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cis-(3*R*,5*R*)-3-Bromo-2,2-dimethyl-5-phenyltetrahydropyran

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.007 Å
R factor = 0.031
wR factor = 0.073
Data-to-parameter ratio = 10.9

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

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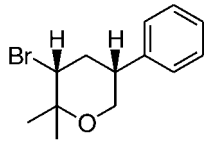
The heterocyclic ring in the title compound, $C_{13}H_{17}BrO$, adopts a $_1C^4$ conformation, with the phenyl and bromo substituents located in equatorial positions.

Comment

Positions 3 and 5 in *cis*-3-bromo-2,2-dimethyl-5-phenyltetrahydropyran, (I), are stereogenic centres. The racemate of (I) was prepared (Hartung *et al.*, 2003) and investigated by X-ray diffraction, in order to establish reference data for an upcoming conformational analysis of multiply substituted tetrahydropyrans.



(I)

Atoms O1 and C4 are displaced in opposite direction [−0.649 (6) Å for O1 and 0.690 (7) Å for C4] from the plane defined by atoms C2, C3, C5, and C6 [deviation of C6 = 0.01 (1) Å], thus leading to a $_1C^4$ arrangement of the tetrahydropyran ring (Fig. 1). This arrangement corresponds to the major conformer of (I) in $CDCl_3$ solution, according to an analysis of the vicinal proton–proton coupling constants (^{1}H NMR) (Hartung *et al.*, 2003). The phenyl and bromo substituents in (I) are equatorially arranged [Br1—C3—C2—O1 = −179.8 (3)° and C9—C5—C4—C3 = 176.4 (4)°]. The absolute value of the six endocyclic torsion angles sum to 349 (3)° for the tetrahydropyran subunit in (I). The racemate of 3-bromosubstituted tetrahydropyran (I) crystallizes as an enantiomorphic conglomerate in $P2_12_12$ (orthorhombic). The absolute 3*R*,5*R* configuration of (I) in the investigated crystal was established with the aid of the Flack (1983) parameter [−0.009 (19)]. No significant close contacts were found in the crystal structure of the brominated tetrahydropyran (I).

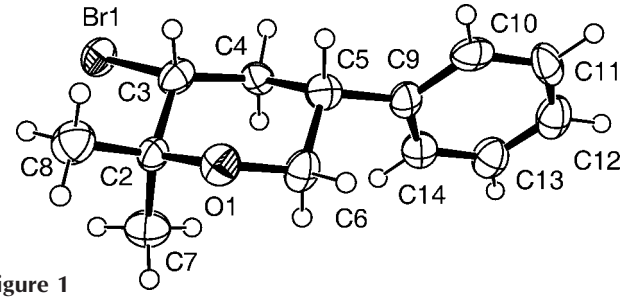


Figure 1
The molecular structure of (I). Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Crystals suitable for X-ray diffraction were obtained from a saturated solution of (I) in diethyl ether, which was kept in an atmosphere of petroleum ether.

Crystal data

$C_{13}H_{17}BrO$
$M_r = 269.18$
Orthorhombic, $P2_12_12$
$a = 13.269\ (2)$ Å
$b = 16.085\ (2)$ Å
$c = 5.7278\ (4)$ Å
$V = 1222.5\ (3)$ Å^3
$Z = 4$
$D_x = 1.462$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 3.34$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.402$, $T_{max} = 0.513$
2915 measured reflections
1598 independent reflections
1360 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 22.5^\circ$
$h = -14 \rightarrow 8$
$k = 0 \rightarrow 17$
$l = -6 \rightarrow 6$
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.073$
$S = 1.07$
1598 reflections
146 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.2533P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: Flack (1983), 628 Friedel pairs
Flack parameter = −0.009 (19)

H atoms attached to C3 and C5 were located in a difference Fourier map and their positions were refined freely with isotropic displacement parameters. All other H atoms were placed in geometrically idealized positions (C—H = 0.95–0.99 Å), and refined as riding, with $U_{iso}(H) = 1.2$ or 1.5 times $U_{eq}(C)$.

Data collection: *CAD*-4 *Diffractometer Control Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *Diffractometer Control Software*; data reduction: *CAD*-4 *Diffractometer Control Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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