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# Bis[*O*-isopropyl (4-ethoxyphenyl)dithiophosphonato-$\kappa^2 S,S'$]lead(II)

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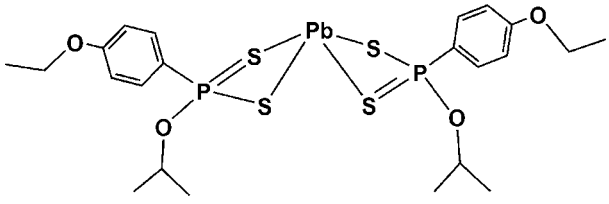
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.020; $wR$ factor = 0.045; data-to-parameter ratio = 23.7.

The title compound, $[Pb(C_{11}H_{16}O_2PS_2)_2]$, is a neutral four-coordinate mononuclear complex with a distorted square-pyramidal geometry of the $PbS_4$ core. The apical $Pb^{II}$ atom of each pyramid is 1.33059 (3) Å above the basal $S_4$ plane. The metal atom is surrounded by two chelating dithiophosphonate ligands binding through the S-donor atoms. The ligands are anisobidentate as the pyramid is comprised of Pb—S bond lengths that vary substantially [2.6999 (7), 2.7128 (6), 2.8877 (7) and 2.9472 (7) Å], clearly indicating two short and two longer bond lengths. The P—S bond lengths are also paired as shorter [1.9959 (9) and 1.9877 (8) Å] and slightly longer [2.0115 (9) and 2.0245 (9) Å], indicating an anisobidentate nature of the ligand whereby the shorter P—S bond has more double-bond character than the other. The S—Pb—S (chelating) bond angles range from 71.841 (18) to 72.692 (19)°, whilst the Pb—S—P bond angles range from 84.70 (3) to 90.51 (3)°.

## Related literature

For information on dithiophosphonate compounds, see: Van Zyl & Fackler (2000); Van Zyl (2010). For similar lead(II) dithiophosphonate complexes, see: Gray *et al.* (2003, 2004).




## Experimental

### Crystal data

$[Pb(C_{11}H_{16}O_2PS_2)_2]$
$M_r$ = 757.85
Triclinic, $P\overline{1}$
$a$ = 10.2092 (8) Å
$b$ = 11.7016 (10) Å
$c$ = 12.9915 (10) Å
$\alpha$ = 89.297 (2)°
$\beta$ = 85.016 (2)°
$\gamma$ = 69.034 (1)°
$V$ = 1443.5 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 6.27 mm$^{-1}$
$T$ = 173 K
0.16 × 0.14 × 0.11 mm

### Data collection

Bruker Kappa DUO APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.434, $T_{max}$ = 0.546
36710 measured reflections
7212 independent reflections
6564 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.037

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.045
$S$ = 1.03
7212 reflections
304 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.61 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.63 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2529).

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# supporting information

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# Bis[*O*-isopropyl (4-ethoxyphenyl)dithiophosphonato-$\kappa^2S,S'$]lead(II)

**Shirveen Sewpersad and Werner E. Van Zyl**

## S1. Comment

The phosphor-1,1,-dithiolate class of compounds is the heavier and softer congener of the more popular phosphonate derivatives. Several sub-categories are known which include the dithiophosphato $[S_2P(OR)_2]^-$, (*R* = typically alkyl), dithiophosphinato $[S_2PR_2]^-$ (*R* = alkyl or aryl), and dithiophosphonato $[S_2PR(OR')]^-$, (*R* = typically aryl or ferrocenyl, *R′* = alkyl) monoanionic ligands. The latter may be described as a hybrid of the former two, and are also much less developed. Lead dithiophosphonate complexes have only been developed during the past 10 years (Gray *et al.*, 2003; 2004) and the solid-state structure of the lead complexes deviate substantially from typical 4-coordinate transition-metals such as Ni(II), Cd(II), Hg(II) of the same ligand.

The title complex exhibits a structure that is built up of distorted square pyramids in dimeric pairs. The dimeric pair is characterised by intermolecular interactions between the Pb atom of one monomer with a S atom of adjacent unit, containing a Pb···S distance of 3.619 Å. Similar intermolecular interactions occur between the Pb atom of one monomeric unit and four carbon atoms in an adjacent aromatic ring in the Pb-Ar (Ar = aromatic ring) bond distance range of 3.421-3.597 Å. A related structure with a different alkoxy group was previously reported (Gray *et al.*, 2004), showing similar inter Pb···S and Pb···Ar interactions, and intermolecular Pb-S bond lenghts. It was postulated that the aforementioned Pb···Ar interactions stabilized the dimeric structure. The structure reported by Gray *et al.* (2004) adopts a similar molecular unit, the lead also resides in the centre of a distorted pyramid, 1.30 Å above the basal $S_4$ plane. General and convenient methods to prepare dithiophosphonate salt derivatives have been reported (Van Zyl & Fackler, 2000).

## S2. Experimental

A colorless methanol (40 ml) solution of $NH_4[S_2P(O^iPr)(4\text{-}C_6H_4OEt)]$ (1.010 g, 3.443 mmol) was prepared. Another solution of $Pb(NO_3)_2$ (0.570 g, 1.721 mmol) in deionized water (20 ml) was prepared, and added to the colorless solution with stirring over a period of 5 min. This resulted in a white precipitate, which proved to be the formation of the title complex. The precipitate was collected by vacuum filtration, washed with water (3 *x* 10 ml) and allowed to dry under vacuum for a period of 3 hrs, yielding a dry, free-flowing white powder. Colorless crystals suitable for X-ray analysis were grown by the slow diffusion of hexane into a dichloromethane solution of the title complex. Yield: 0.926 g, 71%. M.p. 397–398 K.

$^{31}$P NMR ($CDCl_3$): $\delta$ (p.p.m.): 94.20. $^1$H NMR ($CDCl_3$): $\delta$ (p.p.m.): 7.89(2*H*, dd, *o*-ArH), 6.89(2*H*, dd, *m*-ArH), 5.01(1*H*, d quart, CH), 4.03(2*H*, quart, $ArOCH_2$), 1.39(3*H*, t, $ArOCH_2CH_3$), 1.32(6*H*, d, $CH_3$). $^{13}$C NMR ($CDCl_3$): $\delta$ (p.p.m.): 161.97 (*p*-ArC), 133.32 (*ipso*-Ar—C), 131.57 (*m*-ArC), 114.32 (*o*-ArC), 71.34 (CH), 64.03($ArOCH_2$), 24.85 ($CH_3$), 14.91 ($ArOCH_2CH_3$).

## S3. Refinement

All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were placed in idealized positions and refined with geometrical constraints and constrained to ride on their parent atoms, with C–H = 0.95–1 00 Å,and $U_{iso}(H_{aryl})$ = 1.2×$U_{eq}C_{aryl}$ and $U_{iso}(H_{methyl})$ = 1.5×$U_{eq}C_{methyl}$.

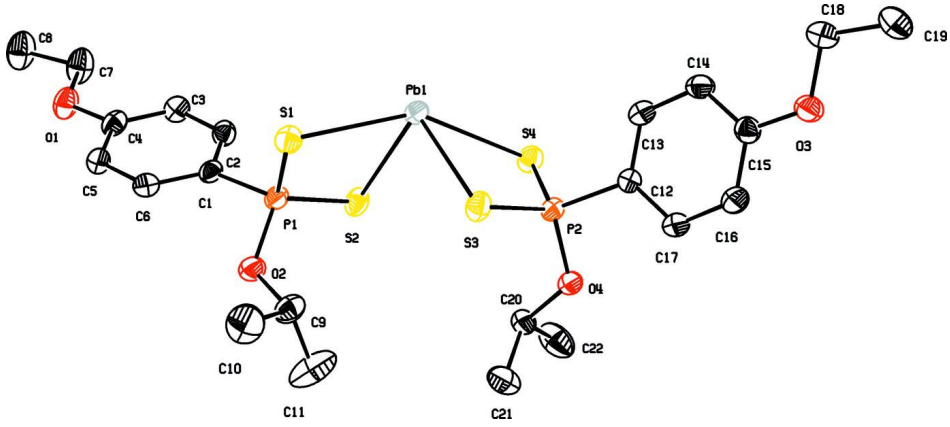


**Figure 1**

The molecular structure of the title complex with displacement ellipsoids shown at the 50% probability level. H-atoms have been omitted for clarity.

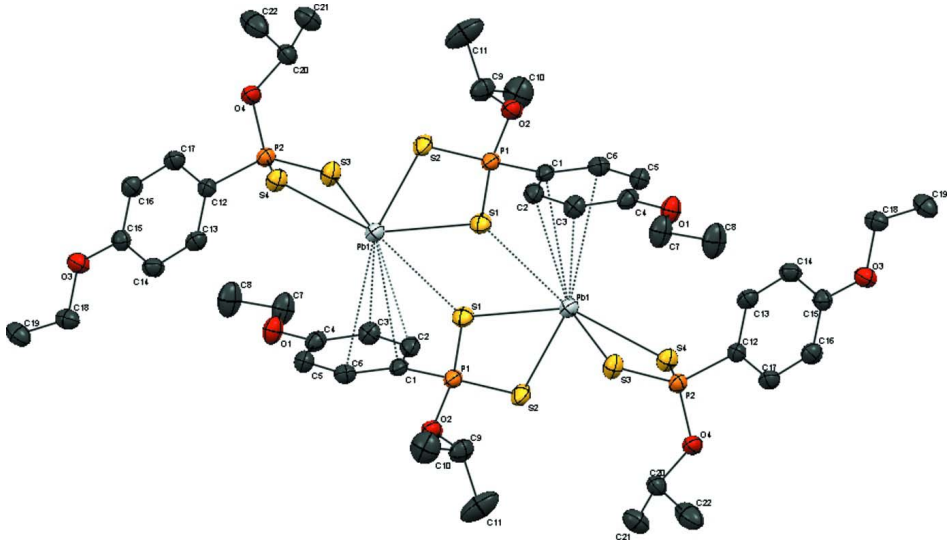

**Figure 2**

The molecular structure of a dimeric pair of the title complex showing intermolecular Pb•••S and Pb•••Ar interactions.

**Bis[*O*-isopropyl (4-ethoxyphenyl)dithiophosphonato- $\kappa^2S,S'$]lead(II)**

*Crystal data*

$[Pb(C_{11}H_{16}O_2PS_2)_2]$
$M_r$ = 757.85
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 10.2092 (8) Å
$b$ = 11.7016 (10) Å
$c$ = 12.9915 (10) Å
$\alpha$ = 89.297 (2)°
$\beta$ = 85.016 (2)°
$\gamma$ = 69.034 (1)°
$V$ = 1443.5 (2) Å$^3$
$Z$ = 2

$F(000)$ = 744
?e
$D_x$ = 1.744 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 36710 reflections

$\theta$ = 1.6–28.4°
$\mu$ = 6.27 mm$^{-1}$
$T$ = 173 K
Block, colourless
0.16 × 0.14 × 0.11 mm

*Data collection*

Bruker Kappa DUO APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.5° $\varphi$ scans and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.434, $T_{max}$ = 0.546

36710 measured reflections
7212 independent reflections
6564 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.037
$\theta_{max}$ = 28.4°, $\theta_{min}$ = 1.6°
$h$ = −13→13
$k$ = −15→15
$l$ = −17→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.045
$S$ = 1.03
7212 reflections
304 parameters
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 0.042P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.61 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.63 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Pb1 | 0.563161 (9) | 0.892734 (7) | 0.676272 (7) | 0.02692 (3) |
| S1 | 0.48642 (6) | 0.83844 (6) | 0.47888 (5) | 0.03000 (13) |
| S2 | 0.80447 (7) | 0.77787 (6) | 0.55716 (5) | 0.03150 (13) |
| S3 | 0.55200 (7) | 0.68031 (6) | 0.75505 (5) | 0.03173 (14) |
| S4 | 0.75140 (7) | 0.82120 (5) | 0.84420 (5) | 0.03020 (13) |
| P1 | 0.69313 (6) | 0.77422 (5) | 0.43769 (5) | 0.02499 (13) |
| P2 | 0.67269 (7) | 0.68943 (5) | 0.86809 (5) | 0.02494 (13) |
| O1 | 0.8185 (2) | 1.06377 (17) | 0.08990 (14) | 0.0368 (4) |
| O2 | 0.74190 (18) | 0.64074 (14) | 0.38776 (13) | 0.0314 (4) |
| O3 | 0.31553 (18) | 0.75446 (15) | 1.26420 (13) | 0.0315 (4) |
| O4 | 0.79180 (18) | 0.55982 (14) | 0.88024 (13) | 0.0299 (4) |
| C1 | 0.7390 (2) | 0.8566 (2) | 0.33187 (18) | 0.0244 (5) |

| | | | | |
|---|---|---|---|---|
| C2 | 0.7854 (2) | 0.9519 (2) | 0.35221 (19) | 0.0280 (5) |
| H2 | 0.7992 | 0.9676 | 0.4213 | 0.034* |
| C3 | 0.8118 (3) | 1.0243 (2) | 0.27399 (19) | 0.0295 (5) |
| H3 | 0.8416 | 1.0898 | 0.2894 | 0.035* |
| C4 | 0.7941 (3) | 1.0000 (2) | 0.17254 (19) | 0.0293 (5) |
| C5 | 0.7474 (3) | 0.9048 (2) | 0.15114 (19) | 0.0294 (5) |
| H5 | 0.7350 | 0.8885 | 0.0819 | 0.035* |
| C6 | 0.7191 (3) | 0.8344 (2) | 0.22993 (18) | 0.0276 (5) |
| H6 | 0.6861 | 0.7707 | 0.2148 | 0.033* |
| C7 | 0.8657 (3) | 1.1634 (3) | 0.1081 (2) | 0.0438 (7) |
| H7A | 0.9567 | 1.1329 | 0.1394 | 0.053* |
| H7B | 0.7958 | 1.2251 | 0.1558 | 0.053* |
| C8 | 0.8824 (4) | 1.2188 (3) | 0.0058 (2) | 0.0507 (8) |
| H8A | 0.9426 | 1.1547 | −0.0432 | 0.076* |
| H8B | 0.9257 | 1.2803 | 0.0139 | 0.076* |
| H8C | 0.7898 | 1.2578 | −0.0203 | 0.076* |
| C9 | 0.7074 (3) | 0.5436 (2) | 0.4444 (2) | 0.0361 (6) |
| H9 | 0.6554 | 0.5776 | 0.5125 | 0.043* |
| C10 | 0.6158 (4) | 0.5027 (3) | 0.3833 (3) | 0.0567 (9) |
| H10A | 0.5333 | 0.5735 | 0.3681 | 0.085* |
| H10B | 0.5850 | 0.4437 | 0.4230 | 0.085* |
| H10C | 0.6684 | 0.4639 | 0.3185 | 0.085* |
| C11 | 0.8454 (4) | 0.4454 (3) | 0.4627 (3) | 0.0702 (11) |
| H11A | 0.8952 | 0.4093 | 0.3962 | 0.105* |
| H11B | 0.8281 | 0.3817 | 0.5054 | 0.105* |
| H11C | 0.9029 | 0.4810 | 0.4983 | 0.105* |
| C12 | 0.5720 (3) | 0.7089 (2) | 0.99137 (18) | 0.0251 (5) |
| C13 | 0.4855 (3) | 0.8252 (2) | 1.02436 (19) | 0.0294 (5) |
| H13 | 0.4853 | 0.8933 | 0.9837 | 0.035* |
| C14 | 0.3992 (3) | 0.8444 (2) | 1.11533 (19) | 0.0297 (5) |
| H14 | 0.3414 | 0.9251 | 1.1376 | 0.036* |
| C15 | 0.3976 (3) | 0.7450 (2) | 1.17388 (18) | 0.0269 (5) |
| C16 | 0.4827 (3) | 0.6269 (2) | 1.14110 (19) | 0.0307 (5) |
| H16 | 0.4811 | 0.5588 | 1.1811 | 0.037* |
| C17 | 0.5694 (3) | 0.6088 (2) | 1.05057 (19) | 0.0297 (5) |
| H17 | 0.6275 | 0.5281 | 1.0283 | 0.036* |
| C18 | 0.2206 (3) | 0.8742 (2) | 1.2982 (2) | 0.0331 (6) |
| H18A | 0.1523 | 0.9098 | 1.2464 | 0.040* |
| H18B | 0.2738 | 0.9290 | 1.3069 | 0.040* |
| C19 | 0.1447 (3) | 0.8615 (2) | 1.3993 (2) | 0.0375 (6) |
| H19A | 0.0849 | 0.8141 | 1.3885 | 0.056* |
| H19B | 0.0861 | 0.9429 | 1.4273 | 0.056* |
| H19C | 0.2134 | 0.8193 | 1.4481 | 0.056* |
| C20 | 0.9080 (3) | 0.5123 (2) | 0.79867 (19) | 0.0305 (5) |
| H20 | 0.8964 | 0.5734 | 0.7425 | 0.037* |
| C21 | 0.9019 (3) | 0.3954 (2) | 0.7559 (2) | 0.0418 (7) |
| H21A | 0.9166 | 0.3347 | 0.8105 | 0.063* |
| H21B | 0.9756 | 0.3638 | 0.6990 | 0.063* |

| | | | | |
|---|---|---|---|---|
| H21C | 0.8095 | 0.4117 | 0.7304 | 0.063* |
| C22 | 1.0424 (3) | 0.4935 (3) | 0.8471 (3) | 0.0529 (8) |
| H22A | 1.0400 | 0.5717 | 0.8750 | 0.079* |
| H22B | 1.1225 | 0.4615 | 0.7948 | 0.079* |
| H22C | 1.0525 | 0.4349 | 0.9032 | 0.079* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pb1 | 0.02779 (5) | 0.02345 (5) | 0.02595 (5) | −0.00485 (3) | −0.00277 (4) | 0.00404 (3) |
| S1 | 0.0212 (3) | 0.0358 (3) | 0.0304 (3) | −0.0072 (2) | −0.0021 (2) | 0.0031 (2) |
| S2 | 0.0231 (3) | 0.0399 (3) | 0.0279 (3) | −0.0064 (2) | −0.0058 (2) | 0.0074 (3) |
| S3 | 0.0417 (4) | 0.0317 (3) | 0.0279 (3) | −0.0191 (3) | −0.0099 (3) | 0.0051 (2) |
| S4 | 0.0366 (4) | 0.0277 (3) | 0.0305 (3) | −0.0156 (3) | −0.0072 (3) | 0.0037 (2) |
| P1 | 0.0224 (3) | 0.0250 (3) | 0.0244 (3) | −0.0050 (2) | −0.0013 (2) | 0.0044 (2) |
| P2 | 0.0297 (3) | 0.0213 (3) | 0.0233 (3) | −0.0084 (2) | −0.0036 (2) | 0.0030 (2) |
| O1 | 0.0457 (11) | 0.0417 (10) | 0.0306 (10) | −0.0245 (9) | −0.0069 (8) | 0.0114 (8) |
| O2 | 0.0349 (10) | 0.0250 (8) | 0.0300 (9) | −0.0067 (7) | 0.0021 (8) | 0.0039 (7) |
| O3 | 0.0326 (10) | 0.0268 (8) | 0.0324 (10) | −0.0093 (7) | 0.0048 (8) | −0.0016 (7) |
| O4 | 0.0327 (10) | 0.0239 (8) | 0.0265 (9) | −0.0035 (7) | 0.0023 (7) | 0.0038 (7) |
| C1 | 0.0196 (11) | 0.0246 (11) | 0.0244 (12) | −0.0028 (8) | −0.0013 (9) | 0.0046 (9) |
| C2 | 0.0258 (13) | 0.0320 (12) | 0.0251 (12) | −0.0086 (10) | −0.0040 (10) | 0.0018 (9) |
| C3 | 0.0290 (13) | 0.0303 (12) | 0.0322 (13) | −0.0139 (10) | −0.0052 (11) | 0.0027 (10) |
| C4 | 0.0263 (13) | 0.0306 (12) | 0.0297 (13) | −0.0090 (10) | −0.0020 (10) | 0.0088 (10) |
| C5 | 0.0316 (14) | 0.0305 (12) | 0.0249 (12) | −0.0093 (10) | −0.0043 (10) | 0.0027 (10) |
| C6 | 0.0264 (13) | 0.0244 (11) | 0.0306 (13) | −0.0075 (9) | −0.0024 (10) | 0.0011 (9) |
| C7 | 0.0547 (19) | 0.0535 (17) | 0.0378 (16) | −0.0360 (15) | −0.0109 (14) | 0.0127 (13) |
| C8 | 0.070 (2) | 0.0589 (19) | 0.0415 (17) | −0.0446 (17) | −0.0103 (16) | 0.0164 (14) |
| C9 | 0.0384 (15) | 0.0304 (13) | 0.0401 (15) | −0.0135 (11) | −0.0029 (12) | 0.0108 (11) |
| C10 | 0.062 (2) | 0.057 (2) | 0.060 (2) | −0.0307 (17) | −0.0116 (18) | 0.0045 (16) |
| C11 | 0.062 (2) | 0.0476 (19) | 0.106 (3) | −0.0207 (17) | −0.034 (2) | 0.035 (2) |
| C12 | 0.0282 (13) | 0.0230 (10) | 0.0224 (11) | −0.0070 (9) | −0.0020 (10) | 0.0008 (9) |
| C13 | 0.0328 (14) | 0.0227 (11) | 0.0307 (13) | −0.0074 (10) | −0.0035 (11) | 0.0042 (9) |
| C14 | 0.0311 (13) | 0.0215 (11) | 0.0333 (13) | −0.0061 (9) | −0.0006 (11) | −0.0008 (9) |
| C15 | 0.0281 (13) | 0.0278 (11) | 0.0254 (12) | −0.0104 (9) | −0.0037 (10) | −0.0013 (9) |
| C16 | 0.0361 (14) | 0.0250 (11) | 0.0309 (13) | −0.0116 (10) | −0.0007 (11) | 0.0043 (10) |
| C17 | 0.0336 (14) | 0.0220 (11) | 0.0298 (13) | −0.0058 (9) | −0.0009 (11) | 0.0017 (9) |
| C18 | 0.0295 (14) | 0.0289 (12) | 0.0380 (15) | −0.0071 (10) | −0.0007 (11) | −0.0054 (10) |
| C19 | 0.0309 (14) | 0.0423 (14) | 0.0382 (15) | −0.0132 (11) | 0.0042 (12) | −0.0093 (12) |
| C20 | 0.0346 (14) | 0.0275 (12) | 0.0257 (12) | −0.0080 (10) | 0.0037 (11) | −0.0012 (9) |
| C21 | 0.0446 (17) | 0.0352 (14) | 0.0438 (16) | −0.0140 (12) | 0.0062 (13) | −0.0100 (12) |
| C22 | 0.0365 (17) | 0.065 (2) | 0.0524 (19) | −0.0132 (15) | 0.0009 (15) | −0.0208 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pb1—S2 | 2.6999 (7) | C9—C10 | 1.480 (4) |
| Pb1—S3 | 2.7128 (6) | C9—C11 | 1.502 (4) |
| Pb1—S1 | 2.8877 (7) | C9—H9 | 1.0000 |

| | | | |
|---|---|---|---|
| Pb1—S4 | 2.9472 (7) | C10—H10A | 0.9800 |
| S1—P1 | 1.9959 (9) | C10—H10B | 0.9800 |
| S2—P1 | 2.0115 (9) | C10—H10C | 0.9800 |
| S3—P2 | 2.0245 (9) | C11—H11A | 0.9800 |
| S4—P2 | 1.9877 (8) | C11—H11B | 0.9800 |
| P1—O2 | 1.5877 (17) | C11—H11C | 0.9800 |
| P1—C1 | 1.794 (2) | C12—C13 | 1.380 (3) |
| P2—O4 | 1.5848 (16) | C12—C17 | 1.400 (3) |
| P2—C12 | 1.797 (2) | C13—C14 | 1.381 (3) |
| O1—C4 | 1.356 (3) | C13—H13 | 0.9500 |
| O1—C7 | 1.441 (3) | C14—C15 | 1.386 (3) |
| O2—C9 | 1.476 (3) | C14—H14 | 0.9500 |
| O3—C15 | 1.363 (3) | C15—C16 | 1.392 (3) |
| O3—C18 | 1.437 (3) | C16—C17 | 1.379 (3) |
| O4—C20 | 1.471 (3) | C16—H16 | 0.9500 |
| C1—C2 | 1.394 (3) | C17—H17 | 0.9500 |
| C1—C6 | 1.399 (3) | C18—C19 | 1.500 (4) |
| C2—C3 | 1.384 (3) | C18—H18A | 0.9900 |
| C2—H2 | 0.9500 | C18—H18B | 0.9900 |
| C3—C4 | 1.392 (3) | C19—H19A | 0.9800 |
| C3—H3 | 0.9500 | C19—H19B | 0.9800 |
| C4—C5 | 1.398 (3) | C19—H19C | 0.9800 |
| C5—C6 | 1.382 (3) | C20—C22 | 1.503 (4) |
| C5—H5 | 0.9500 | C20—C21 | 1.507 (3) |
| C6—H6 | 0.9500 | C20—H20 | 1.0000 |
| C7—C8 | 1.495 (4) | C21—H21A | 0.9800 |
| C7—H7A | 0.9900 | C21—H21B | 0.9800 |
| C7—H7B | 0.9900 | C21—H21C | 0.9800 |
| C8—H8A | 0.9800 | C22—H22A | 0.9800 |
| C8—H8B | 0.9800 | C22—H22B | 0.9800 |
| C8—H8C | 0.9800 | C22—H22C | 0.9800 |

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|---|---|---|---|
| S2—Pb1—S3 | 93.09 (2) | C11—C9—H9 | 109.0 |
| S2—Pb1—S1 | 72.692 (19) | C9—C10—H10A | 109.5 |
| S3—Pb1—S1 | 91.516 (19) | C9—C10—H10B | 109.5 |
| S2—Pb1—S4 | 82.708 (19) | H10A—C10—H10B | 109.5 |
| S3—Pb1—S4 | 71.841 (18) | C9—C10—H10C | 109.5 |
| S1—Pb1—S4 | 149.566 (17) | H10A—C10—H10C | 109.5 |
| P1—S1—Pb1 | 85.24 (3) | H10B—C10—H10C | 109.5 |
| P1—S2—Pb1 | 90.16 (3) | C9—C11—H11A | 109.5 |
| P2—S3—Pb1 | 90.51 (3) | C9—C11—H11B | 109.5 |
| P2—S4—Pb1 | 84.70 (3) | H11A—C11—H11B | 109.5 |
| O2—P1—C1 | 100.90 (10) | C9—C11—H11C | 109.5 |
| O2—P1—S1 | 110.94 (7) | H11A—C11—H11C | 109.5 |
| C1—P1—S1 | 111.88 (8) | H11B—C11—H11C | 109.5 |
| O2—P1—S2 | 111.34 (7) | C13—C12—C17 | 119.0 (2) |
| C1—P1—S2 | 109.69 (8) | C13—C12—P2 | 118.89 (18) |
| S1—P1—S2 | 111.63 (4) | C17—C12—P2 | 121.87 (17) |

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| O4—P2—C12 | 101.33 (10) | C12—C13—C14 | 121.2 (2) |
| O4—P2—S4 | 112.17 (8) | C12—C13—H13 | 119.4 |
| C12—P2—S4 | 111.43 (8) | C14—C13—H13 | 119.4 |
| O4—P2—S3 | 109.88 (7) | C13—C14—C15 | 119.5 (2) |
| C12—P2—S3 | 109.56 (9) | C13—C14—H14 | 120.2 |
| S4—P2—S3 | 111.98 (4) | C15—C14—H14 | 120.2 |
| C4—O1—C7 | 118.0 (2) | O3—C15—C14 | 123.9 (2) |
| C9—O2—P1 | 119.79 (15) | O3—C15—C16 | 116.0 (2) |
| C15—O3—C18 | 117.87 (18) | C14—C15—C16 | 120.1 (2) |
| C20—O4—P2 | 119.72 (14) | C17—C16—C15 | 119.9 (2) |
| C2—C1—C6 | 118.8 (2) | C17—C16—H16 | 120.0 |
| C2—C1—P1 | 119.26 (18) | C15—C16—H16 | 120.0 |
| C6—C1—P1 | 121.73 (18) | C16—C17—C12 | 120.2 (2) |
| C3—C2—C1 | 121.4 (2) | C16—C17—H17 | 119.9 |
| C3—C2—H2 | 119.3 | C12—C17—H17 | 119.9 |
| C1—C2—H2 | 119.3 | O3—C18—C19 | 108.0 (2) |
| C2—C3—C4 | 119.3 (2) | O3—C18—H18A | 110.1 |
| C2—C3—H3 | 120.4 | C19—C18—H18A | 110.1 |
| C4—C3—H3 | 120.4 | O3—C18—H18B | 110.1 |
| O1—C4—C3 | 124.3 (2) | C19—C18—H18B | 110.1 |
| O1—C4—C5 | 115.9 (2) | H18A—C18—H18B | 108.4 |
| C3—C4—C5 | 119.8 (2) | C18—C19—H19A | 109.5 |
| C6—C5—C4 | 120.5 (2) | C18—C19—H19B | 109.5 |
| C6—C5—H5 | 119.8 | H19A—C19—H19B | 109.5 |
| C4—C5—H5 | 119.8 | C18—C19—H19C | 109.5 |
| C5—C6—C1 | 120.1 (2) | H19A—C19—H19C | 109.5 |
| C5—C6—H6 | 119.9 | H19B—C19—H19C | 109.5 |
| C1—C6—H6 | 119.9 | O4—C20—C22 | 107.2 (2) |
| O1—C7—C8 | 107.2 (2) | O4—C20—C21 | 107.8 (2) |
| O1—C7—H7A | 110.3 | C22—C20—C21 | 112.9 (2) |
| C8—C7—H7A | 110.3 | O4—C20—H20 | 109.6 |
| O1—C7—H7B | 110.3 | C22—C20—H20 | 109.6 |
| C8—C7—H7B | 110.3 | C21—C20—H20 | 109.6 |
| H7A—C7—H7B | 108.5 | C20—C21—H21A | 109.5 |
| C7—C8—H8A | 109.5 | C20—C21—H21B | 109.5 |
| C7—C8—H8B | 109.5 | H21A—C21—H21B | 109.5 |
| H8A—C8—H8B | 109.5 | C20—C21—H21C | 109.5 |
| C7—C8—H8C | 109.5 | H21A—C21—H21C | 109.5 |
| H8A—C8—H8C | 109.5 | H21B—C21—H21C | 109.5 |
| H8B—C8—H8C | 109.5 | C20—C22—H22A | 109.5 |
| O2—C9—C10 | 108.7 (2) | C20—C22—H22B | 109.5 |
| O2—C9—C11 | 106.4 (2) | H22A—C22—H22B | 109.5 |
| C10—C9—C11 | 114.4 (3) | C20—C22—H22C | 109.5 |
| O2—C9—H9 | 109.0 | H22A—C22—H22C | 109.5 |
| C10—C9—H9 | 109.0 | H22B—C22—H22C | 109.5 |
| | | | |
| S2—Pb1—S1—P1 | −3.39 (3) | C6—C1—C2—C3 | 0.0 (3) |
| S3—Pb1—S1—P1 | −96.16 (3) | P1—C1—C2—C3 | −175.01 (18) |

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| S4—Pb1—S1—P1 | −40.94 (5) | C1—C2—C3—C4 | −1.2 (4) |
| S3—Pb1—S2—P1 | 93.98 (3) | C7—O1—C4—C3 | −0.1 (4) |
| S1—Pb1—S2—P1 | 3.36 (3) | C7—O1—C4—C5 | 179.5 (2) |
| S4—Pb1—S2—P1 | 165.23 (3) | C2—C3—C4—O1 | −179.1 (2) |
| S2—Pb1—S3—P2 | 87.41 (3) | C2—C3—C4—C5 | 1.3 (4) |
| S1—Pb1—S3—P2 | 160.16 (3) | O1—C4—C5—C6 | −179.9 (2) |
| S4—Pb1—S3—P2 | 6.13 (3) | C3—C4—C5—C6 | −0.2 (4) |
| S2—Pb1—S4—P2 | −101.96 (3) | C4—C5—C6—C1 | −0.9 (4) |
| S3—Pb1—S4—P2 | −6.27 (3) | C2—C1—C6—C5 | 1.0 (3) |
| S1—Pb1—S4—P2 | −66.05 (4) | P1—C1—C6—C5 | 175.93 (18) |
| Pb1—S1—P1—O2 | 129.49 (7) | C4—O1—C7—C8 | −179.2 (2) |
| Pb1—S1—P1—C1 | −118.68 (9) | P1—O2—C9—C10 | 117.6 (2) |
| Pb1—S1—P1—S2 | 4.68 (3) | P1—O2—C9—C11 | −118.6 (2) |
| Pb1—S2—P1—O2 | −129.58 (7) | O4—P2—C12—C13 | 160.0 (2) |
| Pb1—S2—P1—C1 | 119.60 (8) | S4—P2—C12—C13 | 40.5 (2) |
| Pb1—S2—P1—S1 | −4.99 (4) | S3—P2—C12—C13 | −84.0 (2) |
| Pb1—S4—P2—O4 | 132.71 (7) | O4—P2—C12—C17 | −25.8 (2) |
| Pb1—S4—P2—C12 | −114.48 (9) | S4—P2—C12—C17 | −145.23 (19) |
| Pb1—S4—P2—S3 | 8.62 (4) | S3—P2—C12—C17 | 90.3 (2) |
| Pb1—S3—P2—O4 | −134.69 (7) | C17—C12—C13—C14 | 1.4 (4) |
| Pb1—S3—P2—C12 | 114.82 (8) | P2—C12—C13—C14 | 175.8 (2) |
| Pb1—S3—P2—S4 | −9.33 (4) | C12—C13—C14—C15 | −1.1 (4) |
| C1—P1—O2—C9 | −172.27 (18) | C18—O3—C15—C14 | 2.8 (3) |
| S1—P1—O2—C9 | −53.57 (19) | C18—O3—C15—C16 | −176.9 (2) |
| S2—P1—O2—C9 | 71.40 (18) | C13—C14—C15—O3 | −179.5 (2) |
| C12—P2—O4—C20 | −174.11 (17) | C13—C14—C15—C16 | 0.2 (4) |
| S4—P2—O4—C20 | −55.17 (18) | O3—C15—C16—C17 | −179.9 (2) |
| S3—P2—O4—C20 | 70.07 (17) | C14—C15—C16—C17 | 0.4 (4) |
| O2—P1—C1—C2 | −145.78 (18) | C15—C16—C17—C12 | 0.0 (4) |
| S1—P1—C1—C2 | 96.21 (19) | C13—C12—C17—C16 | −0.8 (4) |
| S2—P1—C1—C2 | −28.2 (2) | P2—C12—C17—C16 | −175.1 (2) |
| O2—P1—C1—C6 | 39.3 (2) | C15—O3—C18—C19 | −178.9 (2) |
| S1—P1—C1—C6 | −78.67 (19) | P2—O4—C20—C22 | 119.9 (2) |
| S2—P1—C1—C6 | 156.88 (17) | P2—O4—C20—C21 | −118.3 (2) |