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[1-(Diphenylphosphinoyl)propa-1,2-dienyl]benzene

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.175
Data-to-parameter ratio = 11.9

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

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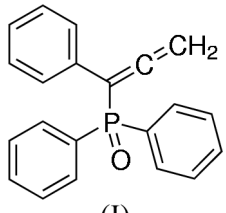
The title compound, $C_{21}H_{17}OP$, is a rare example of a structurally characterized allenyl phosphine oxide. The allenyl moiety is linear, as expected, with a bond angle of 179.2 (3)° at the central C atom.

Comment

Allenyl phosphine oxides have become generally accepted as useful intermediates in organic synthesis. The chemistry of allenyl phosphine oxides has been extensively studied and widely exploited in organic synthesis for several years (Brandsma & Verkruijsse, 1981). However, a search of the Cambridge Structural Database (CSD; Version 1.6; Allen, 2002) resulted in only three hits [CSD refcodes GOCZUT (Ansorge *et al.*, 1999), PUDQEK (Santelli-Rouvier *et al.*, 1997) and RILKUS (Muller & Ansorge, 1997)].



(I)

The structure of the title compound, (I), is shown in Fig. 1. As observed in the previously reported structures, atom P1 has a slightly distorted tetrahedral environment. The two adjacent C=C bonds in the allenyl group are slightly different in length [1.290 (5) and 1.316 (5) Å], the shorter one being for the C atom attached to phosphorus, and this feature seems to be general for all three reported structures (Ansorge *et al.*, 1999; Santelli-Rouvier *et al.*, 1997; Muller & Ansorge, 1997). The allenyl fragment is linear, with an angle of 179.2 (3)° at the central C atom.

The phenyl group attached to the allenic atom C3 is nearly perpendicular to the other two other phenyl rings, forming dihedral angles of 100.28 (12) and 106.16 (13)°.

Experimental

A solution of dried CH_2Cl_2 (100 ml) containing 3-phenylpropargyl alcohol (0.10 mol) and triethylamine (0.11 mol) was cooled to 193 K, and chlorodiphenyl phosphine (0.10 mol) in dried CH_2Cl_2 (75 ml) was added dropwise over a period of 3 min. The resulting solution was warmed to 283 K, and the reaction mixture was then poured into a solution of 36% HCl (2.5 ml) in water (100 ml), extracted with CH_2Cl_2 and dried with anhydrous $MgSO_4$ to give the allenyl phos-

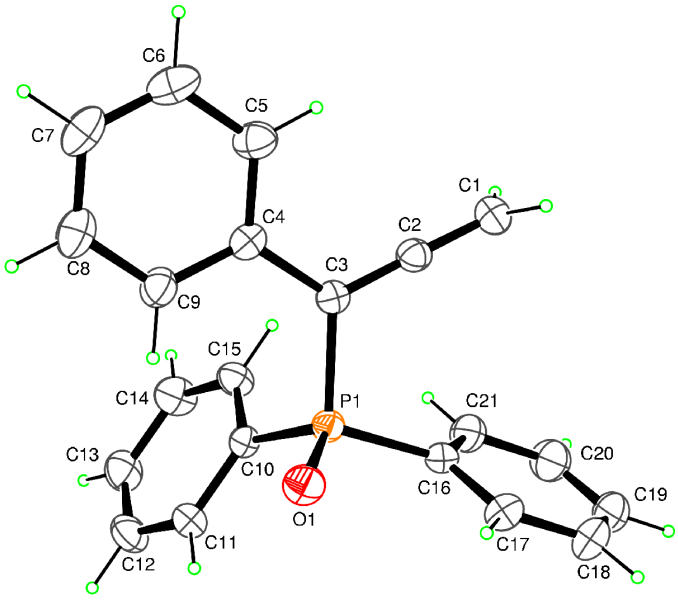


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-labeling scheme.

phine oxide (yield 30.2 g) as a white solid (Sevin & Chodkiewcz, 1967).

Crystal data

$C_{21}H_{17}OP$
M_r = 316.34
Monoclinic, $P2_1/c$
a = 9.5560 (3) Å
b = 15.4559 (5) Å
c = 12.0143 (3) Å
β = 108.904 (2)°
V = 1678.76 (9) Å^3
Z = 4
D_x = 1.252 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9907 reflections
θ = 1.3–27.3°
μ = 0.17 mm^{-1}
T = 296 (1) K
Block, colorless
0.57 × 0.25 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.820, T_{max} = 0.975
15 140 measured reflections
3751 independent reflections
2484 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.038
θ_{max} = 27.4°
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 20$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.175
S = 1.01
2484 reflections
208 parameters
H-atom parameters constrained
$w = 1/[0.0059F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

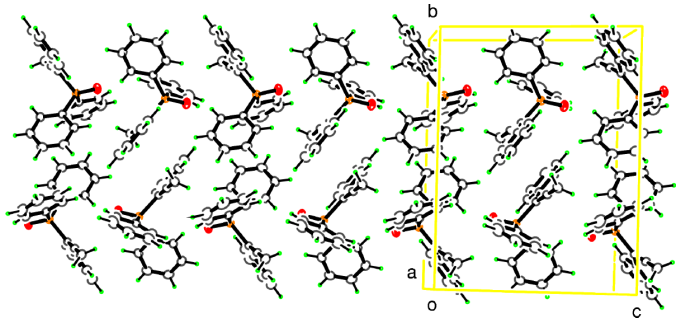


Figure 2
The packing in the structure of (I) along the *c* axis.

Table 1
Selected bond angles (°).

C3—P1—O1	113.9 (1)	C10—P1—C3	105.6 (1)
C10—P1—O1	112.3 (1)	C16—P1—C3	105.9 (1)
C16—P1—O1	111.3 (1)		

Atoms H1 and H2 were located in difference Fourier maps and included in the refinement as found, but they were constrained to ride on the C atom to which they were attached. All other H atoms were placed in calculated positions, with C—H = 0.97 Å, and included in the final cycles of refinement using the riding model, with U_{iso}(H) = 1.2U_{eq} of the carrier atoms.

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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