stacking of phen rings (many C $\cdots$ C distances in the range 3–3.4 Å). These chains are connected to each other in *a* and *c* directions by C–H $\cdots$ S interactions (table 6) causing a 3-D assembly. The importance of H $\cdots$ S interactions have been discussed previously [13, 28].

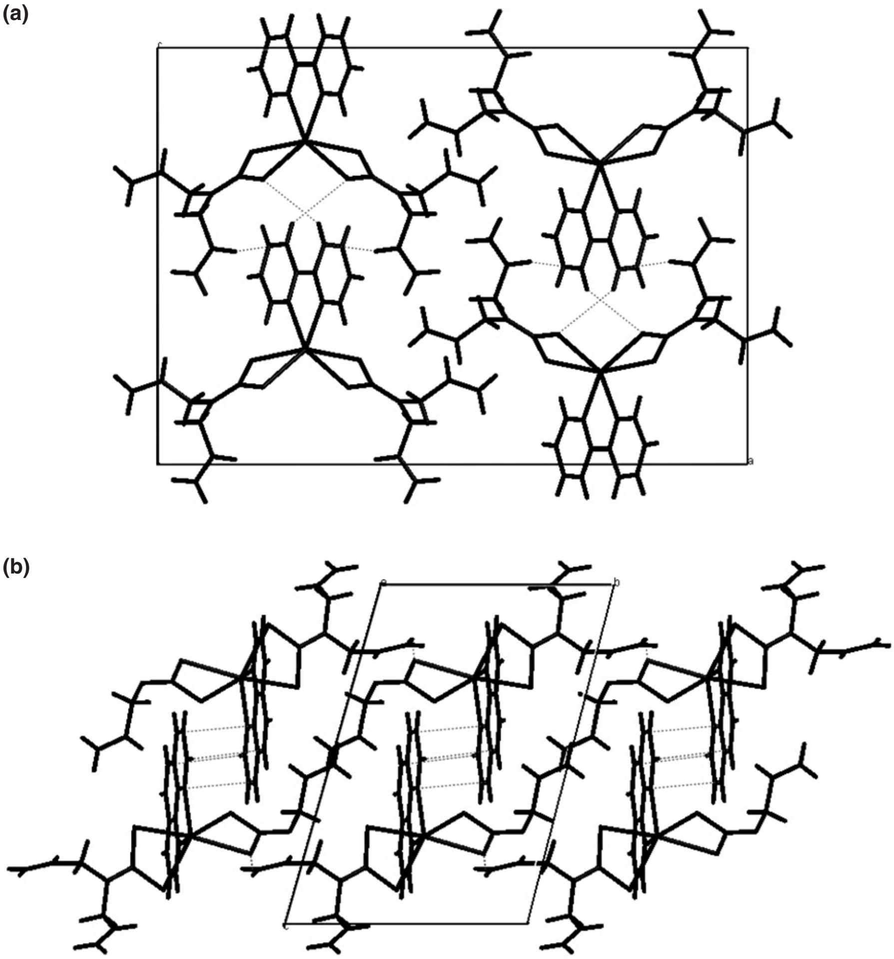

Figure 2. Packing diagrams showing: (a) infinite chains of **1** parallel to crystallographic *c*-axis and (b) chains of **2** parallel to *b*-axis (some of the disordered atoms in **2** omitted for clarity). Dotted lines represent the (π)C–H $\cdots$ S and (allyl)C–H $\cdots$ C(π) and $\pi \cdots \pi$ intermolecular interactions. Hydrogen atoms omitted for clarity.

Supplementary material

CCDC Numbers 696806 and 696807 contains the supplementary crystallographic data for this article. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, by e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44-(0)1223-336033.

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