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(3*S*)-4-Phenyl-3-(phenylaminooxy)butan-2-one

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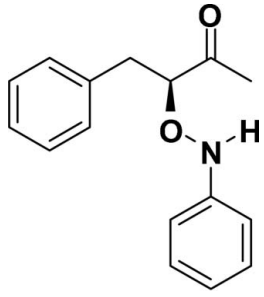
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.055; wR factor = 0.145; data-to-parameter ratio = 8.8.

The title compound, $C_{16}H_{17}NO_2$, is an intermediate in our work on chiral acyloins. The crystal structure shows that molecules are linked by N—H···O hydrogen bonds.

Related literature

For related literature, see: Córdova *et al.* (2004); Davis & Chen (1992); Hayashi *et al.* (2004); Momiyama *et al.* (2004).



Experimental

Crystal data

$C_{16}H_{17}NO_2$
M_r = 255.31
Orthorhombic, $P2_12_12_1$
a = 5.5950 (17) Å
b = 15.847 (5) Å
c = 16.156 (5) Å
V = 1432.4 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.48 × 0.18 × 0.09 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.751, T_{max} = 1.000
7191 measured reflections
1571 independent reflections
781 reflections with $I > 2\sigma(I)$
R_{int} = 0.148

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.145
S = 0.85
1571 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2···O1^{i}	0.86 (2)	2.18 (2)	3.038 (6)	173 (4)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997*b*).

The authors thank Mr Jie Sun of the Shanghai Institute of Organic Chemistry, China, for the X-ray measurements

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2064).

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supplementary materials

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(3*S*)-4-Phenyl-3-(phenylaminooxy)butan-2-one

L. Yang, H. Fang, H. Zheng and L.-L. Weng

Comment

Chiral α-hydroxy ketones (acyloins) are useful synthetic intermediates (Davis & Chen, 1992). In recent years, three groups have reported proline and proline analogue-catalyzed α-aminoxylation of ketones (Córdova *et al.*, 2004; Hayashi *et al.*, 2004; Momiyama *et al.*, 2004), those compounds can be converted to acyloins. In our work of chiral acyloins, we obtain the title compound (I), and here report its crystal structure. In the crystal packing of (I), the carbonyl O atom is engaged in H-bonding with the N—H of the adjacent molecule.

Experimental

The 4-phenyl-butan-2-one (10 mmol) was added to a mixture of nitrosobenzene (1 mmol) and *trans*-4-*tert*-butyldimethyl-siloxy-D-proline (30 mol %) in DMSO (4 ml). The reaction was quenched after 3 h of vigorous stirring at room temperature by addition of aqueous NH_4Cl solution. After work up, the residue was purified by silica gel chromatography. Colorless crystals were obtained by slow evaporation in petroleum and ethyl acetate at room temperature.

Refinement

The hydrogen atoms were generated geometrically (C—H = 0.93, 0.98, 0.97 or 0.96Å for phenyl, tertiary, methylene or methyl H atoms respectively, and N—H = 0.86 Å) were included in the refinement in the riding model approximation except the NH where all parameters were refined. The displacement parameters of methyl H atoms were set to 1.5 times U_{eq} of the equivalent isotropic displacement parameters of their parent atoms, while those of other CH atoms were set to 1.2 times. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assumed from the synthesis.

Figures

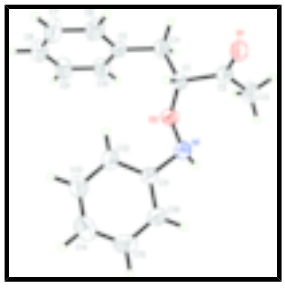


Fig. 1. *ORTEPIII* (Farrugia, 1997) plot of the title compound with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii.

(3*S*)-4-phenyl-3-(phenylaminooxy)butan-2-one

Crystal data

$C_{16}H_{17}NO_2$	F_{000} = 544
M_r = 255.31	D_x = 1.184 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 756 reflections
a = 5.5950 (17) Å	θ = 5.0–47.7º
b = 15.847 (5) Å	μ = 0.08 mm^{-1}
c = 16.156 (5) Å	T = 293 (2) K
V = 1432.4 (7) Å^3	Block, colourless
Z = 4	0.48 × 0.18 × 0.09 mm

Data collection

Bruker APEX area-detector diffractometer	1571 independent reflections
Radiation source: fine-focus sealed tube	781 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.148
T = 293(2) K	θ_{max} = 25.5º
φ scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −6→6
T_{min} = 0.751, T_{max} = 1.000	k = −19→17
7191 measured reflections	l = −19→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.145	$(\Delta/\sigma)_{max} < 0.001$
S = 0.85	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
1571 reflections	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
178 parameters	Extinction correction: none
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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}
O1	0.5061 (6)	0.4253 (2)	0.66177 (17)	0.0762 (10)
O2	0.0167 (5)	0.42246 (18)	0.52562 (16)	0.0597 (8)
N1	−0.0743 (7)	0.4977 (3)	0.5659 (2)	0.0633 (11)
C1	0.2625 (7)	0.4174 (3)	0.5431 (2)	0.0510 (11)
H1	0.3403	0.4699	0.5255	0.061*
C2	0.3146 (9)	0.4024 (3)	0.6342 (3)	0.0613 (12)
C3	0.1367 (10)	0.3571 (3)	0.6863 (3)	0.0830 (15)
H3A	0.0567	0.3153	0.6535	0.124*
H3B	0.0217	0.3966	0.7073	0.124*
H3C	0.2170	0.3302	0.7317	0.124*
C4	0.3649 (9)	0.3440 (3)	0.4935 (2)	0.0645 (13)
H4A	0.5313	0.3365	0.5086	0.077*
H4B	0.2801	0.2929	0.5085	0.077*
C5	0.3484 (8)	0.3561 (3)	0.4006 (3)	0.0536 (11)
C6	0.5259 (10)	0.3996 (3)	0.3615 (3)	0.0810 (16)
H6	0.6546	0.4199	0.3921	0.097*
C7	0.5168 (12)	0.4142 (3)	0.2759 (3)	0.0918 (18)
H7	0.6369	0.4449	0.2498	0.110*
C8	0.3291 (10)	0.3827 (3)	0.2312 (3)	0.0778 (15)
H8	0.3218	0.3917	0.1744	0.093*
C9	0.1530 (10)	0.3382 (3)	0.2696 (3)	0.0828 (16)
H9	0.0259	0.3166	0.2391	0.099*
C10	0.1640 (10)	0.3252 (3)	0.3547 (3)	0.0723 (14)
H10	0.0433	0.2948	0.3806	0.087*
C11	−0.1670 (8)	0.5573 (3)	0.5071 (3)	0.0559 (11)
C12	−0.0695 (9)	0.5646 (3)	0.4280 (3)	0.0732 (15)
H12	0.0569	0.5306	0.4113	0.088*
C13	−0.1665 (12)	0.6238 (3)	0.3757 (3)	0.0846 (17)
H13	−0.1084	0.6283	0.3219	0.101*
C14	−0.3446 (12)	0.6757 (3)	0.4005 (4)	0.0903 (18)
H14	−0.4065	0.7156	0.3642	0.108*
C15	−0.4343 (10)	0.6694 (3)	0.4799 (4)	0.0843 (17)
H15	−0.5564	0.7051	0.4972	0.101*

C16	−0.3424 (9)	0.6101 (3)	0.5332 (3)	0.0713 (13)
H16	−0.4004	0.6062	0.5870	0.086*
H2	−0.202 (6)	0.480 (3)	0.590 (3)	0.083 (19)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.064 (2)	0.116 (3)	0.0491 (18)	−0.007 (2)	−0.0015 (17)	0.0073 (19)
O2	0.0524 (18)	0.0665 (19)	0.0602 (18)	−0.0005 (17)	−0.0042 (16)	−0.0064 (16)
N1	0.065 (3)	0.077 (3)	0.049 (2)	0.010 (2)	−0.002 (2)	−0.006 (2)
C1	0.051 (3)	0.055 (3)	0.047 (3)	0.000 (2)	−0.005 (2)	0.000 (2)
C2	0.046 (3)	0.083 (3)	0.055 (3)	0.002 (3)	0.003 (2)	0.003 (3)
C3	0.085 (4)	0.104 (4)	0.060 (3)	−0.007 (4)	0.006 (3)	0.019 (3)
C4	0.084 (3)	0.059 (3)	0.050 (3)	0.008 (3)	−0.002 (3)	−0.001 (2)
C5	0.053 (3)	0.053 (2)	0.055 (3)	0.007 (3)	0.008 (2)	0.004 (2)
C6	0.085 (4)	0.093 (4)	0.065 (3)	−0.012 (4)	0.002 (3)	−0.012 (3)
C7	0.104 (5)	0.107 (4)	0.065 (4)	−0.015 (4)	0.011 (4)	0.009 (3)
C8	0.093 (4)	0.085 (4)	0.056 (3)	−0.007 (4)	−0.003 (3)	0.003 (3)
C9	0.090 (4)	0.095 (4)	0.063 (3)	−0.016 (4)	−0.014 (3)	0.001 (3)
C10	0.071 (3)	0.083 (3)	0.063 (3)	−0.011 (3)	−0.002 (3)	0.009 (3)
C11	0.053 (3)	0.067 (3)	0.048 (3)	0.005 (3)	−0.003 (2)	−0.001 (2)
C12	0.075 (4)	0.094 (4)	0.051 (3)	0.004 (3)	0.003 (3)	0.008 (3)
C13	0.104 (4)	0.092 (4)	0.058 (3)	0.010 (4)	−0.003 (3)	0.008 (3)
C14	0.103 (5)	0.079 (4)	0.089 (5)	−0.006 (4)	−0.016 (4)	0.016 (3)
C15	0.080 (4)	0.071 (3)	0.102 (5)	0.020 (3)	−0.002 (4)	−0.012 (3)
C16	0.067 (3)	0.079 (3)	0.067 (3)	0.010 (3)	0.006 (3)	−0.011 (3)

Geometric parameters (Å, °)

O1—C2	1.215 (5)	C7—C8	1.369 (7)
O2—C1	1.406 (4)	C7—H7	0.9300
O2—N1	1.451 (5)	C8—C9	1.361 (7)
N1—C11	1.436 (5)	C8—H8	0.9300
N1—H2	0.86 (2)	C9—C10	1.391 (7)
C1—C2	1.519 (6)	C9—H9	0.9300
C1—C4	1.524 (5)	C10—H10	0.9300
C1—H1	0.9800	C11—C16	1.357 (6)
C2—C3	1.488 (6)	C11—C12	1.394 (6)
C3—H3A	0.9600	C12—C13	1.374 (6)
C3—H3B	0.9600	C12—H12	0.9300
C3—H3C	0.9600	C13—C14	1.353 (7)
C4—C5	1.516 (6)	C13—H13	0.9300
C4—H4A	0.9700	C14—C15	1.380 (7)
C4—H4B	0.9700	C14—H14	0.9300
C5—C10	1.362 (6)	C15—C16	1.374 (7)
C5—C6	1.364 (6)	C15—H15	0.9300
C6—C7	1.404 (6)	C16—H16	0.9300
C6—H6	0.9300		

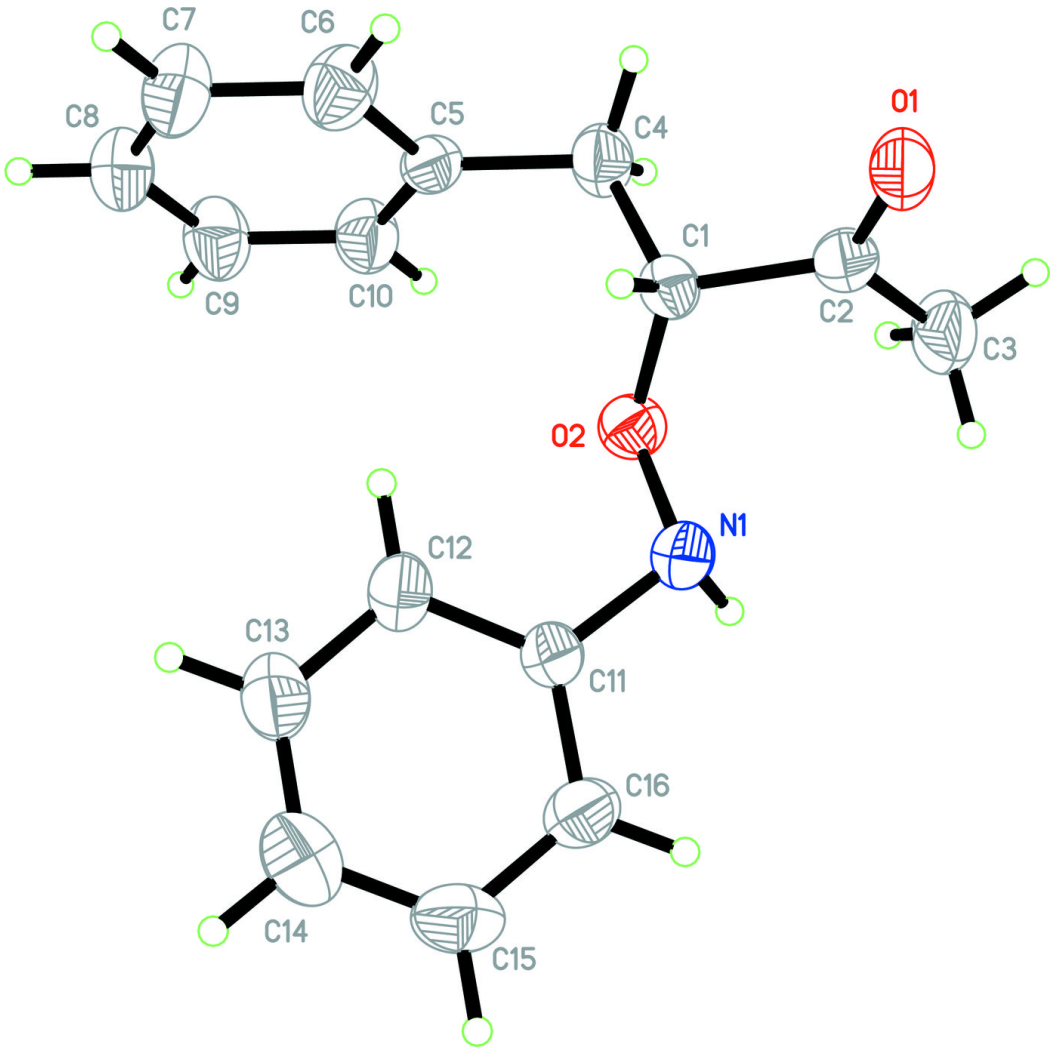
C1—O2—N1	107.4 (3)	C8—C7—C6	119.2 (5)
C11—N1—O2	111.7 (3)	C8—C7—H7	120.4
C11—N1—H2	103 (3)	C6—C7—H7	120.4
O2—N1—H2	103 (3)	C9—C8—C7	120.3 (5)
O2—C1—C2	113.0 (3)	C9—C8—H8	119.9
O2—C1—C4	107.8 (3)	C7—C8—H8	119.9
C2—C1—C4	108.5 (4)	C8—C9—C10	119.7 (5)
O2—C1—H1	109.1	C8—C9—H9	120.2
C2—C1—H1	109.1	C10—C9—H9	120.2
C4—C1—H1	109.1	C5—C10—C9	121.2 (5)
O1—C2—C3	121.7 (4)	C5—C10—H10	119.4
O1—C2—C1	118.5 (4)	C9—C10—H10	119.4
C3—C2—C1	119.7 (4)	C16—C11—C12	121.1 (5)
C2—C3—H3A	109.5	C16—C11—N1	117.5 (4)
C2—C3—H3B	109.5	C12—C11—N1	121.3 (4)
H3A—C3—H3B	109.5	C13—C12—C11	117.8 (5)
C2—C3—H3C	109.5	C13—C12—H12	121.1
H3A—C3—H3C	109.5	C11—C12—H12	121.1
H3B—C3—H3C	109.5	C14—C13—C12	121.5 (5)
C5—C4—C1	113.7 (4)	C14—C13—H13	119.2
C5—C4—H4A	108.8	C12—C13—H13	119.2
C1—C4—H4A	108.8	C13—C14—C15	120.0 (5)
C5—C4—H4B	108.8	C13—C14—H14	120.0
C1—C4—H4B	108.8	C15—C14—H14	120.0
H4A—C4—H4B	107.7	C16—C15—C14	119.6 (5)
C10—C5—C6	118.8 (4)	C16—C15—H15	120.2
C10—C5—C4	122.7 (4)	C14—C15—H15	120.2
C6—C5—C4	118.5 (5)	C11—C16—C15	119.9 (5)
C5—C6—C7	120.8 (5)	C11—C16—H16	120.0
C5—C6—H6	119.6	C15—C16—H16	120.0
C7—C6—H6	119.6		
C1—O2—N1—C11	117.7 (4)	C7—C8—C9—C10	0.3 (8)
N1—O2—C1—C2	66.1 (5)	C6—C5—C10—C9	−0.8 (7)
N1—O2—C1—C4	−174.0 (3)	C4—C5—C10—C9	179.3 (5)
O2—C1—C2—O1	−154.9 (4)	C8—C9—C10—C5	−0.1 (8)
C4—C1—C2—O1	85.6 (5)	O2—N1—C11—C16	152.2 (4)
O2—C1—C2—C3	27.9 (6)	O2—N1—C11—C12	−32.1 (6)
C4—C1—C2—C3	−91.6 (5)	C16—C11—C12—C13	−3.7 (7)
O2—C1—C4—C5	63.6 (5)	N1—C11—C12—C13	−179.3 (4)
C2—C1—C4—C5	−173.7 (4)	C11—C12—C13—C14	2.5 (8)
C1—C4—C5—C10	−94.4 (5)	C12—C13—C14—C15	−0.6 (8)
C1—C4—C5—C6	85.7 (5)	C13—C14—C15—C16	−0.2 (8)
C10—C5—C6—C7	1.5 (7)	C12—C11—C16—C15	3.0 (7)
C4—C5—C6—C7	−178.6 (5)	N1—C11—C16—C15	178.8 (4)
C5—C6—C7—C8	−1.3 (8)	C14—C15—C16—C11	−1.0 (7)
C6—C7—C8—C9	0.4 (8)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2···O1^{i}	0.86 (2)	2.18 (2)	3.038 (6)	173 (4)

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1



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(3*S*)-4-Phenyl-3-(phenylaminooxy)butan-2-one. Corrigendum

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In the paper by Yang, Fang, Zheng & Weng [*Acta Cryst.* (2007), E**63**, o3568], the correspondence author is marked incorrectly. The correct correspondence author and e-mail address are given here.

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