

**William B. Welte and
 Edward R. Tiekink***

Department of Chemistry, The University of
 Texas at San Antonio, One UTSA Circle, San
 Antonio, Texas 78249-0698, USA

Correspondence e-mail:
 edward.tiekink@utsa.edu

Key indicators

Single-crystal X-ray study
 T = 150 K
 Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
 R factor = 0.037
 wR factor = 0.094
 Data-to-parameter ratio = 21.3

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

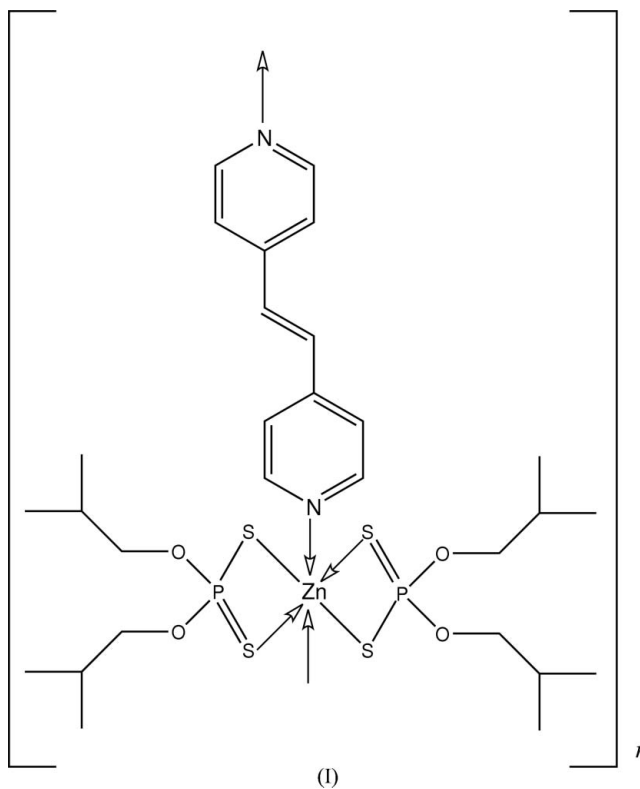
catena-Poly[[bis(O,O'-diisobutyl dithiophosphato- κ^2S,S')zinc(II)]- μ -1,2-di-4-pyridylethylene- $\kappa^2N:N'$]

Received 28 July 2006
 Accepted 30 July 2006

The polymeric title compound, $[\text{Zn}(\text{C}_8\text{H}_{17}\text{O}_2\text{PS}_2)_2(\text{C}_{12}\text{H}_{12}\text{N}_2)]_n$ or $[\text{Zn}\{\text{S}_2\text{P}(\text{O}^i\text{Bu})_2\}_2\{\text{NC}_5\text{H}_4\text{C}(\text{H})=\text{C}(\text{H})\text{C}_5\text{H}_4\text{N}\}]_n$, has the Zn atom within a distorted octahedral N_2S_4 geometry; the polymer topology is a straight chain. The Zn atom is located on a centre of inversion and the bridging bipyridine ligand is situated about another centre of inversion.

Comment

Polymer formation and, once formed, polymer topology of bipyridine-type ligand adducts of zinc and cadmium dithiophosphates has been shown to be dependent upon the steric bulk of remote R groups in the dithiophosphate ligands as well as the coordinating potential of the bipyridine-type ligands (e.g. Lai *et al.*, 2004*a,b*; Lai & Tiekink, 2004; Chen *et al.*, 2006; Tiekink, 2006). The title compound, (I), was investigated in continuation of these studies.



The Zn atom in (I) (Fig. 1) is situated on a crystallographic centre of inversion and the 1,2-di-4-pyridylethylene ligand is similarly disposed about another centre of inversion. An N_2S_4 donor set defines an approximate octahedral coordination geometry with two slightly asymmetrically coordinating

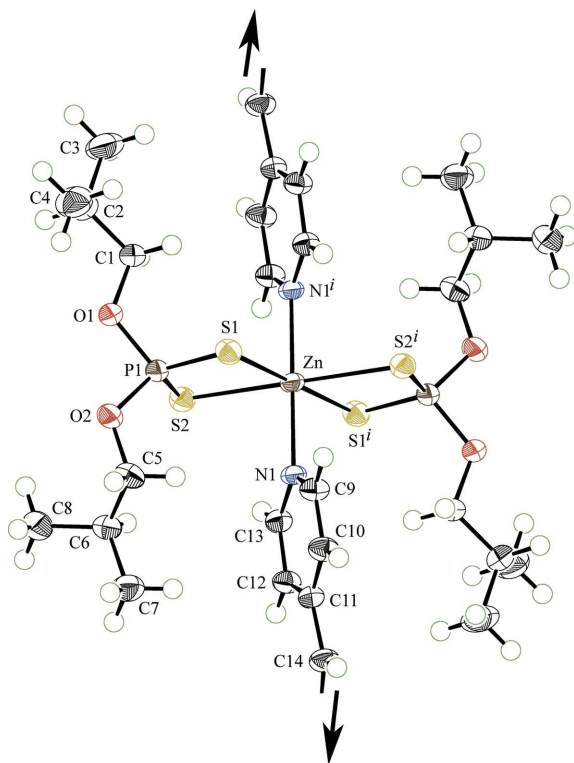


Figure 1
Part of the polymeric structure of (I). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $-x, -y, -z$.]

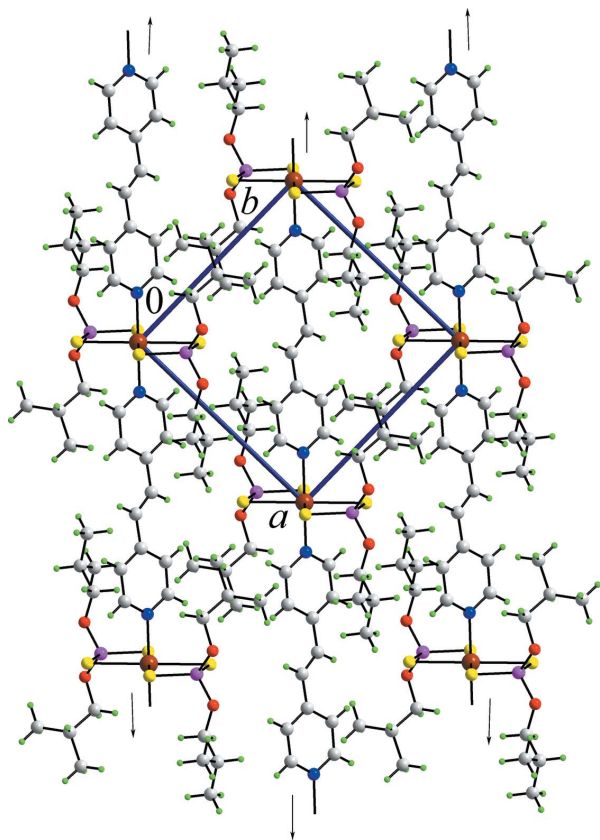


Figure 2
Adjacent straight-chain polymers in the structure of (I). Colour code: Zn orange, S yellow, P pink, O red, N blue, C grey and H green.

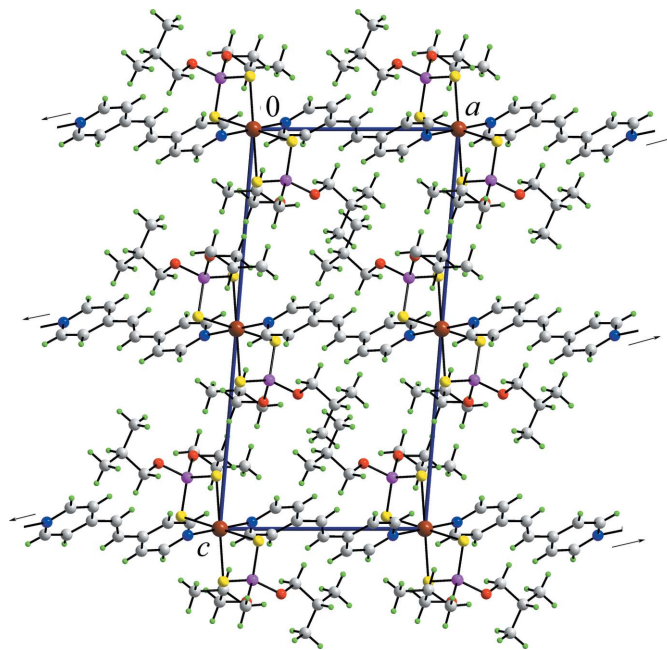


Figure 3
The packing of (I), viewed down the b axis. The colour code is as for Fig. 2.

dithiophosphate ligands occupying the equatorial plane (Table 1). The topology of the resulting polymer is of a straight chain, propagating along $[110]$; from symmetry, the 1,2-di-4-pyridylethylene ligand is planar. As seen from Fig. 2, the adjacent chains are shifted so that the successive ZnS_4 equatorial planes lie between ethylene residues. This arrangement allows for the formation of $\text{C14}\cdots\text{H14}\cdots\text{S1}^{\text{ii}}$ interactions, albeit weak, with $\text{H14}\cdots\text{S1}^{\text{ii}} = 3.20 \text{ \AA}$, $\text{C14}\cdots\text{S1}^{\text{ii}} = 3.905 (1) \text{ \AA}$ and $\text{C14}\text{—H14}\cdots\text{S1}^{\text{ii}} = 132^\circ$ [symmetry code: (ii) $1 + x, y, z$]. Layers thus formed stacked along the b -axis direction (Fig. 3) and are separated by hydrophobic interactions.

The polymeric structure of (I) is isomorphous with the recently reported 1,2-di-4-pyridylethane derivative (Lai *et al.*, 2004b) and resembles closely the straight chain polymers found in both of the cyclohexyl analogues (Lai *et al.*, 2004a), which arise owing to the steric bulk of the remote R groups in the dithiophosphate ligands.

Experimental

The title compound was prepared by refluxing the parent zinc dithiophosphate with 1,2-di-4-pyridylethylene (Aldrich) using a literature procedure (Lai *et al.*, 2004a). Colourless crystals were isolated by the slow evaporation of an acetonitrile/ CHCl_3 (1:1) solution of the compound (m.p. 422–426 K). ^1H NMR (CDCl_3): δ 0.95 (24H, d , $J = 6.69 \text{ Hz}$, CH_3), 2.00 (4H, *nonet*, $J = 6.69 \text{ Hz}$, CHCH_3), 3.87 [8H, dd , $J = 6.77 \text{ Hz}$, $J(^{31}\text{P}\text{—}^1\text{H}) = 8.31 \text{ Hz}$, CH_2], 8.87 (4H, d , $J = 6.35 \text{ Hz}$, CH—N), 7.57 (4H, d , $J = 6.35 \text{ Hz}$, CH—CH—N), 7.31 (2H, s , C=CH). ^{13}C NMR (CDCl_3): δ 19.0 (CH_3), 29.1 [$\text{CH}(\text{—CH}_3)$], 73.8 (OCH_2), 122.2 (C=C), 131.5 (CH—C_q), 145.2 (C_q), 150.3 (CH—N). ^{31}P (CDCl_3): δ 111.6. IR (KBr disk): $\nu(\text{C—O})$ 1176 (w), $\nu(\text{P—O})$ 952 (s), $\nu(\text{P—S})_{\text{asym}}$ 674 (s), $\nu(\text{P—S})_{\text{sym}}$ 553 (s) cm^{-1} . The most significant observation in the TGA (Shimadzu Thermogravimetric Analyser) was a distinct weight loss between 468.8 and 498.2 K, corresponding to loss of 1,2-di-4-pyridylethylene.

Crystal data

[Zn(C ₈ H ₁₇ O ₂ PS ₂) ₂ (C ₁₂ H ₁₂ N ₂)]	Z = 2
<i>M_r</i> = 730.22	<i>D_x</i> = 1.393 Mg m ⁻³
Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 9.735 (2) Å	<i>μ</i> = 1.07 mm ⁻¹
<i>b</i> = 9.428 (2) Å	<i>T</i> = 150 (2) K
<i>c</i> = 19.035 (5) Å	Block, colourless
<i>β</i> = 94.797 (7)°	0.40 × 0.15 × 0.10 mm
<i>V</i> = 1741.0 (7) Å ³	

Data collection

Rigaku AFC12K/SATURN724 diffractometer	43504 measured reflections
<i>ω</i> scans	3981 independent reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	3749 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.662, <i>T</i> _{max} = 1 (expected range = 0.595–0.898)	<i>R</i> _{int} = 0.052
	<i>θ</i> _{max} = 27.5°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.1222P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.094$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.10	$\Delta\rho_{\text{max}} = 0.70 \text{ e } \text{\AA}^{-3}$
3981 reflections	$\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$
187 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Zn–S1	2.5345 (8)	S1–P1	1.9933 (8)
Zn–S2	2.6144 (6)	S2–P1	1.9908 (8)
Zn–N1	2.1349 (15)		
S1–Zn–S2	80.036 (17)	Zn–S1–P1	84.79 (2)
S1–Zn–N1	89.80 (4)	Zn–S2–P1	82.72 (2)
S2–Zn–N1	90.11 (5)		

H atoms were included in the riding-model approximation, with C–H = 0.95–0.99 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by the departmental research grant AX-0026 from The Robert A. Welch Foundation. Cheminova is also thanked for the gift of the dithiophosphate ligand used in this study.

References

- Altomare, A., Casciarano, M., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Chen, D., Lai, C. S. & Tiekink, E. R. T. (2006). *CrystEngComm*, **8**, 51–58. Crystal Impact (2006). *DIAMOND*. Version 3.1. Crystal Impact GbR, Bonn, Germany.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Lai, C. S., Liu, S. & Tiekink, E. R. T. (2004a). *CrystEngComm*, **6**, 221–226.
- Lai, C. S., Liu, S. & Tiekink, E. R. T. (2004b). *Acta Cryst.* **E60**, m1005–m1007.
- Lai, C. S. & Tiekink, E. R. T. (2004). *CrystEngComm*, **6**, 593–605.
- Rigaku/MS (2005). *CrystalClear* User Manual, Rigaku/MS Inc., The Woodlands, Texas, USA.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Tiekink, E. R. T. (2006). *Frontiers in Crystal Engineering*, edited by E. R. T. Tiekink & J. J. Vittal, pp. 117–134. Chichester: John Wiley & Sons Ltd.