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Di-*tert*-butylphosphine oxide

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The title compound, $C_8H_{19}OP$, is the oxidation product of ${}^t\mathrm{Bu_2PH}$. The molecule is located on a crystallographic mirror plane. As a result, there is just one half-molecule in the asymmetric unit.

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Key indicators

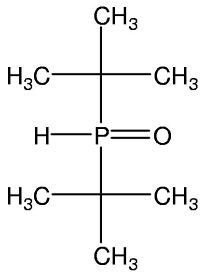
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.114
Data-to-parameter ratio = 19.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Transition metal complexes with phosphine ligands, PR_3 (R = alkyl, aryl, H) play an important role in homogeneous catalysis (Holleman & Wiberg, 1995). A disadvantage of these ligands is their air sensitivity. Upon standing under atmospheric conditions, they are oxidized to the corresponding phosphine oxide R_3PO (R = alkyl, aryl, H). We report here the synthesis and the X-ray crystal structure analysis of di-*tert*-butylphosphine oxide, ${}^t\mathrm{Bu_2HPO}$, (I). The synthesis of (I) was achieved by air oxidation of ${}^t\mathrm{Bu_2PH}$, as indicated in the scheme below.

$$2\ {}^t\mathrm{Bu_2PH} + \mathrm{O_2} \longrightarrow 2\ {}^t\mathrm{Bu_2HPO}$$



(I)

A perspective view of the title compound is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; Mogul Version 1.0.1; Allen, 2002). A crystallographic mirror plane perpendicular to the b axis runs through HP═O. Therefore, there is just one half-molecule in the asymmetric unit.

Experimental

Air oxidation of di-*tert*-butylphosphine (0.5 ml) for 2 d yields di-*tert*-butylphosphine oxide (yield 20%). Single crystals of di-*tert*-butylphosphine oxide were grown from the reaction mixture at ambient temperature. The NMR spectra were recorded on a Bruker DPX 250 and a Bruker Avance 400 spectrometer. ^{1}H NMR (C_6D_6, internal TMS): δ 0.97 (d, 2 × ^{t}Bu, ${}^3J_{PH}$ = 14.64 Hz), 5.82 (d, 1H, ${}^1J_{PH}$ = 422.40 Hz). ^{13}C{^{1}H} NMR (C_6D_6, internal TMS): δ 25.6 (d, 2 × CMe$_3$, ${}^2J_{PC}$ = 1.41 Hz), 33.5 (d, 2 × CMe$_3$, ${}^1J_{PC}$ = 59.29 Hz). ^{31}P NMR (C_6D_6, external H_3PO_4): δ 63.5 (dm, ${}^1J_{PH}$ = 422.40 Hz, ${}^3J_{HP}$ = 14.64 Hz).

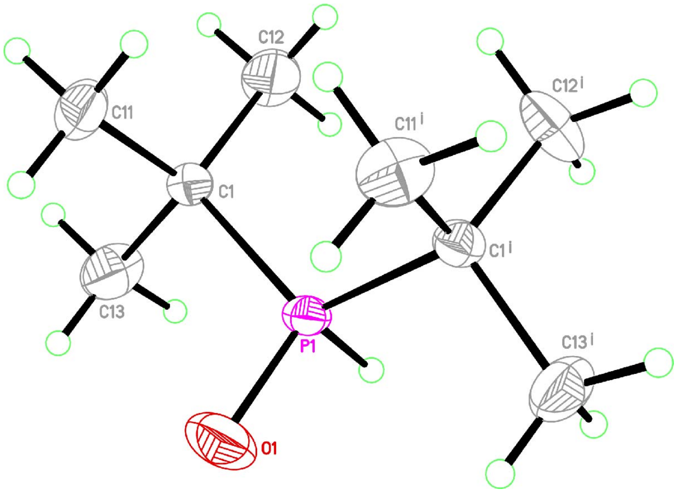


Figure 1
Perspective view of the title compound with the atom numbering and displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $x, -y+\frac{1}{2}, z$.]

Crystal data

$C_8H_{19}OP$
$M_r = 162.20$
Orthorhombic, *Pnma*
$a = 10.4244\ (10)$ Å
$b = 15.9819\ (16)$ Å
$c = 6.0919\ (7)$ Å
$V = 1014.92\ (18)$ Å^3
$Z = 4$
$D_x = 1.062$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 7754 reflections
$\theta = 3.8$–25.6°
$\mu = 0.22$ mm^{-1}
$T = 173\ (2)$ K
Plate, colourless
0.42 × 0.18 × 0.05 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{\min} = 0.915$, $T_{\max} = 0.979$
6207 measured reflections

985 independent reflections
885 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\max} = 25.5°$
$h = -12 \rightarrow 12$
$k = -17 \rightarrow 19$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.114$
$S = 1.06$
985 reflections
51 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.4766P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.32$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.011 (5)

Table 1
Selected bond lengths (Å).

P1—O1	1.4819 (19)	P1—H1	1.28 (3)
P1—C1	1.8484 (16)		

All H atoms were located in a difference map and were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm P})$ or $1.5U_{\rm eq}({\rm C})$] using a riding model (C—H = 0.98 Å). In addition, the P—H bond length was refined.

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).

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