Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[(2*R**,3*S**)-3-(2-Methoxyanilino)-2-methyl-3-phenylpropionyl]spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.063
Data-to-parameter ratio = 8.0

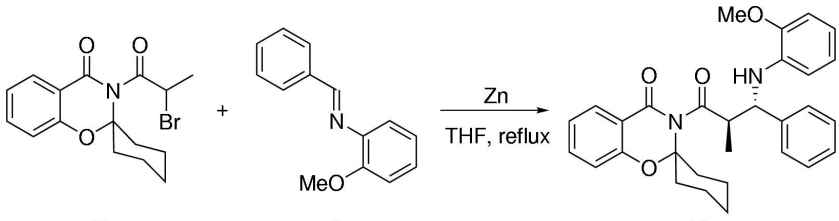
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 December 2004
Accepted 8 March 2005
Online 18 March 2005

The racemic title compound, $C_{30}H_{32}N_2O_4$, was synthesized from spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one and *N*-benzylidene-2-methoxybenzenamine under classical Reformatsky reaction conditions. Intermolecular N—H···O and C—H···O hydrogen bonds link the molecules in the crystal structure into infinite chains along the *a* axis. These chains form layers parallel to the (001) plane, which are stabilized by weak interlayer C—H···O hydrogen bonds.

Comment

Fig. 1 shows the structure of (3). Its preparation involved refluxing a mixture of (1) and (2) in the presence of zinc powder (Jian *et al.*, 2005). The mechanism predicts that the product is a racemic mixture of 3-[(2*S*,3*R*)- and 3-[(2*R*,3*S*)-3-(2-methoxyanilino)-2-methyl-3-phenylpropionyl]spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one. The X-ray crystal structure, reported here, confirms that the mechanism is valid.



(1) (2) (3)

The molecular dimensions (Table 1) may be considered normal. Intermolecular N—H···O and C—H···O hydrogen

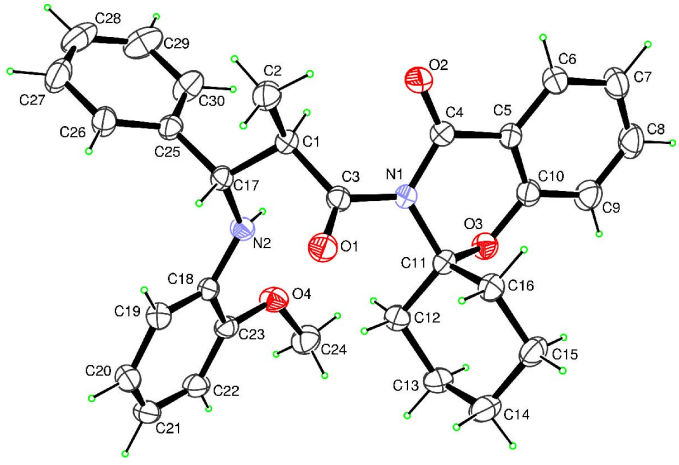


Figure 1
The molecule of compound (3) in the crystal structure. Ellipsoids are drawn at the 30% probability level.

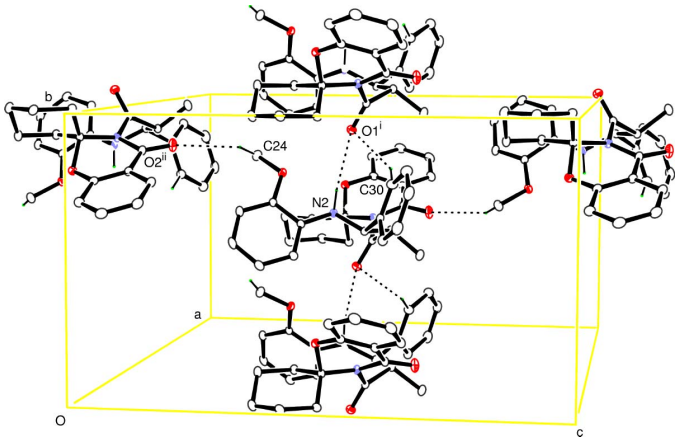


Figure 2
The molecular packing of (3), viewed approximately along the *a* axis. Dashed lines indicate the hydrogen-bonding interactions. H atoms not involved in the hydrogen bonding have been omitted (see Table 2 for symmetry codes).

bonds play an important role in the formation of polymeric chains running along the crystallographic *b* axis (Fig. 2). These chains form layers parallel to the (001) plane. These layers are stabilized by weak interlayer C—H···O hydrogen bonds (Table 2).

Experimental

A mixture of the carboximide (1) (1.2 mmol), the imine (2) (1 mmol) and zinc dust (2 mmol) in tetrahydrofuran (5 ml) was refluxed for 10–20 min, cooled and poured into water (5 ml), and then extracted with CH_2Cl_2 (3 × 5 ml). The combined extracts were washed with brine, dried over anhydrous Na_2SO_4, and evaporated *in vacuo*. The residue was purified by flash column chromatography on silica gel (eluted with hexane/ethyl acetate, 20:1) to give the desired product (402 mg, yield 83%). Colourless crystals were obtained from CH_2Cl_2/EtOH solution after it was left to stand for 4 d. IR (KBr): 3380, 1718, 1678 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 1.08 (*d*, *J* = 7.9 Hz), 1.40–2.30 (*m*, 10H), 3.61 (*qd*, 1H, *J* = 7.9 Hz, *J* = 9.9 Hz), 3.83 (*s*, 3H), 4.60 (*d*, 1H, *J* = 9.9 Hz), 6.47–8.05 (*m*, 13H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.79, 22.56, 22.65, 24.55, 32.79, 33.10, 51.08, 55.96, 61.97, 95.88, 110.02, 111.18, 116.34, 117.43, 117.74, 121.42, 122.48, 127.57, 127.77, 128.60, 136.15, 137.22, 141.82, 147.03, 155.68, 164.02, 183.26. ESI–MS: *m*/*z* 485 ($[M + 1]^+$); HRMS (ESI) *m*/*z* found for $[M + H]^+$ 485.2438, calculated for $C_{30}H_{33}N_2O_4^+$ 485.2435.

Crystal data

$C_{30}H_{32}N_2O_4$	Mo $K\alpha$ radiation
M_r = 484.59	Cell parameters from 42 864 reflections
Orthorhombic, *Pbcn*	θ = 1.4–27.5°
a = 24.1025 (4) Å	μ = 0.08 mm^{-1}
b = 11.0675 (5) Å	*T* = 295 (1) K
c = 19.0716 (2) Å	Block, colourless
V = 5087.4 (3) $Å^3$	0.16 × 0.10 × 0.08 mm
Z = 8	
D_x = 1.265 Mg m^{-3}	

Data collection

Rigaku R-AXIS RAPID diffractometer	2604 reflections with $F^2 > 2\sigma(F^2)$
ω scans	R_{int} = 0.061
Absorption correction: none	θ_{max} = 27.5°
43 304 measured reflections	*h* = −31 → 31
5694 independent reflections	*k* = −14 → 12
	l = −24 → 24

Refinement

Refinement on F^2	$w = 1/[0.0003F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)]$ = 0.031	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.063	$\Delta\rho_{max}$ = 0.12 e $Å^{-3}$
S = 1.03	$\Delta\rho_{min}$ = −0.13 e $Å^{-3}$
2604 reflections	Extinction correction: Larson (1970), equation 22
326 parameters	Extinction coefficient: 2.4 (3) × 10^2
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.213 (2)	N1—C11	1.510 (2)
O2—C4	1.219 (2)	N2—C17	1.457 (2)
O3—C10	1.372 (2)	C1—C2	1.530 (2)
O3—C11	1.443 (2)	C1—C3	1.514 (2)
O4—C23	1.374 (2)	C1—C17	1.537 (2)
O4—C24	1.420 (2)	C4—C5	1.465 (3)
N1—C3	1.430 (2)	C5—C10	1.377 (3)
O1—C3—N1	120.4 (2)	O3—C11—C12	103.8 (1)
O3—C11—N1	106.2 (1)	C3—C1—C2	110.3 (1)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H32···O1^{i}	0.90	2.44	3.329 (2)	168
C30—H31···O1^{i}	0.98	2.48	3.307 (3)	142
C24—H24···O2^{ii}	0.96	2.69	3.544 (2)	148

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, z$; (ii) $\frac{1}{2} - x, \frac{3}{2} - y, z - \frac{1}{2}$.

Atom H32 was found in a difference Fourier map and fixed in position. The other H atoms were placed in calculated positions, with C—H = 0.96 or 0.97 Å, and included in the final cycles of refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atoms.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO* ; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

The authors thank the National Natural Science Foundation of China (grant No. 20272051), as well as the Teaching and Research Award Programme for Outstanding Young Teachers in Higher Education Institutions of MOE, China.

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