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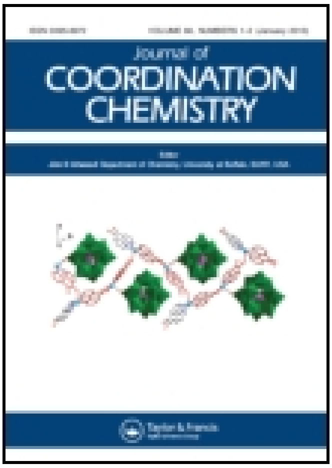

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Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Synthesis and spectroscopic characterization of toluene-3,4-dithiolatoarsenic(III)-O,O′-ditolyl/alkylene dithiophosphate compounds: crystal structure of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$]

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Accepted author version posted online: 10 Mar 2014.Published online: 01 Apr 2014.

To cite this article: Seema Maheshwari, Kavita Bundela & Krishan Gopal Ojha (2014) Synthesis and spectroscopic characterization of toluene-3,4-dithiolatoarsenic(III)-O,O′-ditolyl/alkylene dithiophosphate compounds: crystal structure of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$], Journal of Coordination Chemistry, 67:6, 1088-1096, DOI: 10.1080/00958972.2014.899356

To link to this article: http://dx.doi.org/10.1080/00958972.2014.899356

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Journal of Coordination Chemistry, 2014
Vol. 67, No. 6, 1088–1096, http://dx.doi.org/10.1080/00958972.2014.899356

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Synthesis and spectroscopic characterization of toluene-3,4-dithiolatoarsenic(III)-*O*,*O*'-ditolyl/alkylene dithiophosphate compounds: crystal structure of $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$

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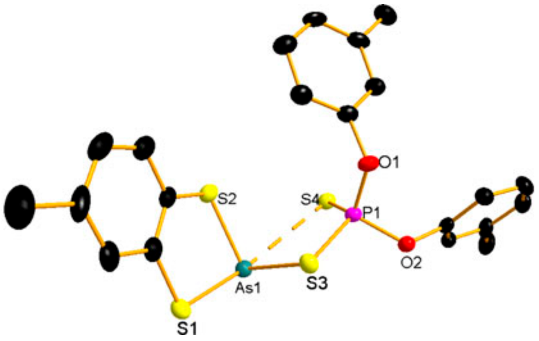
(*Received 8 November 2013; accepted 31 January 2014*)



Toluene-3,4-dithiolate complexes, $[CH_3C_6H_3S_2As\{S_2P(OR)_2\}]$ and $[CH_3C_6H_3S_2AsS_2POGO]$ (R = *o*-, *m*-, *p*-C_6H_4Me; G = $–CH_2C(CH_3)_2CH_2–$, $–CHCH_3CH_2(CH_3)_2C–$), have been synthesized by reaction of toluene-3,4-dithiolatoarsenic(III) chloride $[C_6H_3(CH_3)S_2AsCl]$ with ammonium salts of the corresponding *O*,*O*'-ditolyl/alkylene dithiophosphate by refluxing in benzene followed by stirring. These complexes have been characterized by elemental analysis and IR, ^1H, and ^{31}P NMR spectroscopy. Single-crystal X-ray diffraction analysis of $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$ revealed distorted trigonal-bipyramidal geometry with an anisobidentate chelating mode of the ditolyl moiety in these complexes.

Keywords: Toluene-3,4-dithiolatoarsenic(III) derivatives; Dithiophosphates; Crystal structure; ^1H NMR; ^{31}P NMR

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1. Introduction

Arsenic, antimony, and bismuth halides function as very weak Lewis acids towards thioethers [1] and have great affinity for anionic dithiolato [2–14] ligands. These complexes exhibit remarkable diversity in their coordination patterns because these complexes imitate unpredictable structural behavior due to the presence of a lone pair of electrons on the arsenic, small ligand bite, and molecular associations. The dithiolato ligands can behave as monodentate [8, 9], bidentate [6–10], and bridging [14]. A number of arsenic(III) complexes with these ligands have been set forth and some have also been characterized by single-crystal X-ray structural analysis [4–7]. In the last few decades, a range of dithiolato ligands with group 15 metal complexes have been studied with *O,O*'-dialkyl/ditolyl [4–13], alkylene [15, 16], and mixed ligand [17–19] dithiophosphates. Arsenic thiolates such as British anti-lewisite have been used in chelation therapy for heavy metal poisoning which rely on the high affinity of arsenic for sulfur ligands [20]. Toluene-3,4-dithiol (H_2tdt) is an attractive choice as a potential S,S′-bidentate metal scavenger [21]. Although some of the chemistry of toluene-3,4-dithiolatoarsenic(III) compounds have been already explored, chemistry of heteroleptic toluene-3,4-dithiolatoarsenic(III) compounds with bulky dithiolato ligands are still in their infancy. The lack of single-crystal X-ray structural analysis of arsenic thiolates and their interesting structural behavior prompted us to explore the chemistry of toluene-3,4-dithiolatoarsenic(III) ditolyl/alkylene dithiophosphates.

2. Experimental

2.1. *Materials and methods*

Stringent precautions were taken to exclude atmospheric moisture throughout the experimental manipulations owing to hydrolyzation of the starting materials as well as the newly synthesized compounds. $CH_3C_6H_3(SH)_2$, P_2S_5, and As_2O_3 were purchased from E. Merck and used as received. $SOCl_2$, *o*-, *m*-, *p*-cresols, and solvents (benzene, n-hexane) (E. Merck) were distilled prior to use. Literature methods were used for the preparation of *O,O'*-ditolyl [22–24] and alkylene dithiophosphoric acids [25]. Ammonium salts of the dithiophosphoric acids were prepared by reaction of the parent acid with an equimolar amount of ammonia in benzene. Arsenic trichloride was prepared by reaction of arsenic trioxide and thionyl chloride [26]. Toluene-3,4-dithiolatoarsenic(III) chloride [27] was prepared by reaction of arsenic(III) chloride and toluene-3,4-dithiol in equimolar ratio (1 : 1) in anhydrous benzene.

2.2. *Analytical methods of physical measurements*

Sulfur was estimated gravimetrically as barium sulfate (Messenger's method) [28] and arsenic(III) was estimated iodometrically [28]. IR spectra were recorded on a Perkin-Elmer 577 spectrophotometer in KBr pellets from 350 to 4000 cm^{-1}. ^1H NMR and ^{31}P NMR spectra were recorded on a Bruker DRX-300 spectrometer in $CDCl_3$ solutions using TMS and H_3PO_4 as an internal and external standard, respectively.

X-ray diffraction analysis: a yellow thin plate of $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$ was mounted on a glass fiber. Data were collected on a Bruker-Nonius Kappa CCD area detector diffractometer. Cell refinement gave cell constants corresponding to a monoclinic cell whose dimensions are given in table 1 along with other experimental parameters. An absorption correction was also applied [29]. The structure was solved by direct methods [30] and was refined using the WinGX version [31] of SHELX-97 [32]. All non-hydrogen atoms were treated anisotropically.

2.3. *Synthesis of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$] (1)*

Benzene solution of (10 mL) toluene-3,4-dithiolatoarsenic(III) chloride (0.27 g, 1.02 mM) was added dropwise with constant stirring to benzene suspension of the ammonium salt of di-(*m*-tolyl)dithiophosphoric acid (0.33 g, 1.02 mM). The color of the reaction mixture was changed to yellow and then refluxed for ~5 h. Then the contents were brought to room temperature and ammonium chloride was separated by filtration under anhydrous conditions, and excess solvent was removed from the filtrate in vacuum yielding a pale yellow viscous oily liquid of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$] yellow crystals of which were obtained after several weeks by layering of hexane to the viscous liquid. Yield: 0.45 g, 82%. Anal. Calcd for $C_{21}H_{20}AsO_2PS_4$: As – 13.9%; S – 23.7%. Found: As – 13.5%; S – 22.9%, ^{31}P NMR ($CDCl_3$): 84.97 δ.

2.4. *Synthesis of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}o)_2\}$] (2)*

The aforesaid method gave yellow oily viscous liquid. Yield: 0.29 g, 79%, Anal. Calcd for $C_{21}H_{20}AsO_2PS_4$: As – 13.9%; S – 23.7% Found: As – 13.4%; S – 23.3%, ^{31}P NMR ($CDCl_3$): 83.29 δ.

Table 1. Crystal data and structure refinement for [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$].

Empirical formula	$C_{21}H_{20}AsO_2PS_4$
Formula weight	538.50
Temperature (K)	120(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	*P2(1)/n*
a (Å)	12.437(2)
b (Å)	9.719(2)
c (Å)	19.447(3)
β (°)	106.016(2)
Volume (Å^3)	2259.5(7)
Z	4
Density (Calcd), g/cm^3	1.583
Absorption coefficient (mm^{-1})	1.961
F (0 0 0)	1096
Crystal size (mm^3)	0.10 × 0.08 × 0.02
θ Range for data collection (°)	3.02–27.51
Index ranges	$-16 \le h \le 16$, $-12 \le k \le 12$, $-25 \le l \le 25$
Reflections collected	28,223
Independent reflections	5164 [R(int) = 0.0925]
Max. and min. transmission	0.9618 and 0.8280
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5164/0/262
Goodness-of-fit on F^2	1.148
Final *R* indices [$F^2 > 4\sigma(F^2)$]	$R1 = 0.0705$, $wR2 = 0.1204$
R indices (all data)	$R1 = 0.1110$, $wR2 = 0.1380$
Largest diff. peak and hole (e·Å^{-3})	0.613 and −0.618

2.5. *Synthesis of [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}p)_2\}$] (3)*

The aforesaid method gave yellow oily viscous liquid. Yield: 0.35 g, 88%, Anal. Calcd for $C_{21}H_{20}AsO_2PS_4$: As – 13.9%; S – 23.7%. Found: As – 13.5%; S – 22.9%, ^{31}P NMR ($CDCl_3$): 83.95 δ.

2.6. *Synthesis of [$CH_3C_6H_3S_2As\{S_2POCH_2C(CH_3)_2CH_2O\}$] (4)*

The aforesaid method gave yellow oily viscous liquid. Yield: 0.28 g, 85%, Anal. Calcd for $C_{12}H_{16}AsO_2PS_4$: As – 17.5%; S – 30.0%. Found: As – 16.9%; S – 29.2%, ^{31}P NMR ($CDCl_3$): 84.59 δ.

2.7. *Synthesis of [$CH_3C_6H_3S_2AsS_2POCHCH_3CH_2(CH_3O$] (5)*

The aforesaid method gave yellow oily viscous liquid. Yield: 0.91 g, 85%. Anal. Calcd for $C_{13}H_{18}AsO_2PS_4$: As – 17.0%; S – 29.0%. Found: As – 16.5%; S – 28.7%, ^{31}P NMR ($CDCl_3$): 84.22 δ.

3. Results and discussion

3.1. *Synthesis and characterization*

Toluene-3,4-dithiolatoarsenic(III) ditolyl/alkylene dithiophosphate complexes have been synthesized by reactions of toluene-3,4-dithiolatoarsenic(III) chloride with ammonium ditolyl/alkylene dithiophosphates in equimolar ratio in anhydrous benzene by refluxing the contents for ~5 h.

$$CH_3C_6H_3S_2AsCl + NH_4S_2P(OR)_2 \xrightarrow{\text{Refluxing}}$$

$$[CH_3C_6H_3S_2As\{S_2P(OR)_2\}] + NH_4Cl\downarrow$$

$$(R = o\text{-},\ m\text{-}, p\text{-}C_6H_4Me)$$

$$CH_3C_6H_3S_2AsCl + NH_4S_2POGO \xrightarrow{\text{Refluxing}}$$

$$[CH_3C_6H_3S_2AsS_2POGO] + NH_4Cl\downarrow$$

$$(R = o\text{-},\ m\text{-}, p\text{-}C_6H_4Me);\ G = -CH_2C(CH_3)_2CH_2-, -CHCH_3CH_2(CH_3)_2C-)$$

These derivatives are non-volatile yellow viscous liquids except [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$], yellow crystals of which were obtained after several weeks by layering of hexane to the viscous liquid, and all are soluble in common organic solvents like benzene, chloroform, and dichloromethane. The products were characterized by elemental analysis, IR, ^{1}H, ^{31}P NMR spectroscopy, and single-crystal X-ray diffraction analysis. Elemental analyses are consistent to the molecular formula of the complexes.

3.2. *Infrared spectra*

IR spectra (table 2) of the toluene-3,4-dithiolatoarsenic(III) ditolyl/alkylene dithiophosphate complexes have been measured from 350 to 4000 cm^{-1} and the important bands have been assigned on the basis of comparison with spectra of salts of *O,O'*-di(*o*-, *m*- or *p*-tolyl) [23, 24] and alkylene dithiophosphoric acids [25] and analogous compounds [33].

Two strong bands at 1040–1160 cm^{-1} and 808–851 cm^{-1} are assigned to [(P)–O–C] and [P–O–(C)] stretches of dithiophosphate, respectively, in **1**–**5**. A strong band due to (P=S) stretch present in the spectra of ammonium salts of ditolyldithiophosphoric acids is shifted to lower frequencies (625–668 cm^{-1}) in all these toluene-3,4-dithiolatoarsenic(III)ditolyl/alkylene dithiophosphate derivatives. This shift probably indicates bidentate chelation of ditolyldithiophosphate with arsenic(III). Bands of medium intensities present at 505–546 cm^{-1} may be ascribed to (P–S). Bands of medium to strong intensities at 681–740 cm^{-1} may be attributed to an asymmetrically trisubstituted benzene ring.

3.3. *^{1}H NMR spectra*

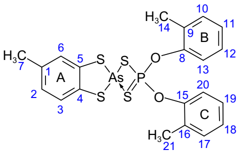
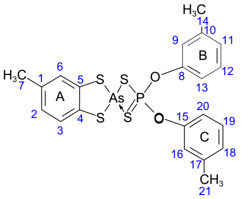
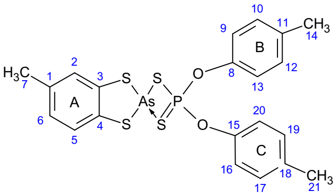
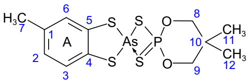
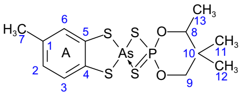
^{1}H NMR spectra of these compounds have been recorded in $CDCl_3$ solution at room temperature using TMS as an internal standard. ^{1}H NMR data (table 3) of these derivatives are similar to those of the corresponding salt of dithiophosphates, probably due to the large separation between arsenic and the hydrogens. In all these compounds ring methyl protons [CH_3 (7)] appear in the region 2.16–2.22 δ as a very sharp three proton singlet. A multiplet due to trisubstituted tolyl ring is at 6.75–7.49 δ. In the ditolyl dithiophosphate derivatives (**1**–**3**) (table 3), a very sharp six proton singlet at 2.25–2.32 δ is due to ditolyl methyl protons on various positions (*-o, -m, -p*). An eight proton multiplet at 7.11–7.28 δ is due to tolyl rings A and B.

In both the alkylene dithiophosphate derivatives (**4** and **5**) (table 3), a six proton singlet at 2.36 δ is due to two similar methyls [CH_3 (11 and 12)] of the ligand. Another four proton singlet at 3.82 δ and two proton singlet at 4.02 δ due to [OCH_2 (8 and 9)] and [OCH_2 (9)] in **4** and **5** (table 3), respectively, are observed. In addition, derivative **5** (table 3) and one proton singlet at 3.82 δ due to [(CH (8)] and a three proton doublet at 3.92–3.94 δ are

Table 2. IR spectroscopic data of toluene-3,4-dithiolatoarsenic(III) ditolyl/alkylenedithiophosphates.

S.No.	Compounds	ν(As–S)	ν[(P)–O–C]	ν[P–O–(C)]	ν(P=S)	ν(P–S)	Trisubstitution aromatic ring
1	$[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}o)_2\}]$	315	1114	808	665	531	712
2	$[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$	312	1142	851	667	546	731
3	$[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}p)_2\}]$	325	1160	819	625	511	740
4	$[CH_3C_6H_3S_2As\{S_2POCH_2C(CH_3)_2CH_2O\}]$	310	1054	811	668	505	681
5	$[CH_3C_6H_3S_2AsS_2P\{CH_3CHOCH_2C(CH_3)_2O\}]$	309	1040	812	630	531	698

Table 3. ^{1}H and ^{31}P NMR spectroscopic data of toluene-3,4-dithiolatoarsenic(III) ditolyl/alkylenedithiophosphates.

S.No.	Compounds	^{1}H Chemical shift (δ)	^{31}P Chemical shift (δ)
1		2.16 [(3H,s, ring CH_3 (7)] 2.32 [(6H,s, ring CH_3 (14) & CH_3 (21)] 6.92–7.10 [(3H,m, ring A] 7.11–7.23 [(8H,m, ring B & C]	83.29
2		2.18 [(3H,s, ring CH_3 (7)] 2.26 [(6H,s, ring CH_3 (14) & CH_3 (21)] 6.85–6.98 [(3H,m, ring A] 7.14–7.27 [(8H,m, ring B & C]	84.97
3		2.22 [(3H,s, ring CH_3 (7)] 2.25 [(6H,s, ring CH_3 (14) & CH_3 (21)] 6.85–6.96 [(3H,m, ring A] 7.12–7.28 [(8H,m, ring B & C]	83.95
4		2.17 [(3H,s, ring CH_3 (7)] 2.36 [(6H,s, ring CH_3 (11) & CH_3 (12)] 3.82 [4H,s,OCH_2 (8) & OCH_2 (9)] 6.93–7.36 [(3H,m, ring A]	84.59
5		2.17 [(3H,s, ring CH_3 (7)] 2.36 [(6H,s, ring CH_3 (11) & CH_3 (12)] 3.82 [1H,s,CH (8)] 3.92–3.94 [(3H,d, CH_3 (13) J = 8.0 Hz] 4.02 [(2H,s, OCH_2 (9)] 6.75–7.46 [(3H,m, ring A]	84.22

Note: s = singlet, d = doublet, m = multiplet.

observed due to methyl proton (13). Although NMR spectra confirm the compounds investigated, they provide no information about the details of the coordinated ligands. Therefore, single-crystal X-ray diffraction was performed to establish the structural details.

3.4. ^{31}P NMR spectra

On the basis of the analysis of proton decoupled ^{31}P NMR spectra of a number of the complexes, Glidewell concluded that complexes showing their NMR signal at 82–101 δ and less than 82 δ exhibit bidentate and monodentate coordination of ligands, respectively [34]. A sharp singlet at 83.29–84.97 δ of ^{31}P NMR spectra of all these toluene-3,4-dithiolatoarsenic ditolyl/alkylenedithiophosphate complexes suggests bidentate coordination of the dithiophosphate to arsenic (table 3). Occurrence of a singlet for **1**–**5** indicates equivalent phosphorous nuclei and purity of the compound.

3.5. *Crystal and molecular structure of* $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$

The single-crystal X-ray structure in the ORTEP diagram in figure 1 demonstrates anisobidentate coordination of the dithiophosphate.

The arsenic is attached with two sulfurs of toluene-3,4-dithiolate with the As(1)–S1 2.2425 Å and As(1)–S2 2.2311 Å bond lengths and one sulfur of dithiophosphate with As (1)–S3 2.3374 Å bond length (table 4). S4 is in close proximity to the arsenic, having As (1)–S4 3.2335 Å. This distance is still within the sum of the van der Waals radii of arsenic and sulfur of 3.74 Å, but this reflects the presence of the weak covalent bond. Hence, the geometry of the molecule is believed to be distorted trigonal bipyramidal where As(1)–S2 and As(1)–S3 are present as equatorial bonds and the stereochemically active lone pair of the electron is present on the remaining equatorial position of the trigonal-bipyramidal plane. S1 and S4 occupy axial positions making a *trans* basal angle of 161.89(54)° with

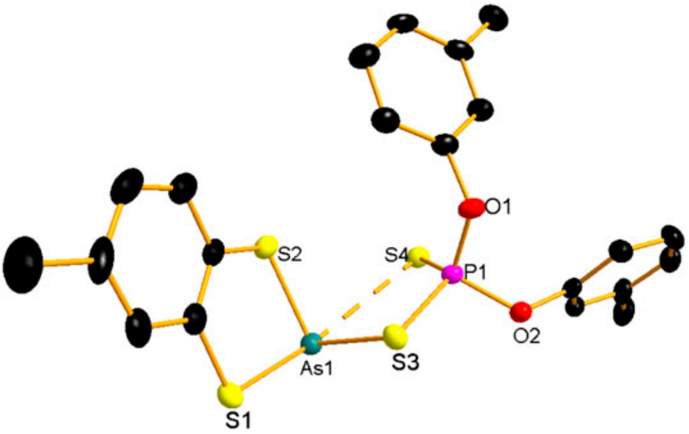


Figure 1. Structure of monomer unit of $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$. Hydrogens are omitted for clarity (teal = As; yellow = S; red = O; pink = P; black = C, see http://dx.doi.org/10.1080/00958972.2014.899356 for color version).

Table 4. Selected bond lengths [Å] and angles [°] for $[CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}]$.

Bond lengths	Angles	Bond lengths	Angles
As(1)–S(1)	2.2425(16)	S(2)–As(1)–S(1)	91.91(6)
As(1)–S(2)	2.2311(16)	S(2)–As(1)–S(3)	99.58(6)
As(1)–S(3)	2.3374(15)	S(1)–As(1)–S(3)	93.35(6)
S(3)–P(1)	2.055(2)	S(3)–As(1)–S(4)	72.93(44)
S(4)–P(1)	1.929(2)	C(1)–S(1)–As(1)	99.8(2)
S(1)–C(1)	1.778(6)	C(7)–S(2)–As(1)	100.0(2)
S(2)–C(7)	1.764(6)	P(1)–S(3)–As(1)	96.41(7)
O(2)–C(15)	1.407(6)	O(2)–P(1)–O(1)	100.2(2)
C(1)–C(2)	1.400(8)	O(2)–P(1)–S(4)	116.70(17)
C(2)–H(2)	0.9500	O(1)–P(1)–S(4)	115.35(16)
P(1)–O(2)	1.596(4)	O(2)–P(1)–S(3)	100.29(16)
P(1)–O(1)	1.599(4)	O(1)–P(1)–S(3)	105.49(16)
O(1)–C(8)	1.417(7)	S(4)–P(1)–S(3)	116.50(9)
C(1)–C(7)	1.385(8)	C(8)–O(1)–P(1)	124.2(3)
C(2)–C(3)	1.380(9)	C(15)–O(2)–P(1)	122.6(3)
C(3)–C(5)	1.406(10)	C(7)–C(1)–S(1)	120.6(4)

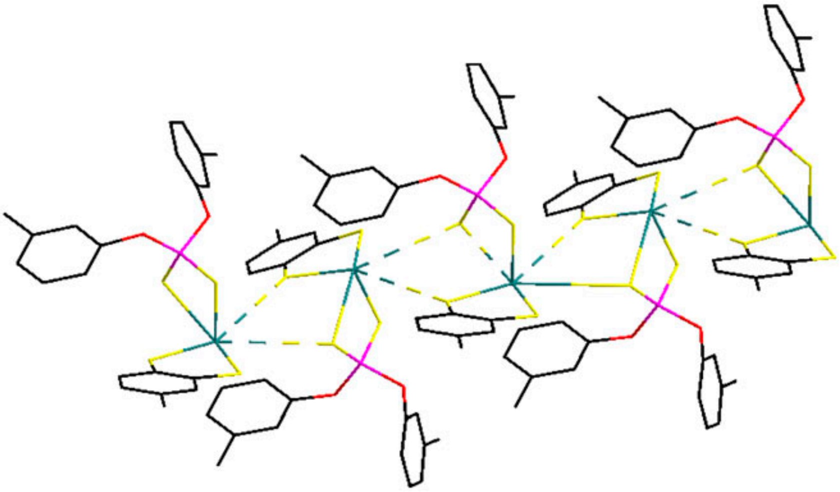


Figure 2. View of the chain polymer showing the long As–S contacts (teal = As; yellow = S; red = O; pink = P; black = C, see http://dx.doi.org/10.1080/00958972.2014.899356 for color version).

each other. S1 is 2.2366 Å above and S4 is 2.9922 Å below the plane defined by S2, S3, and arsenic.

In dithiophosphates, the P–S bond associated with strong As–S bonds is significantly longer (2.055(2) Å) than that associated with weaker As···S bonds (1.929(2) Å), as is plausible to maintain bond order conservation around sulfur. These monomeric units are very loosely assembled into chain polymer via bridging through sulfur of the neighboring unit (As···S = 3.762(3) and 3.873(3) Å) (figure 2). The value of the bite angle (72.937(44)°) between sulfurs of the ligand is similar to the other dithiophosphate complexes. Thus, the crystal structure provides confirmation about the structural conclusion based on IR, ^{1}H, and ^{31}P NMR spectroscopy.

4. Concluding comments

We have prepared and characterized [$CH_3C_6H_3S_2As\{S_2P(OR)_2\}$] and [$CH_3C_6H_3S_2As$-$S_2POGO$] (R = *o*-, *m*-, *p*-$C_6H_4Me$; G = –$CH_2C(CH_3)_2CH_2$–, –$CHCH_3CH_2(CH_3)_2C$–) IR, ^{1}H, and ^{31}P NMR spectral data and elemental analyses of these compounds are consistent with complexes containing bidentate ligands; single-crystal structure confirms distorted trigonal-bipyramidal geometry with an anisobidentate chelating mode of the ligand in these complexes.

Supplementary material

Supplementary data CCDC 905095 contain the supplementary crystallographic data for [$CH_3C_6H_3S_2As\{S_2P(OC_6H_4Me\text{-}m)_2\}$]. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44) 1223-336-033; or Email: deposit@ccdc.cam.ac.uk

Acknowledgments

We are thankful to SAIF, Panjab University, Panjab and CDRI, Lucknow for the ^{1}H and ^{31}P NMR spectral analyses. We also thank Dr W. Zhang, University of Southampton, Southampton, UK for assisting with the crystallographic data collection.

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