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(*E*)-1-(Diphenylphosphinyl)-2-(1-hydroxycyclohexyl)ethene

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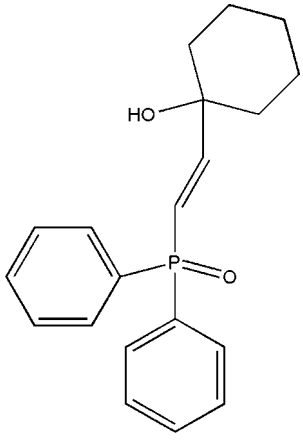
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.098; data-to-parameter ratio = 15.8.

The molecule of the title compound, $C_{20}H_{23}PO_2$, displays an E configuration about the C=C bond. The two phenyl rings are nearly perpendicular to each other with a dihedral angle of 80.09 (8)°. Intermolecular O—H···O hydrogen bonding and π–π stacking between parallel benzene rings of adjacent molecules, with a iperpendicular distance of 3.4928 (3) Å, help to stabilize the crystal structure.

Related literature

For general background, see: Brunner & Limmer (1991). For synthesis, see: Niu *et al.* (2007).



Experimental

Crystal data

$C_{20}H_{23}O_2P$
M_r = 326.35
Monoclinic, $P2_1/n$
a = 6.0174 (15) Å
b = 10.452 (2) Å
c = 28.043 (5) Å
β = 90.81 (2)°
V = 1763.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 295 (2) K
0.4 × 0.3 × 0.2 mm

Data collection

Bruker P4 diffractometer
Absorption correction: none
4757 measured reflections
3293 independent reflections
1621 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.098
S = 1.07
3293 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2B···O1^{i}	0.82	2.01	2.813 (3)	166

Symmetry code: (i) $x-1, y, z$.

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2254).

References

Bruker (1997). *XSCANS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Niu, M. Y., Fu, H., Jiang, Y. Y. & Zhao, Y. F. (2007). *Chem. Commun.* pp. 272–274.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

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supplementary materials

Acta Cryst. (2007). E**63**, o2973 [**doi:10.1107/S1600536807023422**]

(*E*)-1-(Diphenylphosphinyl)-2-(1-hydroxycyclohexyl)ethene

B. Yu, X.-Q. Wang, G.-Q. Shen and D.-Z. Shen

Comment

Alkenylphosphine oxides are useful compounds in numerous synthetic transformations. For example, heteroatom nucleophiles of primary ls@zapatalite:/e/xu2254$ and secondary amines readily add to the olefinic bond in alkenylphosphine oxide to give useful bifunctional adducts, which allow further synthetic elaboration (Brunner & Limmer, 1991).

The molecular structure of (I) is shown in Fig. 1. The molecule displays an E-configuration about C═C bond. Two phenyl rings are nearly perpendicular to each other with a dihedral angle of 80.09 (8)°.

Intermolecular O–H···O hydrogen bonding (Table 1) is observed in the crystal structure of (I). The crystal structure is further stabilized by π-π stacking between C1-benzene and C1^{i}-benzene [symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*], inter-planar separation being 3.4928 (3)Å (Fig. 2).

Experimental

A mixture of diphenylphosphine oxide (5 mmol), 1-ethynyl-1-cyclohexanol (7.5 mmol), CuI (0.5 mmol), ethylenediamine (EDA)(0.75 mmol) in 10 ml DMSO under N_2 atmosphere was heated at 333 K for 18 h. The resulting solution was cooled to room temperature, and then 10 ml chloroform and 10 ml brine were added to the solution. The organic layer was washed with brine (10 ml) and dried with anhydrous Na_2SO_4. After filtration, the filtrate was concentrated *in vacuo* to give a pale yellow semisolid. The crude product was then purified by silica gel column chromatography, EtOAc-hexane (1:1) as the eluent. The single crystals of (I) were obtained from the EtOAc-hexane solution (Niu *et al.*, 2007).

Refinement

H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.97 Å (methylene) and O—H = 0.82 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C,*O*).

Figures

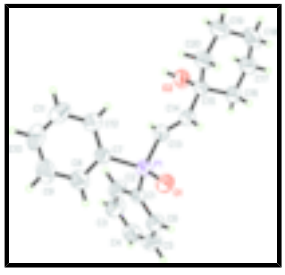

Fig. 1. The molecular structure of (I), with 50% probability displacement ellipsoids.

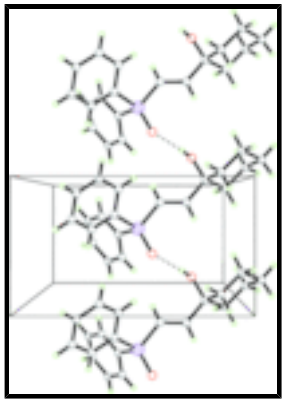

Fig. 2. The packing of (I), showing the hydrogen bonding (dashed lines).

(*E*)-1-(Diphenylphosphinyl)-2-(1-hydroxycyclohexyl)ethene

Crystal data

$C_{20}H_{23}O_2P$	F_{000} = 696
M_r = 326.35	D_x = 1.229 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 6.0174 (15) Å	θ = 4.8–11.1º
b = 10.452 (2) Å	μ = 0.16 mm^{-1}
c = 28.043 (5) Å	T = 295 (2) K
β = 90.81 (2)º	Prism, colorless
V = 1763.6 (6) Å^3	0.4 × 0.3 × 0.2 mm
Z = 4	

Data collection

Bruker P4 diffractometer	R_{int} = 0.055
Radiation source: fine-focus sealed tube	θ_{max} = 25.5º
Monochromator: graphite	θ_{min} = 2.0º
T = 295(2) K	h = −1→7
ω scans	k = −12→1
Absorption correction: none	l = −33→33
4757 measured reflections	3 standard reflections
3293 independent reflections	every 97 reflections
1621 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained

$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.001P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
3293 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.40018 (14)	0.51414 (7)	0.63461 (3)	0.0457 (2)
O1	0.6039 (3)	0.58117 (18)	0.65258 (6)	0.0524 (5)
O2	−0.1924 (3)	0.77017 (19)	0.59673 (7)	0.0590 (6)
H2B	−0.2480	0.7239	0.6170	0.071*
C1	0.4511 (5)	0.4185 (3)	0.58228 (9)	0.0427 (7)
C2	0.2967 (5)	0.3344 (3)	0.56462 (10)	0.0621 (9)
H2A	0.1644	0.3223	0.5807	0.075*
C3	0.3346 (6)	0.2672 (3)	0.52318 (11)	0.0724 (11)
H3A	0.2274	0.2113	0.5112	0.087*
C4	0.5315 (6)	0.2833 (3)	0.49970 (11)	0.0646 (9)
H4A	0.5571	0.2378	0.4718	0.077*
C5	0.6881 (5)	0.3646 (3)	0.51669 (11)	0.0644 (9)
H5A	0.8218	0.3744	0.5008	0.077*
C6	0.6478 (5)	0.4333 (3)	0.55804 (10)	0.0568 (9)
H6A	0.7547	0.4901	0.5696	0.068*
C7	0.2825 (5)	0.4049 (3)	0.67727 (9)	0.0443 (7)
C8	0.4023 (5)	0.2988 (3)	0.69112 (11)	0.0659 (10)
H8A	0.5410	0.2837	0.6780	0.079*
C9	0.3183 (7)	0.2141 (4)	0.72434 (12)	0.0816 (11)
H9A	0.4011	0.1430	0.7336	0.098*
C10	0.1129 (7)	0.2351 (4)	0.74366 (11)	0.0773 (12)
H10A	0.0563	0.1782	0.7659	0.093*
C11	−0.0072 (6)	0.3394 (4)	0.73009 (11)	0.0725 (10)
H11A	−0.1455	0.3540	0.7434	0.087*
C12	0.0741 (5)	0.4246 (3)	0.69656 (10)	0.0585 (9)

H12A	−0.0110	0.4946	0.6871	0.070*
C13	0.1806 (5)	0.6189 (3)	0.61876 (9)	0.0468 (8)
H13A	0.0558	0.5841	0.6038	0.056*
C14	0.1824 (5)	0.7429 (3)	0.62682 (10)	0.0469 (8)
H14A	0.3121	0.7773	0.6399	0.056*
C15	−0.0060 (5)	0.8346 (3)	0.61683 (10)	0.0452 (7)
C16	0.0617 (5)	0.9337 (3)	0.57950 (10)	0.0585 (9)
H16A	0.1980	0.9755	0.5899	0.070*
H16B	0.0900	0.8909	0.5495	0.070*
C17	−0.1181 (5)	1.0336 (3)	0.57195 (11)	0.0700 (10)
H17A	−0.0682	1.0962	0.5489	0.084*
H17B	−0.2506	0.9928	0.5590	0.084*
C18	−0.1741 (6)	1.1005 (3)	0.61773 (12)	0.0817 (11)
H18A	−0.2938	1.1609	0.6119	0.098*
H18B	−0.0456	1.1480	0.6292	0.098*
C19	−0.2443 (5)	1.0046 (3)	0.65560 (11)	0.0751 (10)
H19A	−0.3834	0.9649	0.6459	0.090*
H19B	−0.2683	1.0491	0.6855	0.090*
C20	−0.0674 (5)	0.9015 (3)	0.66299 (10)	0.0588 (9)
H20A	0.0649	0.9403	0.6769	0.071*
H20B	−0.1218	0.8385	0.6854	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0464 (4)	0.0406 (5)	0.0503 (4)	0.0011 (5)	0.0049 (3)	−0.0005 (4)
O1	0.0434 (12)	0.0539 (13)	0.0599 (12)	−0.0062 (11)	−0.0018 (10)	−0.0015 (11)
O2	0.0529 (13)	0.0498 (14)	0.0743 (14)	−0.0060 (12)	−0.0043 (12)	0.0101 (11)
C1	0.0474 (17)	0.0373 (16)	0.0435 (17)	−0.0005 (16)	0.0052 (14)	0.0026 (14)
C2	0.063 (2)	0.064 (2)	0.060 (2)	−0.011 (2)	0.0169 (18)	−0.0109 (19)
C3	0.078 (3)	0.070 (3)	0.070 (2)	−0.017 (2)	0.014 (2)	−0.020 (2)
C4	0.082 (3)	0.062 (2)	0.0500 (19)	0.010 (2)	0.0108 (19)	−0.0059 (18)
C5	0.058 (2)	0.076 (3)	0.060 (2)	0.008 (2)	0.0148 (18)	−0.001 (2)
C6	0.0515 (19)	0.061 (2)	0.0583 (19)	−0.0027 (18)	0.0055 (16)	−0.0047 (18)
C7	0.0497 (18)	0.0390 (18)	0.0443 (17)	−0.0005 (16)	0.0055 (15)	−0.0030 (14)
C8	0.071 (2)	0.063 (2)	0.065 (2)	0.015 (2)	0.0119 (18)	0.0115 (19)
C9	0.106 (3)	0.069 (2)	0.070 (2)	0.011 (3)	0.010 (2)	0.026 (2)
C10	0.104 (3)	0.076 (3)	0.052 (2)	−0.024 (3)	0.011 (2)	0.014 (2)
C11	0.069 (2)	0.090 (3)	0.059 (2)	−0.006 (2)	0.0199 (19)	0.004 (2)
C12	0.058 (2)	0.057 (2)	0.0607 (19)	0.0034 (19)	0.0092 (17)	0.0015 (18)
C13	0.0467 (19)	0.0422 (18)	0.0516 (18)	−0.0031 (16)	0.0022 (15)	0.0002 (15)
C14	0.0468 (18)	0.0454 (18)	0.0486 (17)	−0.0009 (16)	0.0074 (15)	0.0035 (16)
C15	0.0461 (18)	0.0363 (17)	0.0531 (18)	0.0013 (16)	0.0029 (15)	0.0011 (15)
C16	0.067 (2)	0.0463 (19)	0.063 (2)	0.0043 (18)	0.0169 (17)	0.0113 (17)
C17	0.083 (2)	0.056 (2)	0.071 (2)	0.012 (2)	0.0157 (19)	0.020 (2)
C18	0.103 (3)	0.046 (2)	0.097 (3)	0.020 (2)	0.016 (2)	0.008 (2)
C19	0.092 (3)	0.060 (2)	0.074 (2)	0.023 (2)	0.024 (2)	0.002 (2)
C20	0.074 (2)	0.051 (2)	0.0520 (18)	0.0087 (19)	0.0087 (17)	0.0001 (17)

Geometric parameters (Å, °)

P1—O1	1.4933 (18)	C10—H10A	0.9300
P1—C1	1.805 (3)	C11—C12	1.388 (4)
P1—C7	1.806 (3)	C11—H11A	0.9300
P1—C13	1.768 (3)	C12—H12A	0.9300
O2—C15	1.418 (3)	C13—C14	1.315 (3)
O2—H2B	0.8200	C13—H13A	0.9300
C1—C2	1.367 (4)	C14—C15	1.508 (4)
C1—C6	1.382 (3)	C14—H14A	0.9300
C2—C3	1.379 (4)	C15—C20	1.521 (4)
C2—H2A	0.9300	C15—C16	1.532 (3)
C3—C4	1.374 (4)	C16—C17	1.516 (4)
C3—H3A	0.9300	C16—H16A	0.9700
C4—C5	1.350 (4)	C16—H16B	0.9700
C4—H4A	0.9300	C17—C18	1.504 (4)
C5—C6	1.388 (4)	C17—H17A	0.9700
C5—H5A	0.9300	C17—H17B	0.9700
C6—H6A	0.9300	C18—C19	1.524 (4)
C7—C8	1.376 (4)	C18—H18A	0.9700
C7—C12	1.388 (4)	C18—H18B	0.9700
C8—C9	1.386 (4)	C19—C20	1.527 (4)
C8—H8A	0.9300	C19—H19A	0.9700
C9—C10	1.374 (4)	C19—H19B	0.9700
C9—H9A	0.9300	C20—H20A	0.9700
C10—C11	1.360 (4)	C20—H20B	0.9700
O1—P1—C13	113.65 (13)	C14—C13—P1	124.2 (3)
O1—P1—C1	112.72 (12)	C14—C13—H13A	117.9
C13—P1—C1	105.86 (13)	P1—C13—H13A	117.9
O1—P1—C7	113.62 (12)	C13—C14—C15	126.1 (3)
C13—P1—C7	105.04 (14)	C13—C14—H14A	116.9
C1—P1—C7	105.15 (12)	C15—C14—H14A	116.9
C15—O2—H2B	109.5	O2—C15—C14	111.1 (2)
C2—C1—C6	118.4 (3)	O2—C15—C20	110.9 (2)
C2—C1—P1	121.9 (2)	C14—C15—C20	109.0 (2)
C6—C1—P1	119.6 (2)	O2—C15—C16	105.4 (2)
C1—C2—C3	120.9 (3)	C14—C15—C16	110.5 (2)
C1—C2—H2A	119.6	C20—C15—C16	109.9 (2)
C3—C2—H2A	119.6	C17—C16—C15	111.4 (2)
C4—C3—C2	119.7 (3)	C17—C16—H16A	109.3
C4—C3—H3A	120.1	C15—C16—H16A	109.3
C2—C3—H3A	120.1	C17—C16—H16B	109.3
C5—C4—C3	120.7 (3)	C15—C16—H16B	109.3
C5—C4—H4A	119.7	H16A—C16—H16B	108.0
C3—C4—H4A	119.7	C18—C17—C16	111.6 (3)
C4—C5—C6	119.4 (3)	C18—C17—H17A	109.3
C4—C5—H5A	120.3	C16—C17—H17A	109.3
C6—C5—H5A	120.3	C18—C17—H17B	109.3

C1—C6—C5	120.9 (3)	C16—C17—H17B	109.3
C1—C6—H6A	119.5	H17A—C17—H17B	108.0
C5—C6—H6A	119.5	C17—C18—C19	110.9 (3)
C8—C7—C12	118.8 (3)	C17—C18—H18A	109.5
C8—C7—P1	119.2 (2)	C19—C18—H18A	109.5
C12—C7—P1	122.0 (2)	C17—C18—H18B	109.5
C7—C8—C9	120.7 (3)	C19—C18—H18B	109.5
C7—C8—H8A	119.7	H18A—C18—H18B	108.0
C9—C8—H8A	119.7	C18—C19—C20	111.0 (2)
C10—C9—C8	120.1 (4)	C18—C19—H19A	109.4
C10—C9—H9A	119.9	C20—C19—H19A	109.4
C8—C9—H9A	119.9	C18—C19—H19B	109.4
C11—C10—C9	119.7 (3)	C20—C19—H19B	109.4
C11—C10—H10A	120.2	H19A—C19—H19B	108.0
C9—C10—H10A	120.2	C15—C20—C19	112.7 (2)
C10—C11—C12	120.9 (3)	C15—C20—H20A	109.0
C10—C11—H11A	119.6	C19—C20—H20A	109.0
C12—C11—H11A	119.6	C15—C20—H20B	109.0
C7—C12—C11	119.8 (3)	C19—C20—H20B	109.0
C7—C12—H12A	120.1	H20A—C20—H20B	107.8
C11—C12—H12A	120.1		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2B···O1^{i}	0.82	2.01	2.813 (3)	166

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

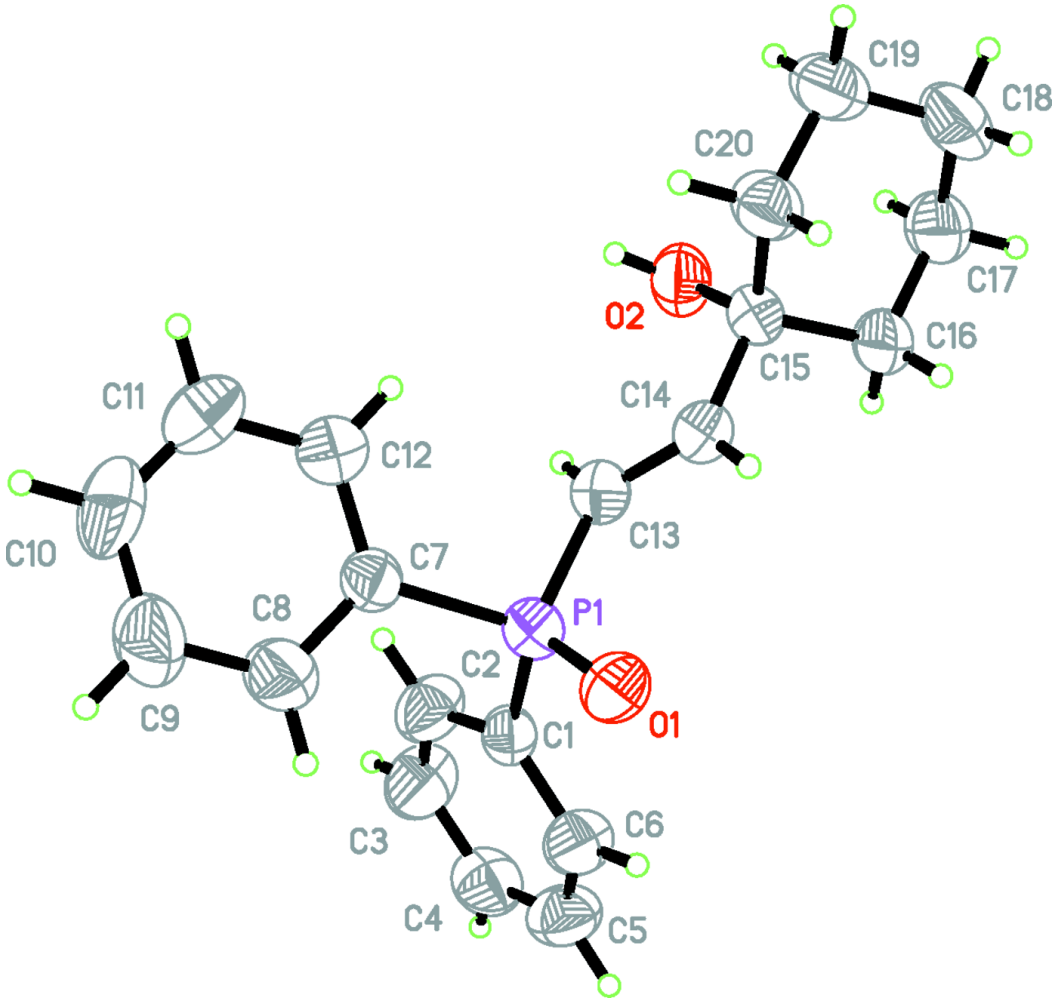
C19
C18
C20
C17
O2
C16
C15
C14
C11
C12
C13
C10
C7
C8
P1
C2
O1
C1
C9
C3
C6
C4
C5

Fig. 2