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1,2-Bis(phenylphosphoryl)ethane

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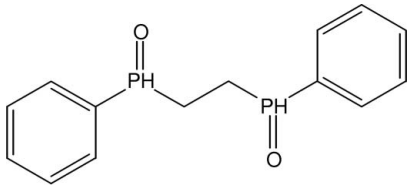
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.038; wR factor = 0.093; data-to-parameter ratio = 15.2.

The geometric parameters of the molecule of the title compound, $C_{14}H_{16}O_2P_2$, are in the usual ranges. It is a *meso* compound with the two chiral P atoms having opposite configurations. The P—CH_2—CH_2—P chain adopts a *trans* conformation [torsion angle −178.59 (17)°]. The P=O bonds are almost coplanar with the adjacent phenyl ring [torsion angles = 3.8 (3) and 0.3 (3)°]. Whereas one of them is synclinal [torsion angle = −59.0 (2)°] to the central C—C bond, the other is anticlinal [torsion angle = 56.6 (2)°] to the central C—C bond. The dihedral angle between the two phenyl rings is 5.2 (3)°. The molecules are linked by weak C—H···O hydrogen bonds. They crystallize in rows running along the *c* axis.

Related literature

For related literature, see: Dornhaus *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_{16}O_2P_2$
M_r = 278.21
Orthorhombic, $Pca2_1$
a = 10.2700 (10) Å
b = 5.1994 (5) Å
c = 26.241 (4) Å
V = 1401.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 173 (2) K
0.31 × 0.24 × 0.08 mm

Data collection

Stoe IPDS II two-circle diffractometer
Absorption correction: multi-scan [*MULABS* (Spek, 2003; Blessing, 1995)]
T_{min} = 0.912, T_{max} = 0.966
4658 measured reflections
2482 independent reflections
2157 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.093
S = 1.00
2482 reflections
163 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 1143 Friedel pairs
Flack parameter: 0.06 (14)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O2^{i}	0.99	2.43	3.159 (4)	130
C1—H1B···O1^{ii}	0.99	2.45	3.436 (4)	173
C2—H2A···O2^{iii}	0.99	2.40	3.386 (4)	176
C2—H2B···O1^{iv}	0.99	2.41	3.146 (4)	131
C12—H12···O1^{ii}	0.95	2.47	3.332 (4)	150
C26—H26···O2^{iii}	0.95	2.52	3.366 (4)	148

Symmetry codes: (i) $x+\frac{1}{2}, -y, z$; (ii) $x-\frac{1}{2}, -y, z$; (iii) $x+\frac{1}{2}, -y+1, z$; (iv) $x-\frac{1}{2}, -y+1, z$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2477).

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supporting information

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1,2-Bis(phenylphosphoryl)ethane

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S1. Comment

Very recently we have reported the syntheses of the diphospine PhHP—CH_2—CH_2—PHPh (Ph = C_6H_5) (Dornhaus *et al.*, 2007). Oxidation of the diphosphine *rac/meso* PhHPH-CH_2—CH_2—PHPh with air provides facile access to the corresponding phosphine oxide *rac/meso* PhHPO—CH_2—CH_2—OPHPh. Single crystals of the pure diastereomer *meso* PhHPO—CH_2—CH_2—OPHPh have been obtained from diphospine PhHP—CH_2—CH_2—PHPh in air at room temperature.

The P—CH_2—CH_2—P chain adopts a *trans* conformation [torsion angle -178.59 (17)°]. The P?O bonds are almost coplanar with the adjacent phenyl ring [torsion angles 3.8 (3)° and 0.3 (3)°]. Whereas one of them is synclinal [torsion angle -59.0 (2)°] to the central C—C bond the other one is anticlinal [torsion angle 56.6 (2)°] to the central C—C bond. The dihedral angle between the two phenyl rings is 5.2 (3)°. The molecules are linked by weak C—H···O hydrogen bonds. They crystallize in rows running along the *c* axis.

S2. Experimental

The diphospine PhHP—CH_2—CH_2—PHPh (0.29 g, 1.2 mmol) has been stored in air for 24 h at room temperature. Single crystals of the pure diastereomer *meso* PhHPO—CH_2—CH_2—OPHPh have been obtained in 5% yield.

S3. Refinement

H atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(C,*P*)] using a riding model with P—H = 1.3 Å, $C_{aromatic}$—H = 0.95 Å and $C_{methylene}$—H = 0.99 Å

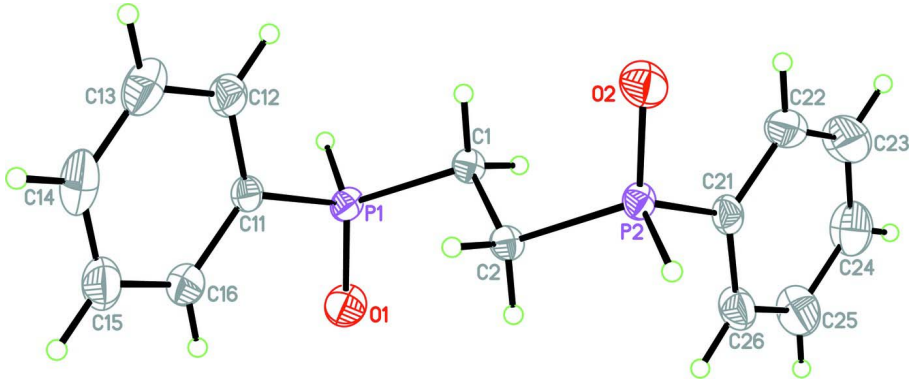

Figure 1

Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

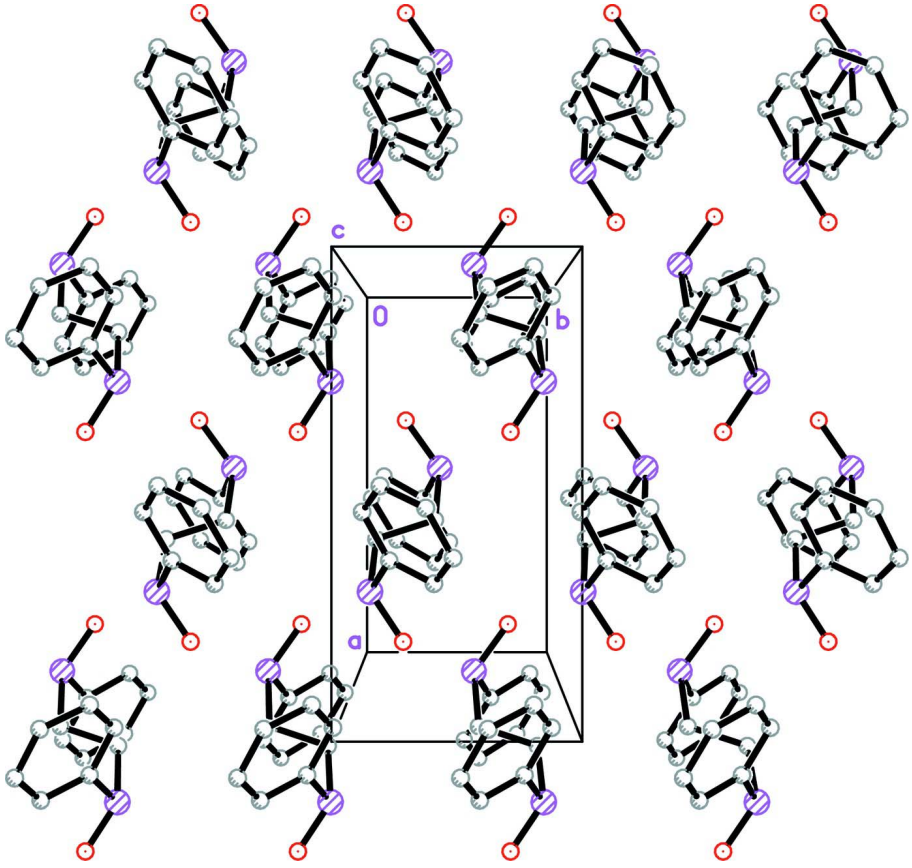

Figure 2

Packing diagram of the title compound.

1,2-Bis(phenylphosphoryl)ethane

Crystal data

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$M_r = 278.21$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 10.270\ (1)$ Å
$b = 5.1994\ (5)$ Å
$c = 26.241\ (4)$ Å
$V = 1401.2\ (3)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5153 reflections
$\theta = 3.9–25.8°$
$\mu = 0.30$ mm^{-1}
$T = 173$ K
Plate, colourless
0.31 × 0.24 × 0.08 mm

Data collection

Stoe IPDS II two-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan [*MULABS* (Spek, 2003; Blessing, 1995)]
$T_{min} = 0.912$, $T_{max} = 0.966$
4658 measured reflections
2482 independent reflections
2157 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.6°$, $\theta_{min} = 4.0°$
$h = -12 \rightarrow 11$
$k = -6 \rightarrow 5$
$l = -31 \rightarrow 30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.093$
$S = 1.00$
2482 reflections
163 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983), with 1143 Friedel pairs
Absolute structure parameter: 0.06 (14)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.75634 (7)	0.09258 (15)	0.55327 (3)	0.01962 (18)
H1	0.7902	−0.1473	0.5577	0.024*
O1	0.8765 (2)	0.2475 (5)	0.54496 (9)	0.0332 (6)
C1	0.6354 (3)	0.1150 (6)	0.50342 (11)	0.0184 (6)
H1A	0.6725	0.0496	0.4711	0.022*
H1B	0.5595	0.0064	0.5123	0.022*
C2	0.5901 (3)	0.3960 (6)	0.49600 (11)	0.0185 (6)
H2A	0.6655	0.5038	0.4862	0.022*
H2B	0.5550	0.4629	0.5285	0.022*
P2	0.46596 (7)	0.41704 (16)	0.44712 (3)	0.01884 (18)
H2	0.4279	0.6551	0.4442	0.023*
O2	0.3486 (2)	0.2528 (5)	0.45636 (9)	0.0340 (6)
C11	0.6761 (3)	0.1889 (7)	0.61166 (11)	0.0205 (7)
C12	0.5615 (3)	0.0667 (8)	0.62867 (12)	0.0277 (7)
H12	0.5267	−0.0746	0.6102	0.033*
C13	0.4993 (4)	0.1532 (8)	0.67257 (12)	0.0377 (9)
H13	0.4228	0.0688	0.6842	0.045*
C14	0.5479 (5)	0.3611 (10)	0.69939 (12)	0.0401 (11)
H14	0.5036	0.4213	0.7288	0.048*
C15	0.6626 (4)	0.4834 (8)	0.68326 (15)	0.0404 (9)
H15	0.6966	0.6249	0.7019	0.048*
C16	0.7263 (4)	0.3955 (8)	0.63960 (12)	0.0317 (8)
H16	0.8045	0.4767	0.6288	0.038*

C21	0.5436 (3)	0.3326 (7)	0.38734 (11)	0.0228 (7)
C22	0.4890 (4)	0.1358 (8)	0.35768 (13)	0.0348 (8)
H22	0.4126	0.0496	0.3689	0.042*
C23	0.5475 (5)	0.0670 (9)	0.31147 (15)	0.0482 (11)
H23	0.5113	−0.0676	0.2915	0.058*
C24	0.6574 (4)	0.1937 (9)	0.29480 (13)	0.0427 (10)
H24	0.6960	0.1467	0.2632	0.051*
C25	0.7122 (4)	0.3893 (9)	0.32372 (13)	0.0420 (10)
H25	0.7890	0.4731	0.3122	0.050*
C26	0.6548 (3)	0.4630 (8)	0.36958 (12)	0.0310 (8)
H26	0.6905	0.6007	0.3888	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0143 (4)	0.0230 (4)	0.0216 (3)	0.0010 (3)	−0.0016 (3)	0.0057 (4)
O1	0.0210 (12)	0.0438 (16)	0.0348 (14)	−0.0042 (11)	−0.0010 (10)	0.0071 (11)
C1	0.0150 (13)	0.0190 (17)	0.0210 (13)	0.0030 (13)	−0.0019 (11)	0.0008 (12)
C2	0.0140 (13)	0.0201 (16)	0.0215 (13)	0.0016 (13)	−0.0020 (10)	0.0009 (12)
P2	0.0140 (3)	0.0233 (4)	0.0192 (3)	0.0013 (3)	−0.0012 (3)	0.0027 (4)
O2	0.0217 (12)	0.0443 (17)	0.0360 (15)	−0.0038 (11)	−0.0010 (10)	0.0018 (11)
C11	0.0195 (16)	0.0225 (17)	0.0194 (13)	−0.0002 (14)	−0.0045 (12)	0.0033 (12)
C12	0.0236 (17)	0.035 (2)	0.0244 (15)	−0.0017 (16)	−0.0032 (12)	0.0040 (14)
C13	0.033 (2)	0.051 (3)	0.0288 (17)	0.0050 (19)	0.0058 (14)	0.0099 (15)
C14	0.054 (3)	0.045 (3)	0.0209 (17)	0.015 (2)	0.0010 (15)	0.0014 (13)
C15	0.065 (3)	0.033 (2)	0.0230 (16)	0.000 (2)	−0.0038 (17)	−0.0014 (15)
C16	0.042 (2)	0.0276 (18)	0.0253 (16)	−0.0032 (17)	−0.0053 (14)	0.0039 (13)
C21	0.0222 (16)	0.0247 (17)	0.0215 (14)	0.0031 (14)	−0.0062 (12)	0.0016 (13)
C22	0.043 (2)	0.033 (2)	0.0282 (17)	−0.0124 (17)	−0.0002 (15)	−0.0011 (14)
C23	0.069 (3)	0.044 (2)	0.0324 (19)	−0.012 (2)	−0.0006 (18)	−0.0133 (19)
C24	0.051 (2)	0.053 (3)	0.0241 (16)	0.006 (2)	0.0063 (16)	−0.0064 (16)
C25	0.032 (2)	0.066 (3)	0.0276 (16)	−0.009 (2)	0.0071 (14)	−0.0015 (18)
C26	0.0290 (18)	0.043 (2)	0.0211 (16)	−0.0067 (17)	−0.0003 (12)	−0.0024 (14)

Geometric parameters (Å, º)

P1—O1	1.490 (2)	C13—H13	0.9500
P1—C1	1.808 (3)	C14—C15	1.404 (6)
P1—C11	1.811 (3)	C14—H14	0.9500
P1—H1	1.3000	C15—C16	1.396 (5)
C1—C2	1.546 (4)	C15—H15	0.9500
C1—H1A	0.9900	C16—H16	0.9500
C1—H1B	0.9900	C21—C22	1.403 (5)
C2—P2	1.812 (3)	C21—C26	1.407 (5)
C2—H2A	0.9900	C22—C23	1.400 (6)
C2—H2B	0.9900	C22—H22	0.9500
P2—O2	1.497 (3)	C23—C24	1.378 (6)
P2—C21	1.814 (3)	C23—H23	0.9500

P2—H2	1.3000	C24—C25	1.388 (6)
C11—C16	1.399 (5)	C24—H24	0.9500
C11—C12	1.410 (5)	C25—C26	1.394 (5)
C12—C13	1.392 (5)	C25—H25	0.9500
C12—H12	0.9500	C26—H26	0.9500
C13—C14	1.383 (6)		

O1—P1—C1	115.39 (14)	C14—C13—C12	120.5 (4)
O1—P1—C11	110.57 (16)	C14—C13—H13	119.7
C1—P1—C11	106.35 (14)	C12—C13—H13	119.7
O1—P1—H1	108.1	C13—C14—C15	120.2 (3)
C1—P1—H1	108.1	C13—C14—H14	119.9
C11—P1—H1	108.1	C15—C14—H14	119.9
C2—C1—P1	111.03 (18)	C16—C15—C14	119.5 (4)
C2—C1—H1A	109.4	C16—C15—H15	120.2
P1—C1—H1A	109.4	C14—C15—H15	120.2
C2—C1—H1B	109.4	C15—C16—C11	120.5 (4)
P1—C1—H1B	109.4	C15—C16—H16	119.7
H1A—C1—H1B	108.0	C11—C16—H16	119.7
C1—C2—P2	110.98 (18)	C22—C21—C26	119.5 (3)
C1—C2—H2A	109.4	C22—C21—P2	118.7 (3)
P2—C2—H2A	109.4	C26—C21—P2	121.8 (3)
C1—C2—H2B	109.4	C23—C22—C21	119.7 (4)
P2—C2—H2B	109.4	C23—C22—H22	120.1
H2A—C2—H2B	108.0	C21—C22—H22	120.1
O2—P2—C2	114.67 (15)	C24—C23—C22	120.3 (4)
O2—P2—C21	110.86 (16)	C24—C23—H23	119.9
C2—P2—C21	106.76 (15)	C22—C23—H23	119.9
O2—P2—H2	108.1	C23—C24—C25	120.6 (4)
C2—P2—H2	108.1	C23—C24—H24	119.7
C21—P2—H2	108.1	C25—C24—H24	119.7
C16—C11—C12	119.2 (3)	C24—C25—C26	120.1 (4)
C16—C11—P1	119.2 (3)	C24—C25—H25	120.0
C12—C11—P1	121.6 (3)	C26—C25—H25	120.0
C13—C12—C11	120.0 (4)	C25—C26—C21	119.8 (4)
C13—C12—H12	120.0	C25—C26—H26	120.1
C11—C12—H12	120.0	C21—C26—H26	120.1

O1—P1—C1—C2	−59.0 (2)	C12—C11—C16—C15	−1.3 (5)
C11—P1—C1—C2	64.0 (2)	P1—C11—C16—C15	176.5 (3)
P1—C1—C2—P2	−178.59 (17)	O2—P2—C21—C22	0.3 (3)
C1—C2—P2—O2	56.6 (2)	C2—P2—C21—C22	125.8 (3)
C1—C2—P2—C21	−66.6 (2)	O2—P2—C21—C26	178.8 (3)
O1—P1—C11—C16	3.8 (3)	C2—P2—C21—C26	−55.6 (3)
C1—P1—C11—C16	−122.2 (3)	C26—C21—C22—C23	1.6 (6)
O1—P1—C11—C12	−178.5 (3)	P2—C21—C22—C23	−179.8 (3)
C1—P1—C11—C12	55.5 (3)	C21—C22—C23—C24	−0.7 (6)
C16—C11—C12—C13	0.5 (5)	C22—C23—C24—C25	0.6 (7)

P1—C11—C12—C13	−177.2 (3)	C23—C24—C25—C26	−1.3 (7)
C11—C12—C13—C14	0.9 (5)	C24—C25—C26—C21	2.2 (6)
C12—C13—C14—C15	−1.5 (6)	C22—C21—C26—C25	−2.3 (5)
C13—C14—C15—C16	0.7 (6)	P2—C21—C26—C25	179.2 (3)
C14—C15—C16—C11	0.7 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	0.99	2.43	3.159 (4)	130
C1—H1*B*···O1^{ii}	0.99	2.45	3.436 (4)	173
C2—H2*A*···O2^{iii}	0.99	2.40	3.386 (4)	176
C2—H2*B*···O1^{iv}	0.99	2.41	3.146 (4)	131
C12—H12···O1^{ii}	0.95	2.47	3.332 (4)	150
C26—H26···O2^{iii}	0.95	2.52	3.366 (4)	148

Symmetry codes: (i) *x*+1/2, −*y*, *z*; (ii) *x*−1/2, −*y*, *z*; (iii) *x*+1/2, −*y*+1, *z*; (iv) *x*−1/2, −*y*+1, *z*.