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Monoclinic modification of 1,2-bis-(diphenylselenophosphinoyl)ethane

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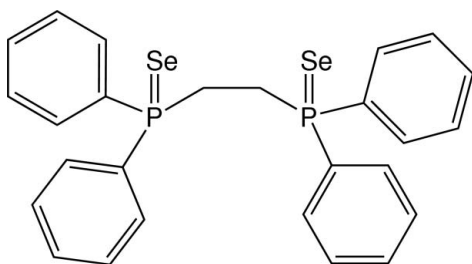
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.052; wR factor = 0.068; data-to-parameter ratio = 17.4.

The complete molecule of the title compound, $\text{C}_{26}\text{H}_{24}\text{P}_2\text{Se}_2$, is generated by crystallographic 2-fold symmetry, with the rotation axis bisecting the central C—C bond. The dihedral angle between the terminal aromatic rings is 74.1 (1)°.

Related literature

For the synthesis and related compounds, see: Lobana (1992); Lobana *et al.* (2007). For the triclinic modification, whose molecule lies on a center-of-inversion, see: Risto *et al.* (2007).



Experimental

Crystal data

$\text{C}_{26}\text{H}_{24}\text{P}_2\text{Se}_2$

$M_r = 556.31$

Monoclinic, $C2/c$
 $a = 15.828$ (2) Å
 $b = 9.2057$ (19) Å
 $c = 19.697$ (3) Å
 $\beta = 121.654$ (8)°
 $V = 2443.0$ (7) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.17$ mm⁻¹
 $T = 295$ (2) K
 $0.20 \times 0.12 \times 0.11$ mm

Data collection

Stoe IPDS-II diffractometer
 Absorption correction: numerical
 (X -SHAPE; Stoe & Cie, 2007)
 $T_{\min} = 0.525$, $T_{\max} = 0.701$

8656 measured reflections
 2361 independent reflections
 1835 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.068$
 $S = 1.15$
 2361 reflections

136 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.49$ e Å⁻³
 $\Delta\rho_{\min} = -0.41$ e Å⁻³

Table 1

Selected bond lengths (Å).

P1—Se1	2.0979 (9)	P1—C2	1.809 (3)
P1—C1	1.822 (3)	P1—C8	1.812 (3)

Data collection: X -AREA (Stoe & Cie, 2007); cell refinement: X -AREA; data reduction: X -RED; program(s) used to solve structure: $SHELXS97$ (Sheldrick, 2008); program(s) used to refine structure: $SHELXL97$ (Sheldrick, 2008); molecular graphics: $DIAMOND$ (Brandenburg, 2001); software used to prepare material for publication: $PLATON$ (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2502).

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supplementary materials

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Monoclinic modification of 1,2-bis(diphenylselenophosphinoyl)ethane

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Comment

Organophosphorous derivatives are an important class of ligands due to their potentially applications as selective homogeneous catalysis. Among this group, the increasing interest in diphosphines and their calcogenide derivatives arises from their interesting coordination properties, extractive metallurgy and catalytic properties (Lobana, 1992; Lobana, *et al.* 2007). Therefore, we prompted to synthesis a new derivative of calcogenide organophosphorous ligands in order to investigate its structure.

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. The title compound was previously published in triclinic crystal system with two half-molecules in the asymmetric unit, having inversion symmetry for both molecules (Risto, *et al.* 2007). Here we presents the structure in monoclinic system in which, the asymmetric unit is one half-molecule. Two Diphenylphosphinoselenoyl, (PPh₂)Se, are connected through an ethane group. Two phenyl rings (C2—C7 & C8—C13) in the asymmetric are located with dihedral angle of 74.1 (1)^o. Weak inter-molecular hydrogen bonds (C10—H10[⋯]Se1ⁱⁱ, 3.751 (4) Å) are present between neighboring molecules, assembling the molecules into a three dimensional network.

Experimental

Diphenylphosphinoethane (dppe) was prepared according to literature (Lobana, 1992). To a mixture of 3.98 g (0.01 mol) dppe in 300 ml of dried chloroform was added 1.58 g (0.02 mol) of red selenium. The reaction mixture was refluxed overnight and filtered the unreacted Se out. The resulting solution was evaporated under reduced pressure. The crystals suitable for crystallography were obtained by recrystallization from chloroform-acetonitrile (1:1).

Refinement

All H atoms were placed in calculated positions and constrained to ride on their parent atoms ($U_{\text{iso}}(\text{H}) = 1.2(\text{C})$), with C—H = 0.93 and 0.97 Å, for aromatic and methylene, respectively.

Figures

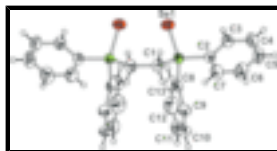


Fig. 1. Molecular structure of (I), with 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

1,2-bis(diphenylselenophosphinoyl)ethane

Crystal data

$C_{26}H_{24}P_2Se_2$

$M_r = 556.31$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 15.828 (2) \text{ \AA}$

$b = 9.2057 (19) \text{ \AA}$

$c = 19.697 (3) \text{ \AA}$

$\beta = 121.654 (8)^\circ$

$V = 2443.0 (7) \text{ \AA}^3$

$Z = 4$

$F_{000} = 1112$

$D_x = 1.513 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8656 reflections

$\theta = 2.5\text{--}29.5^\circ$

$\mu = 3.17 \text{ mm}^{-1}$

$T = 295 (2) \text{ K}$

Rod, colorless

$0.20 \times 0.12 \times 0.11 \text{ mm}$

Data collection

Stoe IPDS-II
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 295(2) \text{ K}$

φ oscillation scans

Absorption correction: numerical
(XSHAPE; Stoe & Cie, 2007)

$T_{\min} = 0.525$, $T_{\max} = 0.701$

8656 measured reflections

2361 independent reflections

1835 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.072$

$\theta_{\max} = 26.0^\circ$

$\theta_{\min} = 2.4^\circ$

$h = -19 \rightarrow 16$

$k = -11 \rightarrow 11$

$l = -23 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.068$

$S = 1.15$

2361 reflections

136 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

$$w = 1/[\sigma^2(F_o^2) + (0.0166P)^2 + 3.9345P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.49 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.41 \text{ e \AA}^{-3}$

Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.44808 (3)	1.02455 (3)	0.10565 (2)	0.04679 (13)
P1	0.52862 (6)	0.83149 (8)	0.15258 (5)	0.02995 (18)
C1	0.5482 (2)	0.7912 (4)	0.25038 (19)	0.0396 (7)
H1A	0.5795	0.6968	0.2678	0.048*
H1B	0.5928	0.8630	0.2884	0.048*
C2	0.6516 (2)	0.8360 (3)	0.16755 (18)	0.0333 (7)
C3	0.6699 (2)	0.9256 (3)	0.1207 (2)	0.0427 (8)
H3	0.6202	0.9872	0.0843	0.051*
C4	0.7609 (3)	0.9253 (4)	0.1271 (2)	0.0563 (10)
H4	0.7720	0.9856	0.0946	0.068*
C5	0.8346 (3)	0.8363 (4)	0.1810 (3)	0.0600 (11)
H5	0.8960	0.8361	0.1853	0.072*
C6	0.8188 (3)	0.7477 (5)	0.2286 (3)	0.0661 (11)
H6	0.8694	0.6873	0.2654	0.079*
C7	0.7277 (3)	0.7473 (4)	0.2225 (2)	0.0536 (9)
H7	0.7174	0.6871	0.2554	0.064*
C8	0.4663 (2)	0.6748 (3)	0.09047 (19)	0.0350 (7)
C9	0.5078 (3)	0.5379 (4)	0.1130 (2)	0.0547 (9)
H9	0.5680	0.5255	0.1607	0.066*
C10	0.4601 (4)	0.4197 (4)	0.0649 (3)	0.0745 (14)
H10	0.4887	0.3280	0.0802	0.089*
C11	0.3726 (4)	0.4361 (5)	-0.0039 (4)	0.0806 (16)
H11	0.3407	0.3554	-0.0356	0.097*
C12	0.3304 (3)	0.5698 (5)	-0.0274 (3)	0.0758 (13)
H12	0.2699	0.5806	-0.0751	0.091*
C13	0.3777 (3)	0.6897 (4)	0.0197 (2)	0.0532 (9)
H13	0.3492	0.7812	0.0033	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0441 (2)	0.03445 (18)	0.0531 (2)	0.00738 (15)	0.01948 (17)	0.00760 (16)

supplementary materials

P1	0.0314 (4)	0.0303 (4)	0.0278 (4)	0.0010 (3)	0.0153 (3)	0.0020 (3)
C1	0.0389 (18)	0.0512 (19)	0.0291 (16)	0.0072 (14)	0.0181 (15)	0.0059 (14)
C2	0.0333 (17)	0.0365 (16)	0.0297 (16)	-0.0014 (13)	0.0161 (14)	-0.0020 (13)
C3	0.042 (2)	0.0475 (19)	0.0397 (19)	0.0000 (15)	0.0220 (17)	0.0029 (15)
C4	0.060 (3)	0.064 (2)	0.062 (3)	-0.0087 (19)	0.043 (2)	0.0009 (19)
C5	0.040 (2)	0.077 (3)	0.070 (3)	-0.006 (2)	0.033 (2)	-0.009 (2)
C6	0.038 (2)	0.079 (3)	0.072 (3)	0.017 (2)	0.023 (2)	0.014 (2)
C7	0.042 (2)	0.063 (2)	0.055 (2)	0.0097 (17)	0.0244 (19)	0.0205 (19)
C8	0.0389 (19)	0.0366 (16)	0.0392 (19)	-0.0057 (13)	0.0272 (17)	-0.0034 (14)
C9	0.069 (3)	0.0364 (18)	0.065 (3)	-0.0034 (17)	0.040 (2)	0.0005 (18)
C10	0.113 (4)	0.034 (2)	0.111 (4)	-0.016 (2)	0.083 (4)	-0.014 (2)
C11	0.096 (4)	0.073 (3)	0.108 (4)	-0.049 (3)	0.078 (4)	-0.053 (3)
C12	0.058 (3)	0.094 (3)	0.072 (3)	-0.028 (2)	0.031 (2)	-0.042 (3)
C13	0.045 (2)	0.060 (2)	0.049 (2)	-0.0064 (17)	0.021 (2)	-0.0139 (18)

Geometric parameters (Å, °)

Se1—P1	2.0979 (9)	C6—C7	1.383 (5)
P1—C1	1.822 (3)	C6—H6	0.9300
P1—C2	1.809 (3)	C7—H7	0.9300
P1—C8	1.812 (3)	C8—C13	1.369 (5)
C1—C1 ⁱ	1.517 (6)	C8—C9	1.382 (4)
C1—H1A	0.9700	C9—C10	1.378 (6)
C1—H1B	0.9700	C9—H9	0.9300
C2—C3	1.377 (4)	C10—C11	1.344 (7)
C2—C7	1.386 (5)	C10—H10	0.9300
C3—C4	1.379 (5)	C11—C12	1.361 (7)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.364 (5)	C12—C13	1.383 (5)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.362 (6)	C13—H13	0.9300
C5—H5	0.9300		
C2—P1—C8	106.64 (13)	C5—C6—C7	120.1 (4)
C2—P1—C1	105.13 (14)	C5—C6—H6	120.0
C8—P1—C1	106.37 (15)	C7—C6—H6	120.0
C2—P1—Se1	113.91 (10)	C6—C7—C2	120.4 (3)
C8—P1—Se1	112.79 (11)	C6—C7—H7	119.8
C1—P1—Se1	111.41 (11)	C2—C7—H7	119.8
C1 ⁱ —C1—P1	112.2 (3)	C13—C8—C9	118.6 (3)
C1 ⁱ —C1—H1A	109.2	C13—C8—P1	120.7 (2)
P1—C1—H1A	109.2	C9—C8—P1	120.7 (3)
C1 ⁱ —C1—H1B	109.2	C10—C9—C8	120.2 (4)
P1—C1—H1B	109.2	C10—C9—H9	119.9
H1A—C1—H1B	107.9	C8—C9—H9	119.9
C3—C2—C7	118.2 (3)	C11—C10—C9	120.5 (4)
C3—C2—P1	119.4 (2)	C11—C10—H10	119.8
C7—C2—P1	122.3 (2)	C9—C10—H10	119.8
C2—C3—C4	121.1 (3)	C10—C11—C12	120.4 (4)

C2—C3—H3	119.5	C10—C11—H11	119.8
C4—C3—H3	119.5	C12—C11—H11	119.8
C5—C4—C3	119.8 (3)	C11—C12—C13	119.8 (4)
C5—C4—H4	120.1	C11—C12—H12	120.1
C3—C4—H4	120.1	C13—C12—H12	120.1
C6—C5—C4	120.3 (3)	C8—C13—C12	120.5 (4)
C6—C5—H5	119.8	C8—C13—H13	119.7
C4—C5—H5	119.8	C12—C13—H13	119.7
C2—P1—C1—C1 ⁱ	176.96 (11)	P1—C2—C7—C6	176.0 (3)
C8—P1—C1—C1 ⁱ	-70.17 (14)	C2—P1—C8—C13	-123.7 (3)
Se1—P1—C1—C1 ⁱ	53.12 (12)	C1—P1—C8—C13	124.5 (3)
C8—P1—C2—C3	97.9 (3)	Se1—P1—C8—C13	2.1 (3)
C1—P1—C2—C3	-149.4 (3)	C2—P1—C8—C9	55.5 (3)
Se1—P1—C2—C3	-27.1 (3)	C1—P1—C8—C9	-56.3 (3)
C8—P1—C2—C7	-79.1 (3)	Se1—P1—C8—C9	-178.7 (2)
C1—P1—C2—C7	33.6 (3)	C13—C8—C9—C10	-0.2 (5)
Se1—P1—C2—C7	155.8 (3)	P1—C8—C9—C10	-179.4 (3)
C7—C2—C3—C4	1.2 (5)	C8—C9—C10—C11	-0.6 (6)
P1—C2—C3—C4	-176.0 (3)	C9—C10—C11—C12	0.8 (7)
C2—C3—C4—C5	-0.7 (6)	C10—C11—C12—C13	-0.1 (7)
C3—C4—C5—C6	0.0 (6)	C9—C8—C13—C12	0.8 (5)
C4—C5—C6—C7	0.1 (7)	P1—C8—C13—C12	-180.0 (3)
C5—C6—C7—C2	0.5 (6)	C11—C12—C13—C8	-0.7 (6)
C3—C2—C7—C6	-1.1 (5)		

Symmetry codes: (i) $-x+1, y, -z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C10—H10 \cdots Se1 ⁱⁱ	0.93	2.97	3.751 (4)	143
C1—H1B \cdots Se1 ⁱ	0.97	2.90	3.533 (3)	124
C13—H13 \cdots Se1	0.93	2.87	3.410 (4)	119

Symmetry codes: (ii) $x, y-1, z$; (i) $-x+1, y, -z+1/2$.

Fig. 1

