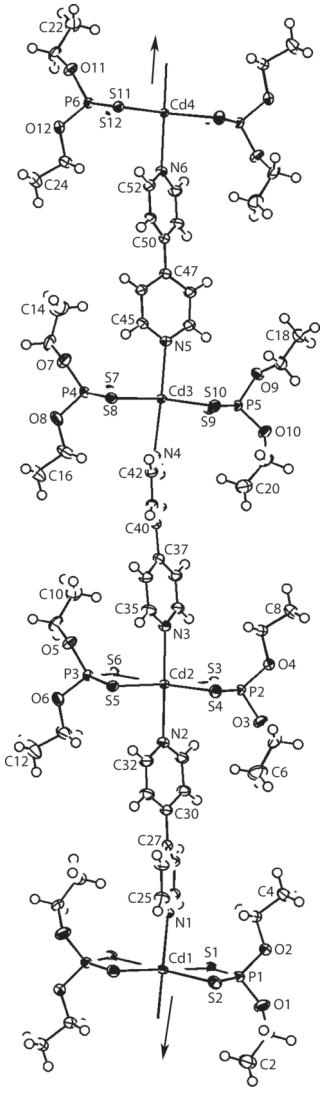
Yee Seng Tan and Edward R.T. Tiekink*

The pseudosymmetric low temperature polymorph of *catena*-poly[(μ_2-4,4′-bipyridyl-κ*N*,*N*′)-bis(*O*,*O*′-diethyldithiophosphato-κ*S*)-cadmium(II)], $\{C_{18}H_{28}CdN_2O_4P_2S_4\}_n$



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***Corresponding author: Edward R.T. Tiekink,** Research Centre for Crystalline Materials, School of Science and Technology, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia, e-mail: edwardt@sunway.edu.my. https://orcid.org/0000-0003-1401-1520

Yee Seng Tan: Research Centre for Crystalline Materials, School of Science and Technology, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia

Abstract

$C_{18}H_{28}CdN_2O_4P_2S_4$, triclinic, $P\bar{1}$ (no. 2), $a = 10.3735(1)$ Å, $b = 15.2066(2)$ Å, $c = 26.5231(2)$ Å, $\alpha = 85.178(1)°$, $\beta = 81.311(1)°$, $\gamma = 71.541(1)°$, $V = 3920.17(7)$ Å^3, $Z = 6$, $R_{gt}(F) = 0.0214$, $wR_{ref}(F^2) = 0.0556$, $T = 100(2)$ K.

CCDC no.: 1957394

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.17 × 0.12 mm
Wavelength:	Cu *Kα* radiation (1.54184 Å)
μ:	11.1 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	93196, 13999, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 13237
$N(param)_{refined}$:	863
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

The $Cd[S_2P(OEt)_2]_2$ precursor was prepared in high yield from the *in situ* reaction of $Cd(NO_3)_2 \cdot 4\ H_2O$ (Acros Organics; 15.42 g, 0.05 mol), EtOH (Merck; 12.25 mL, 0.21 mol), P_2S_5 (Sigma-Aldrich; 11.11 g, 0.05 mol) and 50% *w/w* NaOH solution (Merck; 8.80 mL, 0.11 mol). The title compound was obtained by mixing a suspension of this precursor (0.50 g, 1.04 mmol) and 4,4′-bipyridine (Merck; 0.17 g, 1.09 mmol) in dimethylformamide (Merck; 5 mL), followed by stirring for 30 min. at 373 K. The solution was filtered and the filtrate was collected in a sample vial containing acetonitrile (Merck; 1 mL). Yellow blocks formed after one day. Yield: 0.31 g, (46.7%, based on $Cd[S_2P(OEt)_2]_2$). **M.pt** (Stuart SMP 30 Melting point apparatus): 441.2–442.8 K. **IR** (Bruker Vertex 70 V equipped with Platinum ATR from 400 to 80 cm^{-1}): 1161(w) ν(C—O); 1011(s) ν(P—O); 651(s) $\nu(P—S)_{asym}$; 526(m) $\nu(P—S)_{sym}$; 294(m) ν(Zn—S); 380(w) ν(Zn—N).

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cd1	1.5000	1.5000	1.0000	0.01370(4)
Cd2	0.99420(2)	1.00774(2)	0.82902(2)	0.01264(4)
Cd3	0.48610(2)	0.50816(2)	0.66753(2)	0.01261(4)
Cd4	0.0000	0.0000	0.5000	0.01143(4)
S1	1.75458(4)	1.39011(3)	0.97086(2)	0.01671(8)
S2	1.57426(4)	1.58190(3)	0.91043(2)	0.01844(8)
S3	1.24404(4)	0.89418(2)	0.79515(2)	0.01407(7)
S4	1.05640(4)	1.08708(3)	0.73771(2)	0.01660(8)
S5	0.74364(4)	1.11995(2)	0.86411(2)	0.01433(8)
S6	0.92630(4)	0.92253(3)	0.91902(2)	0.01681(8)
S7	0.40848(4)	0.43106(3)	0.75742(2)	0.01660(8)
S8	0.23302(4)	0.62640(2)	0.69826(2)	0.01414(7)
S9	0.74970(4)	0.40599(2)	0.64378(2)	0.01585(8)
S10	0.56458(4)	0.58649(2)	0.57677(2)	0.01529(8)
S11	−0.25303(3)	0.11972(2)	0.52146(2)	0.01302(7)
S12	−0.09398(4)	−0.06757(3)	0.59040(2)	0.01622(8)
P1	1.74614(4)	1.47611(3)	0.90928(2)	0.01594(8)
P2	1.23319(4)	0.98531(3)	0.73605(2)	0.01319(8)
P3	0.75004(4)	1.02437(3)	0.92047(2)	0.01323(8)
P4	0.23685(4)	0.53738(3)	0.75789(2)	0.01405(8)
P5	0.73483(4)	0.47971(3)	0.57818(2)	0.01306(8)
P6	−0.26432(4)	0.03654(3)	0.58310(2)	0.01269(8)
O1	1.88007(12)	1.50737(8)	0.90042(5)	0.0281(3)
O2	1.77512(11)	1.41986(8)	0.85841(4)	0.0194(2)
O3	1.36172(11)	1.02159(8)	0.73289(4)	0.0194(2)
O4	1.26937(10)	0.93314(7)	0.68345(4)	0.0172(2)
O5	0.62149(11)	0.98987(7)	0.91951(4)	0.0191(2)
O6	0.70919(11)	1.07153(8)	0.97464(4)	0.0202(2)
O7	0.10438(11)	0.50591(8)	0.76055(4)	0.0224(2)
O8	0.20335(12)	0.59014(8)	0.81030(4)	0.0217(2)
O9	0.75048(10)	0.40605(7)	0.53653(4)	0.0170(2)
O10	0.86847(11)	0.51008(7)	0.55949(4)	0.0204(2)
O11	−0.39348(10)	0.00284(7)	0.58138(4)	0.0183(2)
O12	−0.31358(11)	0.09511(7)	0.63343(4)	0.0186(2)
N1	1.41047(13)	1.40314(9)	0.95834(5)	0.0149(3)
N2	1.10313(13)	1.10200(9)	0.86709(5)	0.0159(3)
N3	0.88472(13)	0.91414(9)	0.79173(5)	0.0155(3)
N4	0.56824(13)	0.60897(9)	0.71062(5)	0.0155(3)
N5	0.38850(13)	0.41550(9)	0.62524(5)	0.0151(3)
N6	0.09556(13)	0.10237(9)	0.53759(5)	0.0144(3)
C1[a]	1.8900(2)	1.59983(14)	0.89223(8)	0.0206(6)
H1A[a]	1.8080	1.6407	0.8778	0.025*
H1B[a]	1.9717	1.5990	0.8673	0.025*
C2[a]	1.9008(3)	1.63750(18)	0.94127(10)	0.0343(7)
H2A[a]	1.8184	1.6403	0.9655	0.051*
H2B[a]	1.9093	1.7000	0.9348	0.051*
H2C[a]	1.9817	1.5969	0.9556	0.051*
C1′[b]	1.8849(7)	1.5718(5)	0.9376(3)	0.0216(19)*
H1C[b]	1.9303	1.5371	0.9667	0.026*
H1D[b]	1.7908	1.6096	0.9509	0.026*
C2′[b]	1.9625(9)	1.6320(6)	0.9115(3)	0.029(2)*
H2D[b]	1.9780	1.6703	0.9364	0.043*
H2E[b]	1.9104	1.6722	0.8858	0.043*
H2F[b]	2.0509	1.5937	0.8947	0.043*
C3	1.67726(16)	1.37370(11)	0.84969(6)	0.0201(3)
H3A	1.5865	1.4202	0.8471	0.024*
H3B	1.6671	1.3298	0.8786	0.024*
C4	1.72805(17)	1.32226(11)	0.80105(6)	0.0206(3)
H4A	1.7366	1.3662	0.7725	0.031*
H4B	1.6629	1.2908	0.7951	0.031*
H4C	1.8178	1.2763	0.8038	0.031*
C5	1.36800(17)	1.10798(12)	0.70569(6)	0.0251(4)
H5A	1.2886	1.1335	0.6865	0.030*
H5B	1.4530	1.0963	0.6811	0.030*
C6	1.3660(2)	1.17591(13)	0.74389(8)	0.0387(4)
H6A	1.2792	1.1900	0.7668	0.058*
H6B	1.3752	1.2331	0.7260	0.058*
H6C	1.4424	1.1490	0.7639	0.058*
C7	1.17944(16)	0.88240(11)	0.67220(6)	0.0208(3)
H7A	1.0851	0.9251	0.6717	0.025*
H7B	1.1770	0.8331	0.6988	0.025*
C8	1.23364(17)	0.84036(11)	0.62106(6)	0.0217(3)
H8A	1.2323	0.8898	0.5948	0.033*
H8B	1.1761	0.8043	0.6135	0.033*
H8C	1.3279	0.7996	0.6216	0.033*
C9	0.60966(18)	0.90446(11)	0.94661(6)	0.0227(3)
H9A	0.5211	0.9173	0.9692	0.027*
H9B	0.6846	0.8792	0.9680	0.027*
C10	0.6182(2)	0.83547(12)	0.90801(7)	0.0316(4)
H10A	0.5460	0.8619	0.8861	0.047*
H10B	0.6061	0.7788	0.9256	0.047*
H10C	0.7080	0.8206	0.8870	0.047*
C11	0.80059(18)	1.11678(14)	0.99069(7)	0.0295(4)
H11A	0.8064	1.1694	0.9668	0.035*
H11B	0.8938	1.0722	0.9908	0.035*
C12	0.74375(19)	1.15086(14)	1.04332(7)	0.0344(4)
H12A	0.6510	1.1942	1.0430	0.052*
H12B	0.8026	1.1826	1.0546	0.052*
H12C	0.7405	1.0981	1.0668	0.052*
C13	0.08984(18)	0.42154(12)	0.78799(6)	0.0248(4)
H13A	0.0194	0.4376	0.8182	0.030*
H13B	0.1779	0.3844	0.7997	0.030*
C14	0.0482(2)	0.36684(13)	0.75249(7)	0.0349(4)
H14A	−0.0339	0.4062	0.7384	0.052*
H14B	0.0286	0.3132	0.7712	0.052*
H14C	0.1229	0.3453	0.7246	0.052*
C15	0.30080(19)	0.63438(13)	0.82192(7)	0.0279(4)
H15A	0.3195	0.6760	0.7929	0.033*
H15B	0.3883	0.5867	0.8277	0.033*
C16	0.2403(2)	0.68926(12)	0.86909(6)	0.0332(4)
H16A	0.1532	0.7358	0.8633	0.050*
H16B	0.3040	0.7204	0.8769	0.050*
H16C	0.2244	0.6474	0.8978	0.050*
C17	0.73606(16)	0.43716(11)	0.48346(5)	0.0203(3)
H17A	0.7965	0.4756	0.4712	0.024*
H17B	0.6402	0.4749	0.4804	0.024*
C18	0.77553(16)	0.35233(11)	0.45246(6)	0.0202(3)
H18A	0.8707	0.3157	0.4556	0.030*
H18B	0.7667	0.3710	0.4166	0.030*
H18C	0.7150	0.3149	0.4649	0.030*
C19	0.87354(17)	0.60431(11)	0.56071(6)	0.0227(3)
H19A	0.7893	0.6482	0.5492	0.027*
H19B	0.9528	0.6113	0.5367	0.027*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C20	0.88595(18)	0.62833(13)	0.61323(7)	0.0331(4)
H20A	0.8044	0.6258	0.6366	0.050*
H20B	0.8939	0.6910	0.6121	0.050*
H20C	0.9676	0.5838	0.6252	0.050*
C21	−0.41148(16)	−0.07922(11)	0.61057(6)	0.0207(3)
H21A	−0.5034	−0.0635	0.6309	0.025*
H21B	−0.3418	−0.1017	0.6343	0.025*
C22	−0.39642(19)	−0.15352(11)	0.57427(7)	0.0287(4)
H22A	−0.4644	−0.1304	0.5504	0.043*
H22B	−0.4110	−0.2082	0.5935	0.043*
H22C	−0.3040	−0.1704	0.5552	0.043*
C23	−0.22312(17)	0.14232(12)	0.64848(6)	0.0239(3)
H23A	−0.2114	0.1904	0.6222	0.029*
H23B	−0.1318	0.0972	0.6519	0.029*
C24	−0.28638(19)	0.18625(12)	0.69846(6)	0.0265(4)
H24A	−0.3760	0.2315	0.6946	0.040*
H24B	−0.2265	0.2177	0.7093	0.040*
H24C	−0.2980	0.1383	0.7242	0.040*
C25	1.29635(16)	1.43635(10)	0.93601(6)	0.0189(3)
H25	1.2556	1.5017	0.9332	0.023*
C26	1.23473(16)	1.38024(10)	0.91682(6)	0.0183(3)
H26	1.1536	1.4071	0.9013	0.022*
C27	1.29227(15)	1.28403(10)	0.92030(5)	0.0139(3)
C28	1.41259(15)	1.24951(10)	0.94286(5)	0.0145(3)
H28	1.4567	1.1845	0.9456	0.017*
C29	1.46699(15)	1.31065(10)	0.96118(5)	0.0153(3)
H29	1.5486	1.2859	0.9766	0.018*
C30	1.22807(15)	1.22111(10)	0.90173(5)	0.0147(3)
C31	1.08718(16)	1.24792(11)	0.89998(6)	0.0201(3)
H31	1.0307	1.3076	0.9106	0.024*
C32	1.03005(16)	1.18723(11)	0.88276(6)	0.0199(3)
H32	0.9338	1.2069	0.8820	0.024*
C33	1.23860(15)	1.07632(10)	0.86864(6)	0.0185(3)
H33	1.2925	1.0165	0.8574	0.022*
C34	1.30425(15)	1.13229(10)	0.88567(6)	0.0185(3)
H34	1.4005	1.1104	0.8864	0.022*
C35	0.75204(16)	0.94409(10)	0.78487(6)	0.0180(3)
H35	0.7023	1.0074	0.7905	0.022*
C36	0.68364(15)	0.88783(10)	0.77009(6)	0.0174(3)
H36	0.5897	0.9126	0.7655	0.021*
C37	0.75411(15)	0.79405(10)	0.76200(5)	0.0148(3)
C38	0.89275(16)	0.76309(11)	0.76879(6)	0.0177(3)
H38	0.9455	0.7002	0.7634	0.021*
C39	0.95226(16)	0.82423(11)	0.78335(6)	0.0177(3)
H39	1.0465	0.8016	0.7877	0.021*
C40	0.68703(15)	0.73058(10)	0.74606(5)	0.0141(3)
C41	0.57448(15)	0.76317(10)	0.71928(5)	0.0143(3)
H41	0.5356	0.8279	0.7127	0.017*
C42	0.51964(15)	0.70092(10)	0.70238(5)	0.0153(3)
H42	0.4434	0.7246	0.6839	0.018*
C43	0.67309(16)	0.57796(11)	0.73796(6)	0.0212(3)
H43	0.7071	0.5131	0.7453	0.025*
C44	0.73455(16)	0.63493(10)	0.75613(6)	0.0208(3)
H44	0.8088	0.6092	0.7754	0.025*
C45	0.25348(15)	0.42772(10)	0.63383(5)	0.0166(3)
H45	0.1968	0.4786	0.6534	0.020*
C46	0.19205(15)	0.37032(10)	0.61571(5)	0.0163(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H46	0.0955	0.3826	0.6225	0.020*
C47	0.27323(15)	0.29416(10)	0.58737(5)	0.0140(3)
C48	0.41351(15)	0.28243(10)	0.57759(6)	0.0179(3)
H48	0.4726	0.2326	0.5578	0.021*
C49	0.46629(15)	0.34360(10)	0.59683(6)	0.0180(3)
H49	0.5621	0.3344	0.5896	0.022*
C50	0.21231(15)	0.22850(10)	0.56971(5)	0.0134(3)
C51	0.07757(15)	0.25666(10)	0.55870(5)	0.0149(3)
H51	0.0227	0.3196	0.5619	0.018*
C52	0.02439(15)	0.19246(10)	0.54304(5)	0.0153(3)
H52	−0.0676	0.2132	0.5357	0.018*
C53	0.22560(15)	0.07549(10)	0.54801(6)	0.0171(3)
H53	0.2782	0.0122	0.5441	0.021*
C54	0.28700(15)	0.13476(10)	0.56401(6)	0.0165(3)
H54	0.3791	0.1121	0.5711	0.020*

[a]Occupancy: 0.777(4), [b]Occupancy: 0.223(4).

Experimental details

The C-bound H atoms were geometrically placed (C—H = 0.95–0.99 Å) and refined as riding with $U_{iso}(H) = 1.2–1.5U_{eq}(C)$. The C1-ethyl group was disordered over two positions. From refinement, the major component had a site occupancy factor of 0.777(4). The major component was refined with anisotropic displacement parameters.

Comment

In continuation of systematic studies into the generation of coordination polymers of the zinc-triad elements [5–7], a new low-temperature (100 K) polymorph of $\{Zn[S_2P(OEt)_2]_2(4,4'\text{-bipy})\}_n$ was described recently [8]; 4,4′-bipy is 4,4′-bipyridine. In common with the high temperature (296 K) form [9], the structure crystallised as a one-dimensional chain with a zig-zag topology. The particularly interesting feature of the 100 K structure was the presence of four independent repeat units in the asymmetric unit compared with two repeat units in the 296 K form. In the present contribution, the crystal and molecular structures of a new polymorph (100 K) of the cadmium analogoue, $\{Cd[S_2P(OEt)_2]_2(4,4'\text{-bipy})\}_n$, (I), is described and compared with 293 K form [10].

The asymmetric unit of (I) is shown in the figure (70% probability displacement ellipsoids; the minor component of the disordered ethyl group is omitted) and comprises four independent $Cd[S_2P(OEt)_2]_2(4,4'\text{-bipy})$ repeat units, two, with the Cd1- and Cd4-containing entities, having the cadmium atom located on a centre of inversion, and two, with the Cd2- and Cd3-containing entities, in general positions. The full repeat unit containing the Cd1 atom is generated by the application of symmetry operation (i) $3-x$, $3-y$, $2-z$, and that for the Cd4 atom by the application

of (ii) $-x$, $-y$, $1-z$. The coordination geometries for the Cd1–Cd4 atoms are closely related being based on a *trans*-N_2S_4 donor set defined by four sulphur atoms, derived from two chelating dithiophosphate ligands, and two pyridyl-nitrogen atoms derived from two bridging 4,4′-bipy ligands. The coordination geometries are each based on an octahedron. The dithiophosphate ligands are effectively symmetrically chelating with the range of Cd—S bond lengths being 2.6777(4) Å, for Cd1—S1, to 2.7246(4) Å, for Cd2—S6. The most symmetrically bound dithiophosphate ligands coordinate the Cd2 (Cd2—S3, S4 = 2.6888(4) and 2.6957(4) Å) and Cd4 (Cd4—S11, S12 = 2.6879(3) and 2.6960(4) Å) atoms, and the least symmetrically bound ligands are connected to the Cd1 (Cd1—S1, S2 = 2.6777(4) and 2.7104(4) Å) and Cd3 (Cd3—S9, S10 = 2.6910(4) and 2.7232(4) Å) atoms. These observations indicate there are no correlations in the magnitudes of the Cd—S bonds and the symmetry of the specific repeat unit. Consistent with the observed symmetric mode of coordination of the dithiophosphate ligands is the observation that the associated P—S bonds fall in a narrow range, i.e. 1.9846(5) Å, for P3—S6, to 1.9993(5) Å, for P1—S1. The three independent 4,4′-bipy ligands each lie in general positions and display relatively similar twists as seen in the sequence of N1-/N2-pyridyl, N3-/N4-pyridyl and N5-/N6-pyridyl dihedral angles of 27.42(7), 26.26(7) and 30.16(7)°, respectively. Each of the 4,4′-bipy ligands forms disparate Cd—N bonds lengths [Cd1—N2 = 2.3881(13) Å & Cd2—N2 = 2.4423(13) Å, Cd2—N3 = 2.4277(13) Å & Cd3—N4 = 2.4080(12) Å and Cd3—N5 = 2.4043(12) Å & Cd4—N6 = 2.4365(12) Å].

As indicated in the figure, the repeat units comprising the asymmetric unit in (I) generate a one-dimensional coordination polymer with a linear topology. A linear coordination geometry is also found in the room temperature polymorph where the independent cadmium atom is located on a centre of inversion [10]. Analogous linear coordination polymers are also found in $\{Cd[S_2P(OR)_2]_2(4,4'\text{-bipy})\}_n$, for R = iPr and cyclohexyl [11] but, when R = Me [12], a zig-zag chain is observed instead, mimicking the zinc(II) analogue [13]. The structural chemistry of bipyridyl-like adducts of the zinc-triad elements is capricious [5] as is that of their homoleptic precursors [14].

In the crystal, the coordination polymers are orientated along the [3 3 1] direction and are connected laterally by methylene-C—H···O(ethoxy) interactions [C5—H5b···O12^{iii}: H5b···O12^{iii} = 2.55 Å, C5b···O12^{iii} = 3.516(2) Å with angle at H5b = 164°, C9—H9a···O6^{iv}: H9a···O6^{iv} = 2.58 Å, C9···O6^{iv} = 3.565(2) Å with angle at H9a = 172°, C13—H13a···O1^{v}: H13a···O1^{v} = 2.54 Å, C13···O1^{v} = 3.497(2) Å with angle at H13a = 163° and C21—H21a···O4^{v}: H21a···O4^{v} = 2.56 Å, C21···O4^{v} = 3.531(2) Å with angle at H21a = 166° for (iii) $2+x$, $1+y$, z, (iv) $1-x$, $2-y$, $2-z$ and (v) $-2+x$, $-1+y$, z]. The three-dimensional architecture is consolidated by pyridyl-C—H···O(ethoxy) interactions [C54—H54···O11^{vi}: H54···O11^{vi} = 2.46 Å, C54···O11^{vi} = 3.3642(19) Å with angle at H54 = 160°].

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