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(2*S*)-1-(4-Bromophenyl)-2-hydroxy-2-[(2*S*,5*R*)-5-(1-hydroxy-1-methylethyl)-tetrahydrofuran-2-yl]ethanone

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Key indicators

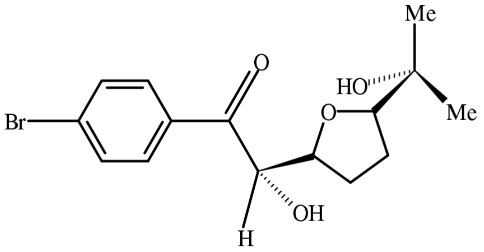
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.015 Å
R factor = 0.069
wR factor = 0.193
Data-to-parameter ratio = 10.0

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

The title compound, $C_{15}H_{19}BrO_4$, was synthesized during studies on the oxidation of 1,5-dienes. The molecular structure exhibits *R* and *S* chiral centres at the 2- and 5- positions on the central tetrahydrofuran moiety, and an *S* centre at the hydroxyethanone pivot atom. Intermolecular hydrogen bonding gives rise to a one-dimensional-chain structure.

Comment

During the development of chiral phase-transfer catalysed permanganate oxidations of 1,5-dienes the title compound, (I), was prepared (Brown & Keily, 2001). The molecular structure is shown in Fig. 1. The structure is composed of a tetrahydrofuran (thf) ring substituted at the 2- and 5-positions by bromophenylhydroxyethanone and hydroxymethylethyl groups, respectively. The geometric parameters conform to standard values derived from a systematic study of similarly hybridized atoms in the Cambridge Structural Database (Allen & Kennard, 1993; Allen *et al.*, 1992).



(I)

The 2 position in the thf ring (C7) exhibits *R* chirality and the 5 position (C4) is *S*, whilst C8 is a centre of chirality (= *S*). The thf ring exhibits an envelope conformation about C5. Hydrogen bonding exists between the two hydroxyl groups in the structure where the donor–acceptor distance of O3—H3$\cdots$O1^{i} is 2.722 (6) Å [symmetry code: (i) $-x+1$, $y+\frac{1}{2}$, $-z+\frac{5}{2}$]. Moreover, the hydroxyl group involving O3 is involved in a second, weaker intermolecular hydrogen bond, with a donor–acceptor distance C8—H8$\cdots$O3^{i} of 3.475 Å. These interactions form a one-dimensional-chain network in the crystal structure.

Experimental

(*E*)-1-(4-Bromophenyl)-7-methylocta-2,6-dien-1-one was oxidized by potassium permanganate under solid–liquid phase-transfer conditions using a chiral quaternary ammonium salt. Following a standard work-up and column chromatography, the product was recrystallized from ethanol/hexane to give colourless crystals of the title compound.

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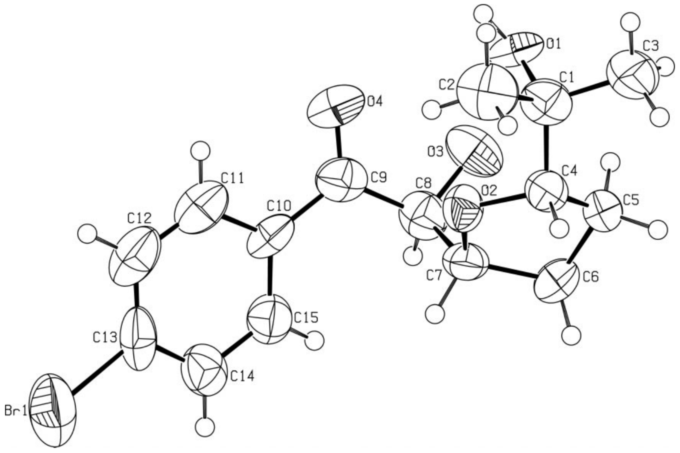

Figure 1
View of (I) with 50% probability displacement ellipsoids.

Crystal data

$C_{15}H_{19}BrO_4$
$M_r = 343.21$
Orthorhombic, $P2_12_12_1$
$a = 7.7184\ (15)$ Å
$b = 7.8674\ (16)$ Å
$c = 25.374\ (5)$ Å
$V = 1540.8\ (5)$ Å^3
$Z = 4$
$D_x = 1.48$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1626 reflections
$\theta = 2.9$–$27.5°$
$\mu = 2.68$ mm^{-1}
$T = 120\ (2)$ K
Plate, colourless
$0.30 \times 0.20 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1997)
$T_{\min} = 0.501$, $T_{\max} = 0.878$
2895 measured reflections
1851 independent reflections
1051 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 27.4°$
$h = -8 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.193$
$S = 1.04$
1851 reflections
186 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0957P)^2 + 0.1591P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.015$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.013 (3)
Absolute structure: Flack (1983)
Flack parameter = 0.00 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H3$\cdots$O1$^{\rm i}$	0.84	1.90	2.722 (9)	167
C8—H8$\cdots$O3$^{\rm i}$	1.00	2.60	3.475 (8)	146

Symmetry code: (i) $1 - x, \frac{1}{2} + y, \frac{5}{2} - z$.

H atoms were observed in a difference map, but were included in idealized positions with coordinates and thermal parameters riding on those of the parent atom. Refinement of a Flack parameter (Flack, 1983) indicated that the correct absolute structure had been identified.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

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