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(1′*S*,2′*S*,5′*R*)-3-(2-Bromopropionyl)-2′-isopropyl-5′-methylspiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one

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The absolute configuration of the title compound, $C_{20}H_{26}BrNO_3$, was determined from both the synthetic precursor and anomalous scattering effects. In the crystal structure, non-classical C—H···O hydrogen bonds link the molecules into a sheet parallel to the *b* axis.

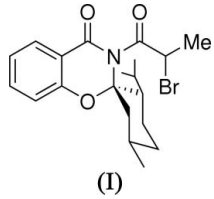
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.069
Data-to-parameter ratio = 19.0

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

Comment

The molecular structure of (I) is shown in Fig. 1. The absolute configuration was found to be the same as that of the starting material; thus the chiral centres were not affected by the reaction. The compound crystallizes in space group $P2_12_12_1$ with one molecule in the asymmetric unit. In the crystal structure, non-classical C—H···O hydrogen bonds play an important role (Table 1). C9—H9···O1^{ii} links the planar parts of the molecules into sheets parallel to the *b* axis, while C2—H2···O3^{i} links the molecules into chains parallel to the *a* axis.



(I)

Experimental

To a mixture of (1′*S*,2′*S*,5′*R*)-5′-methyl-2′-isopropylspiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one (273 mg, 1 mmol), pyridine (95 mg, 1.2 mmol) and toluene (10 ml) was added 2-bromopropanoyl

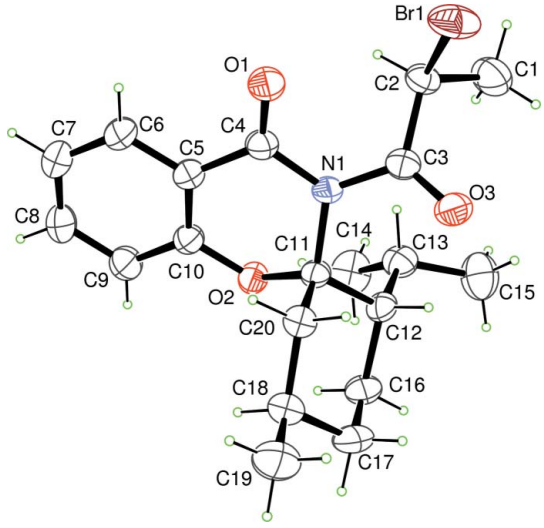


Figure 1
The molecular structure of compound (I); displacement ellipsoids are drawn at the 50% probability level.

bromide (260 mg, 1.2 mmol) dropwise at 278–288 K. This mixture was stirred at the same temperature for 30 min and then at 298 K for 20 h. The reaction mixture was poured into water (10 ml). The organic layer was washed successively with saturated aqueous $NaHCO_3$ (5 ml) and brine (5 ml), dried over anhydrous Na_2SO_4, and evaporated *in vacuo*. The residue was dissolved in 2-propanol (3 ml) at 323–325 K, gradually cooled to 283 K and stirred at the same temperature for 1 h. The resulting crystals were collected, washed with 2-propanol (3 ml) and dried at 313 K for 20 h to afford 357 mg (86% yield) of product. Colourless crystals of (I) were obtained from a CH_2Cl_2/EtOH (1:10 *v*/*v*) solution after leaving it to stand for 4 d.

Crystal data

$C_{20}H_{26}BrNO_3$
M_r = 408.33
Orthorhombic, $P2_12_12_1$
a = 8.7388 (3) Å
b = 14.0200 (8) Å
c = 15.9341 (7) Å
V = 1952.21 (16) Å^3
Z = 4
D_x = 1.389 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.12 mm^{-1}
T = 295 (2) K
Block, colourless
0.36 × 0.30 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.438, T_{max} = 0.575
18127 measured reflections
4380 independent reflections
3817 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.069
S = 1.04
4380 reflections
230 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2 + 0.3642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Absolute structure: Flack (1983), 1827 Friedel pairs
Flack parameter: 0.001 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O3^{i}	0.98	2.37	3.187 (2)	141
C9—H9···O1^{ii}	0.93	2.62	3.430 (3)	146

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(C), and were allowed to rotate freely about the C—C bonds. The other H atoms were placed in calculated positions (C—H = 0.93–0.98 Å), with U_{iso}(H) = 1.2U_{eq}(carrier atom), and included in the final cycles of refinement in the riding-model approximation.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

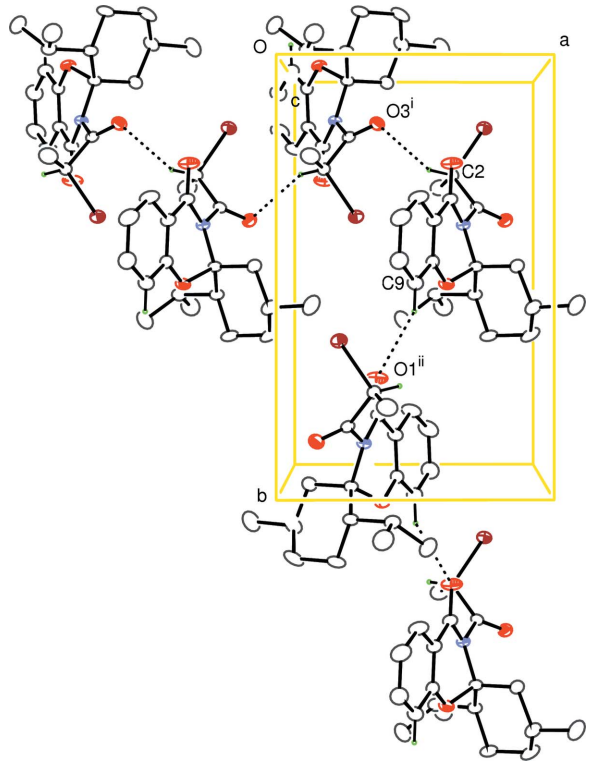


Figure 2
The molecular packing of (I), viewed along the *c* axis. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (ii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.]

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