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(1*R*,4*R*)-1-(Hydroxydiphenylmethyl)-7,7-dimethylbicyclo[2.2.1]heptan-2-one

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Key indicators

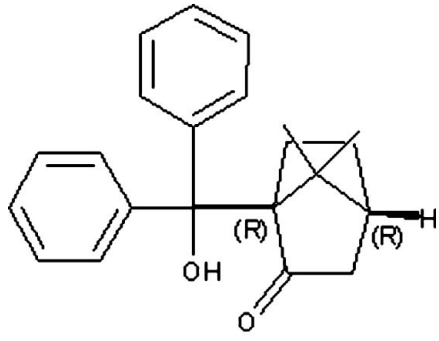
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.150
Data-to-parameter ratio = 8.8

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

The title compound, $C_{22}H_{24}O_2$, has been obtained by a Grignard reaction of (1*S*,4*R*)-methyl 7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carboxylate with phenylmagnesium bromide. Intramolecular hydrogen bonding is observed between the carbonyl group and the hydroxy group.

Comment

The title compound, (I), is an important intermediate in the synthesis of *exo*-10,10-diphenyl-2,10-camphanediol, which is widely used in asymmetric reduction as a chiral auxiliary (Chen *et al.*, 1999). The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are in agreement with those reported for similar compounds (Chen *et al.*, 2006). The dihedral angle between the phenyl planes is 72.10 (6)°.



(I)

Intramolecular hydrogen bonding is observed between carbonyl groups and hydroxy groups; O2—H2*A* = 0.82 Å, H2*A*···O1 = 2.02 Å, O2···O1 = 2.722 (3) Å and O2—H2*A*···O1 = 143°.

Experimental

To a precooled solution of (1*S*,4*R*)-methyl 7,7-dimethyl-2-oxobicyclo[2.2.1]heptane-1-carboxylate (0.78 g, 4 mmol) in dry tetrahydrofuran (30 ml) at 273 K was added dropwise a 3 *M* solution of phenylmagnesium bromide (6.8 ml, 20 mmol) in tetrahydrofuran. The cooling bath was removed and the mixture warmed to 308 K for 10 h. The reaction was quenched with a saturated NH_4Cl solution (16 ml) and extracted with ethyl acetate. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, concentrated under vacuum and the crude product was purified by column chromatography (petroleum ether–ethyl acetate, 30:1) to give the title compound as a white solid in 58% yield. Single crystals of (I) were obtained by slow evaporation of a petroleum ether–ethyl acetate solution (15:1 *v*/*v*).

Crystal data

$C_{22}H_{24}O_2$
M_r = 320.41
Orthorhombic, $P2_12_12_1$
a = 9.168 (2) Å
b = 10.053 (2) Å
c = 18.405 (4) Å
V = 1696.3 (6) Å^3
Z = 4
D_x = 1.255 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 (2) K
Chunk, colorless
0.65 × 0.56 × 0.41 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: none
9220 measured reflections
1912 independent reflections
1848 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.150
S = 1.19
1912 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 0.1778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in idealized positions, with C—H = 0.93 (aromatic), 0.98 (methine), 0.97 Å (methylene) and O—H = 0.82 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration of (I) was assigned assuming that the absolute configuration of the starting material was retained during the synthesis.

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

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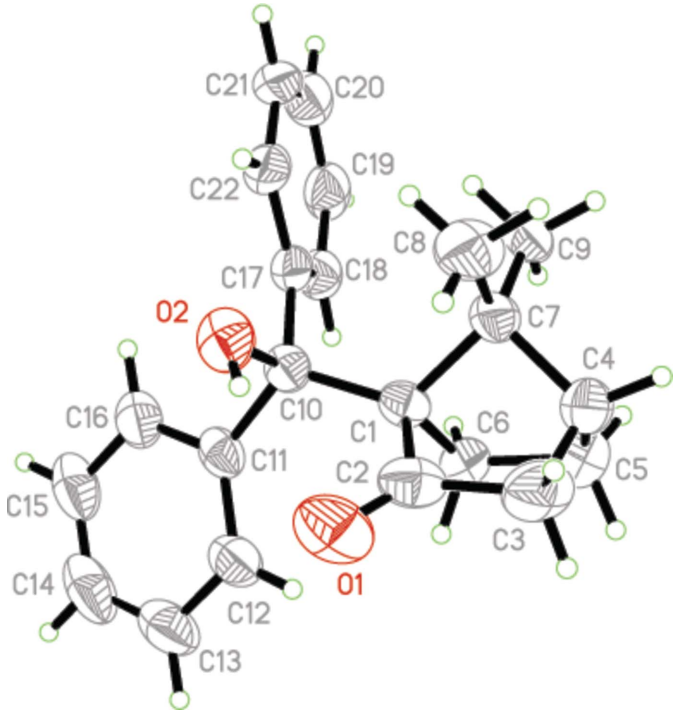

Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

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