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(2*R*,*S*P)-2-Hydroxymethyl-1-phenylphospholane 1-oxide

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Abstract

A single_crystal structure analysis of the new title compound reveals an intermolecular hydrogen bond between the 2-methylhydroxyl group and the P═O group.

Comment

Optically active phosphines play a most important role as the chiral ligand in variours metal-catalyzed asymmetric reactions, and numberous chiral Phosphine have been designed and synthesized over the past three decades. The configuration of phosphine have played a signification role in the enantioselectively catalystic reactions. To study the relationship of enantioselectivety of reaction and the configuration of phosphine (Imamoto *et al.*, 1998) we designed a serious of phosphine compounds containing chiral phosphorus atom. The compound is one of them, and the single-crystal of this new compound was obtained by recrystallization from EtOH at room temperature.

Molecules are linked babout twofold screw axis by O2—H20···O1(1 − *x*, *y* + 1/2, 3/2 − *z*) hydrogen bonds with O···O 2.726 (3), O—H 0.90 (3), H···O 1.83 (3)Å and O—H···O 173 (3)°.

Experimental

The title compound was synthesized from 5-benzyloxy-1,4-dimesylate–pentane treated with H_2PPh and n-BuLi at 195 K followed by oxidation in air. Hydrolization to deprotection to give the title compound. And the single-crystal was obtained from EtOH at room temperature.

Computing details

Data collection: *XSCANS* (Siemens, 1994a); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1994b); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL94* (Sheldrick, 1994); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

(hz01)

Crystal data

$C_{11}H_{15}O_2P$	V = 1057.4 (3) Å^3
M_r = 210.20	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 5.910 (1) Å	μ = 0.23 mm^{-1}

b = 11.210 (2) Å	T = 292 (2) K
c = 15.961 (2) Å	0.60 × 0.40 × 0.40 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.009
Absorption correction: none	3 standard reflections
1681 measured reflections	every 97 reflections
1543 independent reflections	intensity decay: 3.8%
1358 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	No H atoms present
$wR(F^2)$ = 0.075	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 0.98	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1543 reflections	Absolute structure: Flack (1983)
136 parameters	Flack parameter: 0.05 (12)

References

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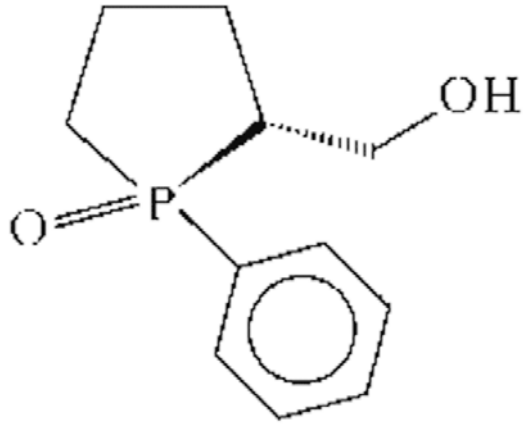
Scheme 1



supplementary materials

(hz01)

Crystal data

$C_{11}H_{15}O_2P$	D_x = 1.320 Mg m^{-3}
M_r = 210.20	Mo $K\alpha$ radiation λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 28 reflections
a = 5.910 (1) Å	θ = 3.9–16.8º
b = 11.210 (2) Å	μ = 0.23 mm^{-1}
c = 15.961 (2) Å	T = 292 (2) K
V = 1057.4 (3) Å^3	Irregular, colorless
Z = 4	0.60 × 0.40 × 0.40 mm
F_{000} = 448	

Data collection

Siemens P4 diffractometer	R_{int} = 0.009
Radiation source: normal-focus sealed tube	θ_{max} = 27.0º
Monochromator: graphite	θ_{min} = 2.2º
T = 292(2) K	h = 0→7
ω scans	k = −1→14
Absorption correction: none	l = −1→20
1681 measured reflections	3 standard reflections
1543 independent reflections	every 97 reflections
1358 reflections with $I > 2\sigma(I)$	intensity decay: 3.8%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	No H atoms present
$R[F^2 > 2\sigma(F^2)]$ = 0.030	Calculated $w = 1/[\sigma^2(F_o^2) + (0.0483P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$?
$wR(F^2)$ = 0.075	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 0.98	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1543 reflections	Extinction correction: SHELXL94, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
136 parameters	Extinction coefficient: 0.012 (2)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: 0.05 (12)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}

P	0.17237 (9)	0.07405 (5)	0.80816 (3)	0.0299 (2)
O1	0.3829 (2)	0.00070 (13)	0.80974 (11)	0.0443 (4)
O2	0.4631 (3)	0.3039 (2)	0.7728 (2)	0.0682 (6)
C1	−0.1381 (4)	0.1528 (2)	0.6888 (2)	0.0437 (6)
H1	−0.2303	0.1727	0.7338	0.052*
C2	−0.2102 (5)	0.1753 (3)	0.6078 (2)	0.0536 (7)
H2	−0.3486	0.2126	0.5987	0.064*
C3	−0.0785 (5)	0.1430 (2)	0.5410 (2)	0.0560 (7)
H3	−0.1276	0.1577	0.4866	0.067*
C4	0.1253 (5)	0.0889 (3)	0.5545 (2)	0.0595 (8)
H4	0.2132	0.0654	0.5091	0.071*
C5	0.2028 (4)	0.0686 (3)	0.63551 (14)	0.0483 (6)
H5	0.3434	0.0334	0.64407	0.058*
C6	0.0716 (3)	0.1005 (2)	0.70286 (13)	0.0320 (5)
C7	−0.0633 (4)	0.0166 (2)	0.86993 (14)	0.0395 (5)
H7A	−0.1991	0.0115	0.83599	0.047*
H7B	−0.0284	−0.0621	0.89153	0.047*
C8	−0.0964 (5)	0.1048 (2)	0.9416 (2)	0.0518 (7)
H8A	−0.2526	0.1037	0.9602	0.062*
H8B	−0.0004	0.0834	0.9885	0.062*
C9	−0.0346 (4)	0.2279 (2)	0.9095 (2)	0.0452 (6)
H9A	−0.0205	0.2832	0.9560	0.054*
H9B	−0.1512	0.2571	0.8720	0.054*
C10	0.1910 (4)	0.2176 (2)	0.86297 (14)	0.0346 (5)
C11	0.2531 (4)	0.3249 (2)	0.8109 (2)	0.0428 (5)
H11A	0.1385	0.3386	0.7685	0.051*
H11B	0.2619	0.3952	0.8462	0.051*
H2O	0.508 (8)	0.367 (3)	0.742 (2)	0.124 (15)*
H10	0.313 (4)	0.202 (2)	0.902 (2)	0.050 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P	0.0253 (2)	0.0291 (2)	0.0352 (3)	0.0033 (2)	0.0005 (3)	−0.0011 (3)
O1	0.0348 (8)	0.0398 (8)	0.0582 (10)	0.0112 (7)	−0.0023 (8)	−0.0016 (9)
O2	0.0498 (11)	0.0421 (10)	0.113 (2)	0.0014 (9)	0.0283 (12)	0.0165 (12)
C1	0.0366 (12)	0.0509 (13)	0.0438 (12)	0.0050 (11)	0.0006 (12)	0.0020 (12)
C2	0.045 (2)	0.063 (2)	0.0530 (14)	−0.0014 (14)	−0.0128 (13)	0.0153 (14)
C3	0.069 (2)	0.059 (2)	0.0406 (13)	−0.015 (2)	−0.0115 (13)	0.0085 (13)
C4	0.074 (2)	0.067 (2)	0.0373 (12)	0.002 (2)	0.0144 (13)	0.0009 (14)
C5	0.0459 (14)	0.0532 (14)	0.0457 (12)	0.0049 (14)	0.0108 (11)	0.0030 (14)
C6	0.0307 (9)	0.0306 (10)	0.0347 (10)	−0.0013 (9)	−0.0006 (9)	−0.0026 (9)
C7	0.0369 (12)	0.0391 (11)	0.0426 (12)	−0.0043 (11)	0.0018 (10)	0.0056 (11)
C8	0.0551 (15)	0.056 (2)	0.0441 (13)	0.0009 (14)	0.0160 (12)	0.0004 (12)
C9	0.0493 (15)	0.0451 (14)	0.0412 (12)	0.0059 (13)	0.0055 (13)	−0.0103 (11)
C10	0.0344 (12)	0.0320 (10)	0.0375 (11)	0.0006 (11)	−0.0048 (11)	−0.0041 (9)
C11	0.0400 (11)	0.0318 (11)	0.0565 (14)	0.0010 (10)	0.0014 (13)	−0.0013 (13)

Geometric parameters (Å, °)

P—O1	1.4916 (14)	C3—C4	1.365 (4)
P—C6	1.808 (2)	C4—C5	1.390 (3)
P—C7	1.824 (2)	C5—C6	1.373 (3)
P—C10	1.834 (2)	C7—C8	1.524 (3)
O2—C11	1.403 (3)	C8—C9	1.516 (4)
C1—C2	1.384 (3)	C9—C10	1.531 (3)
C1—C6	1.389 (3)	C10—C11	1.508 (3)
C2—C3	1.369 (4)		
O1—P—C6	112.39 (10)	C5—C6—C1	119.1 (2)
O1—P—C7	115.67 (10)	C5—C6—P	120.0 (2)
C6—P—C7	107.99 (10)	C1—C6—P	120.9 (2)
O1—P—C10	115.18 (10)	C8—C7—P	105.9 (2)
C6—P—C10	108.63 (10)	C9—C8—C7	107.9 (2)
C7—P—C10	95.60 (11)	C8—C9—C10	107.7 (2)
C2—C1—C6	120.2 (2)	C11—C10—C9	114.8 (2)
C3—C2—C1	120.2 (3)	C11—C10—P	116.9 (2)
C4—C3—C2	119.8 (2)	C9—C10—P	104.2 (2)
C3—C4—C5	120.7 (3)	O2—C11—C10	108.7 (2)
C6—C5—C4	120.0 (2)		
C6—C1—C2—C3	2.0 (4)	C6—P—C7—C8	−120.5 (2)
C1—C2—C3—C4	−0.4 (4)	C10—P—C7—C8	−8.8 (2)
C2—C3—C4—C5	−1.4 (4)	P—C7—C8—C9	32.5 (2)
C3—C4—C5—C6	1.5 (4)	C7—C8—C9—C10	−47.0 (3)
C4—C5—C6—C1	0.1 (4)	C8—C9—C10—C11	167.2 (2)
C4—C5—C6—P	180.0 (2)	C8—C9—C10—P	38.1 (2)
C2—C1—C6—C5	−1.8 (4)	O1—P—C10—C11	93.8 (2)
C2—C1—C6—P	178.3 (2)	C6—P—C10—C11	−33.2 (2)
O1—P—C6—C5	−8.8 (2)	C7—P—C10—C11	−144.3 (2)
C7—P—C6—C5	−137.6 (2)	O1—P—C10—C9	−138.3 (2)
C10—P—C6—C5	119.8 (2)	C6—P—C10—C9	94.6 (2)
O1—P—C6—C1	171.1 (2)	C7—P—C10—C9	−16.5 (2)
C7—P—C6—C1	42.3 (2)	C9—C10—C11—O2	179.4 (2)
C10—P—C6—C1	−60.3 (2)	P—C10—C11—O2	−58.1 (3)
O1—P—C7—C8	112.6 (2)		