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tert-Butyl diphenylphosphinate

I. Darren Grice,[a] Ian D. Jenkins,[b] W. Ken Busfield,[c] Karl A. Byriel[d] and Colin H. L. Kennard[e]*

[a]Institute for Glycomics, Griffith University, Gold Coast, Queensland, 4215, Australia, [b]Natural Product Discovery, Griffith University, Nathan, Brisbane, Queensland, 4111, Australia, [c]School of Science, Griffith University, Nathan, Brisbane, Queensland, 4111, Australia, [d]Centre for Drug Design and Development, The University of Queensland, Brisbane, Queensland, 4072, Australia, and [e]Chemistry Department, School of Molecular and Microbial Sciences, The University of Queensland, Brisbane, Queensland, 4072, Australia

Correspondence e-mail: C.Kennard@uq.edu.au

Molecules of the title compound, $C_{16}H_{19}O_2P$, pack simply in the orthorhombic space group *Pbca*, the dominant molecular feature being a tetrahedral P atom attached to two phenyl rings, a phosphate O atom and a tertiary butyloxy group.

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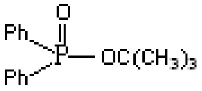
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.039
wR factor = 0.142
Data-to-parameter ratio = 15.2

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

Comment

The title compound, (I), was part of an investigation using the radical scavenger 1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxyl to study the reaction of phosphorus-centred radicals with alkenes and alkynes (Busfield *et al.*, 1995, Bottle *et al.*, 1994).



(3)

There is an intramolecular short distance between H26 and O4 of 2.46 Å, with an angle of 106° for C26—H26···O4. Reaction of the title compound with polyether ligands has been reported by Albanese *et al.* (2001).

Experimental

The title compound was obtained in 16% yield from the reaction of diphenylphosphine (100 mg), di-*tert*-butyl peroxyoxalate (DTBP) (58 mg) and benzene (2.5 ml) (solvent) in the presence of the radi-

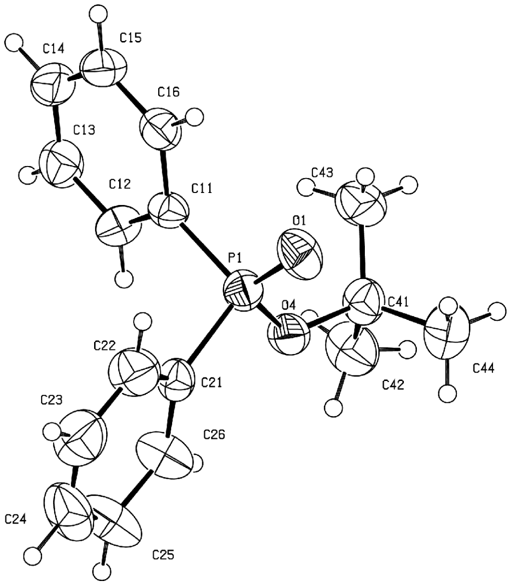


Figure 1
The molecular structure of the title compound, (3), with displacement ellipsoids drawn at the 50% probability level.

cal scavenger 1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxyl (103 mg), along with five other phosphorus-containing compounds. Reaction mixtures were degassed using repeated freezing/evacuating/thawing cycles on a high vacuum line, then sealed under vacuum in glass and heated for 10 half lives of the initiator, DTBP (68 minutes at 333 K). The reaction mixture was then separated by HPLC (Whatman Partisil 10-ODS-3 500 x 10 mm C18) using an isocratic 80:20 methanol/water, 4.0 ml/min method.

The isolated phosphorus-containing reaction products were eluted from the reversed-phase HPLC column in the following order.

(1) Diphenylphosphinic acid (0.4%), identical to an authentic sample by HPLC, m.p. 466–468 K. (Edmundson, 1986).

(2) Diphenylphosphine oxide (4.8%), identical to an authentic sample by HPLC and NMR.

(3) *tert*-Butyl diphenylphosphinate (16%). ^{1}H NMR δ (250 MHz, $CDCl_3$) 1.51, *s*, 9H, $(CH_3)_3$; 7.42, *m*, 6H, 7.79, *m*, 4H, aromatic H. ^{13}C NMR δ (62.8 MHz, $CDCl_3$) 30.8, $^3J_{P,C}$ 18.5, $(CH_3)_3$; 83.4, $C(CH_3)_3$; 128.2, $^3J_{P,C}$ 13.2, *meta* C; 129.1, *para* C; 131.3, $^2J_{P,C}$ 10.7, *ortho* C; 134.8, $J_{P,C}$ 138.5, *ipso* C. ^{31}P NMR (121 MHz, $CDCl_3$) 9.94. Structural confirmation by X-ray structural analysis.

(4) 2-(Diphenylphosphinyl)-1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindole (6.3%), white solid, m.p. 458–461 K (Found: C, 77.3; H, 6.5; N, 3.6. $C_{24}H_{26}NOP$ requires C, 76.8; H, 6.9; N, 3.7%). ^{1}H NMR δ (250 MHz, $CDCl_3$) 1.56, *br s*, 12H, $4CH_3$; 7.04, *m*, 2H, H4, H7; 7.23, *m*, 2H, H5, H6; 7.44, *m*, 6H, 7.83, *m*, 4H, Phenyl; ^{13}C NMR δ (62.8 MHz, $CDCl_3$) 32.2, *br s*, 4 × ring CH_3; 68.6, C1, C3; 120.9, C4, C7; 127.5, C5, C6; 127.8, *d*, $^2J_{P,C}$ 15.7, *d*, *ortho* C; 131.5, *s*, *para* C; 133.4, $^3J_{P,C}$ 10.7, *d*, *meta* C; 133.7, $^1J_{P,C}$ 138.1, *d*, *ipso* C; 145.9, C3a, C7a. ^{31}P NMR δ(121 MHz, $CDCl_3$) 9.70.

(5) 2-(Diphenylphosphinyloxy)-1,1,3,3-tetramethyl-2,3-dihydro-1 H-isoindole (34.3%), identical to an authentic sample (Busfield *et al.*, 1995).

(6) The material, 1,1,3,3-tetramethyl-2,3-dihydro-1*H*-isoindol-2-yloxy diphenylphosphinite (12%), was unstable, decomposing almost immediately in warmed aqueous THF to give by HPLC the free radical scavenger and diphenylphosphine oxide. High resolution MS failed to give a parent ion and the material was too unstable for elemental analysis. However, the following spectroscopic data were obtained: ^{1}H NMR δ (250 MHz, $CDCl_3$) 1.36, *br s*, 12H, $4CH_3$; 7.18–7.30, *m*, 8H, H4—H7, 4*H*-phenyl; 7.47, *m*, 6H, Phenyl; ^{13}C NMR δ (62.8 MHz, $CDCl_3$) 29.9, 30.0, *d*, 4 × ring CH_3; 120.0, C4, C7; 128.1, *d*, $^3J_{P,C}$ 6.97, *meta* C; 128.6, *s*, *para* C; 130.0, *d*, $^2J_{P,C}$ 22.1, *ortho* C; 143.7, 144.0, C3a, C7a. ^{31}P NMR δ (121 MHz, $CDCl_3$) 86.90. C1, C3, C5 and C6 from the isoindole moiety and the two *ipso* C atoms of the phenyl rings were not detected in the ^{13}C NMR spectrum. Percentage yields are based on moles of diphenylphosphine.

Sosnovsky & Zaret (1969) prepared the title compound by reacting diphenylchlorophosphine with *tert*-butyl hydroperoxide in pyridine. Another synthesis was reported by Sosnovsky *et al.* (1970).

Crystal data

$C_{16}H_{19}O_2P$
$M_r = 274.28$
Orthorhombic, *Pbca*
$a = 11.530\ (2)$ Å
$b = 17.118\ (2)$ Å
$c = 15.285\ (2)$ Å
$V = 3016.8\ (7)$ Å^3
$Z = 8$
$D_x = 1.208$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0–12.0°$
$\mu = 0.18$ mm^{-1}
$T = 293\ (2)$ K
Needle, colourless
0.40 × 0.13 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.954$, $T_{max} = 0.979$
2631 measured reflections
2631 independent reflections

802 reflections with $I > 2\sigma(I)$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 18$
25 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.142$
$S = 0.90$
2631 reflections
173 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0030 (4)

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.476 (3)	P1—O4	1.569 (3)
O1—P1—O4	118.00 (18)	O1—P1—C11	112.3 (2)
O1—P1—C21	113.3 (2)	O4—P1—C11	106.40 (19)
O4—P1—C21	99.7 (2)	C21—P1—C11	105.7 (2)

The crystal selected was a poor diffractor and only 30% of the intensities were significantly greater than background. H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with $U_{iso}(H) = 1.2$ or 1.5 times $U_{eq}(C)$.

Data collection: *SDP* (Frenz, 1985); cell refinement: *SDP*; data reduction: *WinGX* (Farrugia, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*98 (Spek, 1988); software used to prepare material for publication: *SHELXL*97.

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