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1-(Diphenylphosphinoyl)-3,3-dimethylbut-1-yne

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.088
Data-to-parameter ratio = 12.8

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

The title compound, $C_{18}H_{19}OP$, has a mirror plane passing through the phosphinoyl and acetylene groups. There is a weak intermolecular C—H···O interaction.

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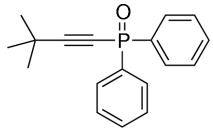
Comment

Acetylenic phosphine oxides have attracted considerable interest because they are versatile intermediates for the synthesis of complex natural products and biologically active compounds (Braga *et al.*, 2002). The asymmetric unit of the title compound, (I), consists of a half molecule. The molecule contains *tert*-butyl and $-O{=}P(Ph)_2$ moieties linked by an acetylene fragment. Atoms C9 and P1 and the acetylene group form a linear moiety and are located on a mirror plane. Atom P1 has a slightly distorted tetrahedral configuration. Bond angles around P1 are in the range 104.5 (1)–114.2 (2)° (Table 1). There are two benzene-ring planes, forming a dihedral angle of 119.15 (9)°. The three-dimensional framework of (I) is stabilized by a weak intermolecular C—H···O interaction (Table 1).



(I)

Experimental

tert-Butylethynylphosphine (50 mmol) was dissolved in acetone (50 ml), the mixture cooled to 273 K and 30% H_2O_2 (6 ml) in acetone

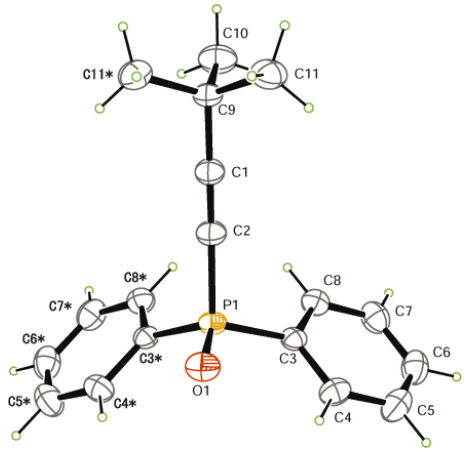


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. [Symmetry code: (*) 1 − *x*, *y*, *z*.]

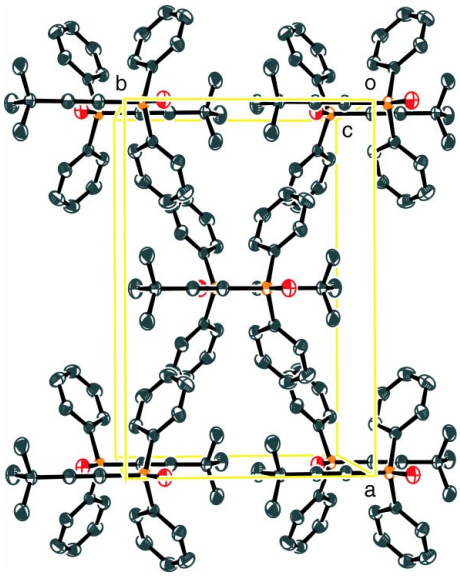


Figure 2
The crystal structure of (I). H atoms have been omitted.

(30 ml) added dropwise in 1 h. The mixture was extracted with ethyl acetate, dried with $MgSO_4$ and crystallized from diethyl ether and petroleum ether to obtain the title compound, (I), as a colorless solid (yield 13.5 g, 96%; Charrier *et al.*, 1966).

Crystal data

$C_{18}H_{19}OP$
$M_r = 282.32$
Orthorhombic, $Cmc2_1$
$a = 14.4742$ (7) Å
$b = 9.5767$ (7) Å
$c = 11.2075$ (6) Å
$V = 1553.5$ (2) Å^3
$Z = 4$
$D_x = 1.207$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8920 reflections
$\theta = 2.5$–$27.5°$
$\mu = 0.17$ mm^{-1}
$T = 296$ (1) K
Block, colorless
0.60 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.873$, $T_{max} = 0.983$
7260 measured reflections
1688 independent reflections
1309 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.036$
$\theta_{max} = 27.5°$
$h = -18 \rightarrow 18$
$k = -12 \rightarrow 12$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.088$
$S = 1.00$
1309 reflections
102 parameters
H-atom parameters constrained
$w = 1/[0.001F_o^2 + 1.1\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Absolute structure: (Flack, 1983), 632 Friedel pairs
Flack parameter = 0.01 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.97	2.55	3.464 (3)	156

Symmetry code: (i) $x, 1-y, z-\frac{1}{2}$.

The H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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