data reports

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Crystal structure of *tert*-butyldiphenyl-phosphine oxide

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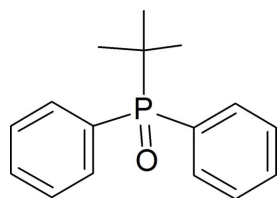
In the structure of the title triorganophosphine oxide, $C_{16}H_{19}OP$, the P—O bond is 1.490 (1) Å. The P atom has a distorted tetrahedral geometry. The O atom interacts with both phenyl groups of a neighboring molecule [C···O = 2.930 (3) and 2.928 (4) Å]. The C—O interaction directs an extended supramolecular arrangement along the *a*-axis.

1. Related literature

For the structures of related phosphine oxides $Ph_3P{=}O$, $EtPh_2P{=}O$ and $BuPh_2P{=}O$, see: Al-Farhan (1992), Orama & Koskinen (1994) and Caddy *et al.* (2007), respectively.

2. Experimental

2.1. Crystal data

$C_{16}H_{19}OP$
$M_r = 258.28$

Monoclinic, $P2_1$
$a = 6.3432$ (6) Å

$b = 9.5219$ (9) Å
$c = 12.4556$ (15) Å
$\beta = 101.665$ (10)°
$V = 736.78$ (13) Å^3
$Z = 2$

Mo $K\alpha$ radiation
$\mu = 0.17$ mm^{-1}
$T = 293$ K
$0.4 \times 0.15 \times 0.04$ mm

2.2. Data collection

Agilent Xcalibur, Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
$T_{min} = 0.842$, $T_{max} = 1.000$

11029 measured reflections
2713 independent reflections
2203 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.086$
$S = 1.05$
2713 reflections
166 parameters
1 restraint

H-atom parameters constrained
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Absolute structure: Flack (1983)
Absolute structure parameter: 0.19 (10)

Table 1
Selected bond lengths (Å).

P1—O1	1.4897 (14)	P1—C7	1.825 (3)
P1—C1	1.821 (3)	P1—C13	1.841 (3)

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *publCIF* (Westrip, 2010).

Acknowledgements

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supporting information

Crystal structure of *tert*-butyldiphenylphosphine oxide

George Agbeworvi, Zerihun Assefa, Richard E. Sykora and Jared D. Taylor

S1. Synthesis and crystallization

The tert-butyldiphenylphosphine oxide was unintentionally obtained during the reaction used to coordinate the unoxidized ligand to gold(I). The product was obtained after mixing tert-butyldiphenylphosphine (0.0600 g, 0.24 mmol) with a solution of (C_4H_8S)AuCl (0.0264 g, 0.08 mmol) in tetrahydrofuran (20 mL) at -80ºC. The reaction solution was stirred for 3 hours and the solvent removed totally by purging nitrogen gas into the solution. The residue was then recrystallized from CH_2Cl_2/n-hexane mixture within six days. Partial evaporation of the solvent provided quality crystals of the title compound. Yield 90%. Melting point 137ºC (decomposition). ^{1}H NMR (CD_2Cl_2) [δ (ppm)]: 1.3(s),7.3(m), 7.6(m), 7.9(m). For ^{31}P NMR in CD_2Cl_2 [δ (ppm)]: 38.96. Infrared data (cm^{-1}): 3032 (C–H, Ar), 2908 (C–H, CH_3), 1597 (C=C, Ar), 1165 (P=O), 1126 (P–Ar).

S2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H-atoms were placed in calculated positions and allowed to ride during subsequent refinement, with U_{iso}(H) = 1.2U_{eq}(C) and C—H distances of 0.93 Å for ring hydrogens and with U_{iso}(H) = 1.5U_{eq}(O) and C—H distances of 0.96 Å for methyl hydrogens.
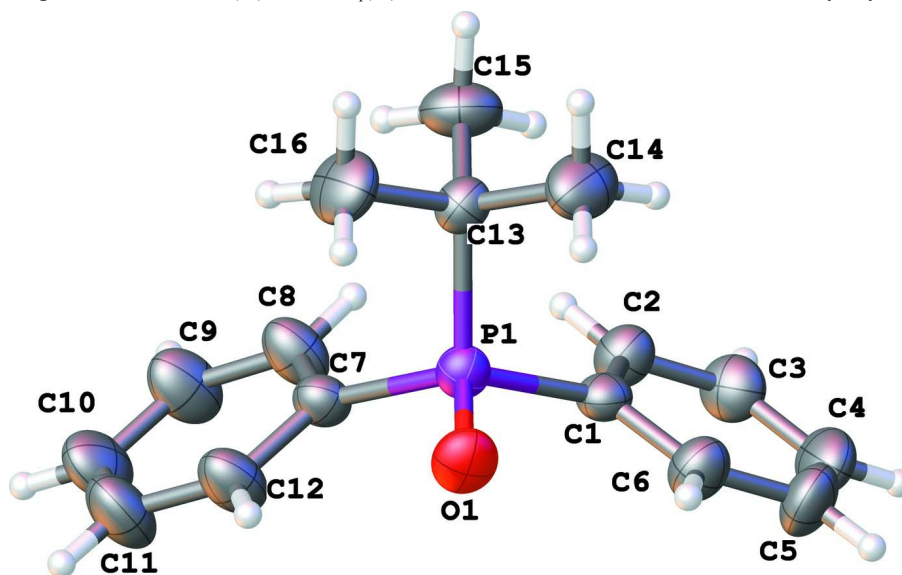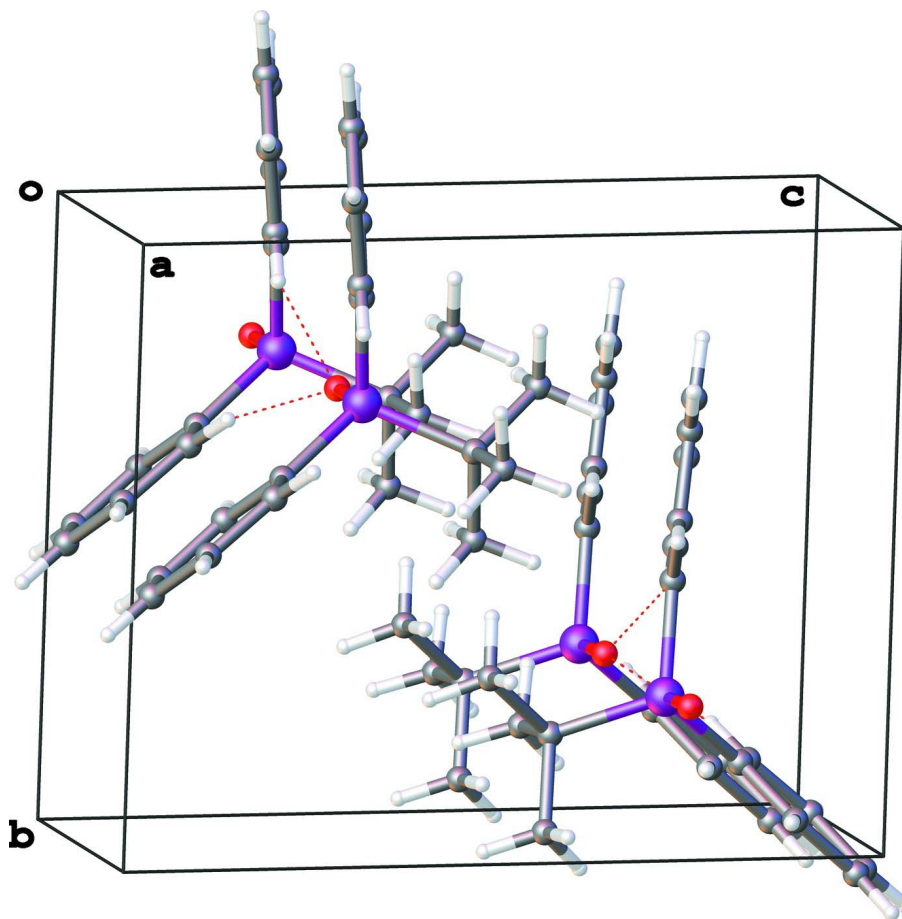

Figure 1

A ball-and-stick representation of the molecular structure of **I**.

Figure 2

Molecular packing with short C—H···O contacts indicated by dashed lines

tert-Butyldiphenylphosphine oxide

Crystal data

$C_{16}H_{19}OP$

$M_r = 258.28$

Monoclinic, $P2_1$

$a = 6.3432\ (6)$ Å

$b = 9.5219\ (9)$ Å

$c = 12.4556\ (15)$ Å

$\beta = 101.665\ (10)°$

$V = 736.78\ (13)$ Å^3

$Z = 2$

$F(000) = 276$

$D_x = 1.164$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2279 reflections

$\theta = 3.3–23.1°$

$\mu = 0.17$ mm^{-1}

$T = 293$ K

Plate, colourless

$0.4 \times 0.15 \times 0.04$ mm

Data collection

Agilent Xcalibur, Eos
  diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0514 pixels mm^{-1}

ω scans

Absorption correction: multi-scan
  (*CrysAlis PRO*; Agilent, 2014)

$T_{min} = 0.842$, $T_{max} = 1.000$

11029 measured reflections

2713 independent reflections

2203 reflections with $I > 2\sigma(I)$

$R_{int} = 0.049$

$\theta_{max} = 25.4°$, $\theta_{min} = 2.7°$	$k = -11 \rightarrow 11$
$h = -7 \rightarrow 7$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Hydrogen site location: inferred from
Least-squares matrix: full	neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 0.0459P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2713 reflections	$(\Delta/\sigma)_{max} < 0.001$
166 parameters	$\Delta\rho_{max} = 0.18$ e Å⁻³
1 restraint	$\Delta\rho_{min} = -0.24$ e Å⁻³
Primary atom site location: heavy-atom method	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.19 (10)

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 $F^2 > 2\sigma(F^2)$ 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	$U_{iso}*/U_{eq}$
P1	0.87488 (9)	0.74951 (7)	0.71978 (5)	0.03371 (18)
O1	1.1119 (2)	0.7455 (2)	0.72615 (15)	0.0475 (5)
C2	0.5797 (4)	0.5351 (3)	0.7611 (2)	0.0437 (7)
H2	0.4792	0.6045	0.7664	0.052*
C1	0.7771 (4)	0.5727 (3)	0.7363 (2)	0.0349 (7)
C14	0.7903 (5)	0.7211 (4)	0.4984 (2)	0.0631 (10)
H14A	0.9440	0.7152	0.5077	0.095*
H14B	0.7310	0.7586	0.4271	0.095*
H14C	0.7323	0.6291	0.5050	0.095*
C6	0.9206 (5)	0.4655 (3)	0.7265 (2)	0.0485 (8)
H6	1.0527	0.4876	0.7093	0.058*
C4	0.6771 (5)	0.2920 (3)	0.7685 (3)	0.0565 (9)
H4	0.6443	0.1988	0.7802	0.068*
C3	0.5318 (5)	0.3973 (3)	0.7776 (2)	0.0499 (8)
H3	0.4003	0.3745	0.7951	0.060*
C7	0.8146 (4)	0.8617 (3)	0.8285 (2)	0.0375 (7)
C8	0.6180 (4)	0.8754 (3)	0.8609 (3)	0.0513 (8)
H8	0.4985	0.8257	0.8246	0.062*
C10	0.7712 (5)	1.0408 (4)	1.0008 (2)	0.0609 (9)
H10	0.7562	1.0998	1.0583	0.073*
C9	0.6004 (5)	0.9632 (4)	0.9473 (3)	0.0615 (10)
H9	0.4692	0.9697	0.9694	0.074*

C12	0.9867 (4)	0.9407 (3)	0.8838 (3)	0.0543 (9)
H12	1.1197	0.9334	0.8637	0.065*
C11	0.9640 (5)	1.0303 (4)	0.9686 (3)	0.0671 (10)
H11	1.0807	1.0836	1.0036	0.081*
C5	0.8706 (5)	0.3264 (3)	0.7418 (3)	0.0590 (9)
H5	0.9682	0.2560	0.7341	0.071*
C13	0.7324 (4)	0.8175 (3)	0.5864 (2)	0.0361 (6)
C16	0.8231 (5)	0.9656 (3)	0.5733 (3)	0.0646 (10)
H16A	0.7806	1.0281	0.6255	0.097*
H16B	0.7678	0.9994	0.5004	0.097*
H16C	0.9774	0.9613	0.5858	0.097*
C15	0.4891 (4)	0.8239 (4)	0.5752 (3)	0.0577 (9)
H15A	0.4356	0.7323	0.5876	0.087*
H15B	0.4240	0.8552	0.5028	0.087*
H15C	0.4543	0.8885	0.6283	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0276 (3)	0.0354 (4)	0.0388 (4)	−0.0001 (4)	0.0084 (3)	0.0003 (4)
O1	0.0296 (8)	0.0516 (11)	0.0634 (12)	0.0015 (12)	0.0142 (8)	−0.0006 (14)
C2	0.0414 (16)	0.0407 (17)	0.0509 (19)	0.0010 (14)	0.0138 (13)	0.0036 (15)
C1	0.0355 (15)	0.0370 (17)	0.0306 (16)	0.0023 (12)	0.0031 (12)	0.0032 (12)
C14	0.081 (2)	0.068 (3)	0.0415 (18)	0.0185 (19)	0.0145 (16)	−0.0007 (17)
C6	0.0436 (17)	0.0429 (19)	0.060 (2)	0.0050 (13)	0.0127 (16)	0.0019 (15)
C4	0.070 (2)	0.036 (2)	0.059 (2)	−0.0047 (15)	0.0044 (17)	0.0081 (14)
C3	0.0496 (18)	0.047 (2)	0.054 (2)	−0.0094 (15)	0.0141 (15)	0.0064 (15)
C7	0.0312 (14)	0.0432 (17)	0.0368 (17)	−0.0002 (13)	0.0036 (12)	−0.0016 (13)
C8	0.0411 (17)	0.067 (2)	0.0468 (19)	−0.0115 (15)	0.0110 (14)	−0.0145 (16)
C10	0.063 (2)	0.078 (2)	0.0397 (19)	0.005 (2)	0.0079 (16)	−0.0177 (19)
C9	0.050 (2)	0.086 (3)	0.052 (2)	−0.0016 (18)	0.0181 (17)	−0.0197 (19)
C12	0.0380 (17)	0.071 (2)	0.052 (2)	−0.0032 (16)	0.0048 (15)	−0.0153 (18)
C11	0.055 (2)	0.081 (3)	0.061 (2)	−0.010 (2)	0.0004 (18)	−0.028 (2)
C5	0.060 (2)	0.0360 (19)	0.081 (3)	0.0131 (16)	0.0143 (18)	−0.0008 (17)
C13	0.0351 (14)	0.0347 (16)	0.0405 (17)	0.0010 (12)	0.0128 (12)	0.0037 (13)
C16	0.078 (2)	0.041 (2)	0.073 (3)	−0.0094 (17)	0.0122 (19)	0.0163 (17)
C15	0.0439 (16)	0.075 (2)	0.051 (2)	0.0054 (16)	0.0026 (15)	0.0143 (18)

Geometric parameters (Å, º)

P1—O1	1.4897 (14)	C7—C12	1.388 (3)
P1—C1	1.821 (3)	C8—H8	0.9300
P1—C7	1.825 (3)	C8—C9	1.385 (4)
P1—C13	1.841 (3)	C10—H10	0.9300
C2—H2	0.9300	C10—C9	1.368 (4)
C2—C1	1.395 (3)	C10—C11	1.366 (4)
C2—C3	1.372 (4)	C9—H9	0.9300
C1—C6	1.390 (3)	C12—H12	0.9300

C14—H14A	0.9600	C12—C11	1.388 (4)
C14—H14B	0.9600	C11—H11	0.9300
C14—H14C	0.9600	C5—H5	0.9300
C14—C13	1.530 (4)	C13—C16	1.544 (4)
C6—H6	0.9300	C13—C15	1.523 (3)
C6—C5	1.384 (4)	C16—H16A	0.9600
C4—H4	0.9300	C16—H16B	0.9600
C4—C3	1.382 (4)	C16—H16C	0.9600
C4—C5	1.374 (4)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600
C7—C8	1.393 (3)	C15—H15C	0.9600
O1—P1—C1	109.48 (12)	C9—C10—H10	120.5
O1—P1—C7	109.63 (12)	C11—C10—H10	120.5
O1—P1—C13	111.29 (10)	C11—C10—C9	119.0 (3)
C1—P1—C7	109.29 (12)	C8—C9—H9	119.2
C1—P1—C13	108.10 (12)	C10—C9—C8	121.6 (3)
C7—P1—C13	109.02 (12)	C10—C9—H9	119.2
C1—C2—H2	119.5	C7—C12—H12	119.4
C3—C2—H2	119.5	C11—C12—C7	121.2 (3)
C3—C2—C1	120.9 (3)	C11—C12—H12	119.4
C2—C1—P1	127.1 (2)	C10—C11—C12	120.4 (3)
C6—C1—P1	115.15 (19)	C10—C11—H11	119.8
C6—C1—C2	117.7 (3)	C12—C11—H11	119.8
H14A—C14—H14B	109.5	C6—C5—H5	119.9
H14A—C14—H14C	109.5	C4—C5—C6	120.1 (3)
H14B—C14—H14C	109.5	C4—C5—H5	119.9
C13—C14—H14A	109.5	C14—C13—P1	106.90 (19)
C13—C14—H14B	109.5	C14—C13—C16	108.9 (2)
C13—C14—H14C	109.5	C16—C13—P1	106.9 (2)
C1—C6—H6	119.4	C15—C13—P1	113.58 (18)
C5—C6—C1	121.2 (3)	C15—C13—C14	110.2 (2)
C5—C6—H6	119.4	C15—C13—C16	110.2 (2)
C3—C4—H4	120.3	C13—C16—H16A	109.5
C5—C4—H4	120.3	C13—C16—H16B	109.5
C5—C4—C3	119.3 (3)	C13—C16—H16C	109.5
C2—C3—C4	120.7 (3)	H16A—C16—H16B	109.5
C2—C3—H3	119.7	H16A—C16—H16C	109.5
C4—C3—H3	119.7	H16B—C16—H16C	109.5
C8—C7—P1	127.2 (2)	C13—C15—H15A	109.5
C12—C7—P1	115.06 (19)	C13—C15—H15B	109.5
C12—C7—C8	117.8 (3)	C13—C15—H15C	109.5
C7—C8—H8	120.0	H15A—C15—H15B	109.5
C9—C8—C7	120.0 (3)	H15A—C15—H15C	109.5
C9—C8—H8	120.0	H15B—C15—H15C	109.5
P1—C1—C6—C5	177.2 (3)	C3—C2—C1—C6	1.5 (4)
P1—C7—C8—C9	178.4 (3)	C3—C4—C5—C6	1.3 (5)

P1—C7—C12—C11	−179.9 (3)	C7—P1—C1—C2	43.6 (3)
O1—P1—C1—C2	163.7 (2)	C7—P1—C1—C6	−134.0 (2)
O1—P1—C1—C6	−14.0 (2)	C7—P1—C13—C14	179.33 (18)
O1—P1—C7—C8	−169.2 (3)	C7—P1—C13—C16	62.9 (2)
O1—P1—C7—C12	10.6 (3)	C7—P1—C13—C15	−58.9 (2)
O1—P1—C13—C14	58.3 (2)	C7—C8—C9—C10	1.7 (5)
O1—P1—C13—C16	−58.2 (2)	C7—C12—C11—C10	1.2 (5)
O1—P1—C13—C15	−180.0 (2)	C8—C7—C12—C11	0.0 (5)
C2—C1—C6—C5	−0.6 (4)	C9—C10—C11—C12	−1.0 (5)
C1—P1—C7—C8	−49.2 (3)	C12—C7—C8—C9	−1.4 (4)
C1—P1—C7—C12	130.6 (2)	C11—C10—C9—C8	−0.5 (5)
C1—P1—C13—C14	−62.0 (2)	C5—C4—C3—C2	−0.4 (5)
C1—P1—C13—C16	−178.44 (19)	C13—P1—C1—C2	−75.0 (2)
C1—P1—C13—C15	59.8 (2)	C13—P1—C1—C6	107.4 (2)
C1—C2—C3—C4	−1.0 (4)	C13—P1—C7—C8	68.7 (3)
C1—C6—C5—C4	−0.7 (5)	C13—P1—C7—C12	−111.4 (2)
C3—C2—C1—P1	−176.1 (2)		