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3-[(2*R**,3*S**)-3-(4-Chlorophenyl)-3-(2-methoxyanilino)-2-methylpropionyl]spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one

Shan-Zhong Jian,[a] Jian-Ming Gu[b] and Yan-Guang Wang[a]*

[a]Department of Chemistry, Zhejiang University, 310027 Hangzhou, People's Republic of China, and [b]Analysis and Measurement Center, Zhejiang University, 310028 Hangzhou, People's Republic of China

Correspondence e-mail: orgwyg@zju.edu.cn

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Key indicators

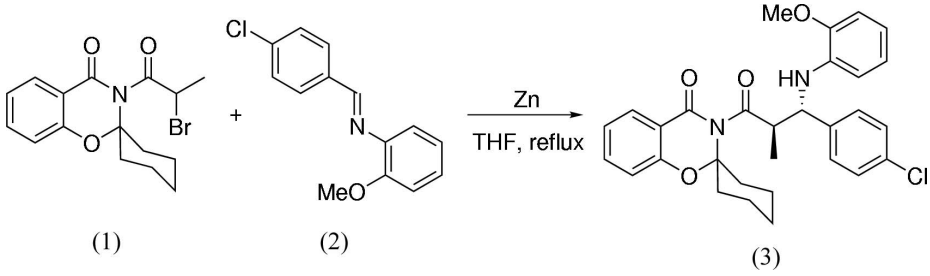
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 15.6

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

The title compound, $C_{30}H_{31}ClN_2O_4$, crystallizes as discrete molecules. Non-classical C—H···O hydrogen bonds link the molecules in the crystal structure into a sheet parallel to ($\bar{1}$01).

Comment

Fig. 1 shows the structure of (3). Selected molecular parameters and hydrogen-bond geometric characteristics are listed in Tables 1 and 2, respectively.



(1) (2) (3)

The compound crystallizes in the monoclinic space group $P2_1/n$ with four symmetry-equivalent molecules per unit cell. In the crystal structure, non-classical C—H···O hydrogen bonds play an important role, resulting in the formation of a polymeric sheet parallel to ($\bar{1}$01).

Experimental

A mixture of spiro[2*H*-1,3-benzoxazine-2,1′-cyclohexan]-4(3*H*)-one, (1) (422 mg, 1.2 mmol), *N*-(4-chlorobenzylidene)-2-methoxybenzenamine, (2) (246 mg, 1 mmol), and zinc dust (130 mg, 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 (eluting with hexane/ethyl acetate, 20:1) to give the desired product (440 mg, yield 85%). Colorless crystals were obtained from a CH_2Cl_2/EtOH solution after allowing it to stand for 4 d. IR (KBr): 3388, 1718, 1678 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.08 (*d*, *J* = 6.9 Hz), 1.40–2.30 (*m*, 10H), 3.54 (*qd*, 1H, *J* = 6.9 Hz, *J* = 9.9 Hz), 3.83 (*s*, 3H), 4.57 (*d*, 1H, *J* = 9.9 Hz), 5.90 (*b*, 1H), 6.35–8.04 (*m*, 12H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.79, 22.56, 22.61, 24.53, 32.84, 33.10, 50.78, 55.94, 61.35, 95.85, 110.05, 111.16, 116.68, 117.45, 117.64, 121.36, 122.53, 128.58, 128.82, 129.12, 136.24, 136.90, 140.48, 147.08, 155.65, 164.06, 182.85. ESI–MS: *m*/*z* 519 ([*M* + 1]$^+$), HRMS (ESI) *m*/*z* found for [*M* + H]$^+$ 519.2037, calculated for $C_{30}H_{32}ClN_2O_4^+$ 519.2045.

Crystal data

$C_{30}H_{31}ClN_2O_4$
M_r = 519.04
Monoclinic, $P2_1/n$
a = 14.7792 (3) Å
b = 16.7390 (2) Å
c = 14.6920 (3) Å
β = 132.0900 (5)°
V = 2697.24 (8) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
Mo Kα radiation
Cell parameters from 13437 reflections
θ = 2.2–27.5°
μ = 0.18 mm^{-1}
T = 293 (1) K
Block, colorless
0.33 × 0.30 × 0.17 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.925, T_{max} = 0.970
25182 measured reflections
6039 independent reflections
5221 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.028
θ_{max} = 27.5°
$h = -19 \rightarrow 19$
$k = -21 \rightarrow 21$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.01
5221 reflections
335 parameters
H-atom parameters constrained
$w = 1/[0.0009F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 2.3 (4) × 10^2

Table 1
Selected geometric parameters (Å, °).

O2—C5	1.223 (3)	N1—C4	1.432 (3)
O3—C11	1.357 (2)	N1—C5	1.385 (2)
O3—C12	1.446 (3)	N1—C12	1.493 (3)
O4—C23	1.361 (3)	N2—C1	1.460 (1)
O4—C24	1.431 (2)	N2—C18	1.370 (3)
C5—N1—C4	117.5 (2)	C12—N1—C5	118.6 (2)
C12—N1—C4	123.1 (1)	C18—N2—C1	123.2 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H201···O2	0.91	2.37	3.251 (3)	161
N2—H201···O4	0.91	2.36	2.609 (1)	96
C24—H243···O1^{i}	0.96	2.46	3.287 (2)	144
C24—H241···O4^{ii}	0.96	2.62	3.259 (2)	124

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

Atom H201 was found in a difference Fourier map and included in the final cycles of refinement as riding. The other H atoms were placed in calculated positions (C—H = 0.96–0.98 Å), with U_{iso}(H) = 1.2U_{eq} of the carrier atom, and included in the final cycles of refinement as riding.

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 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* (Watkin *et al.*, 1996); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

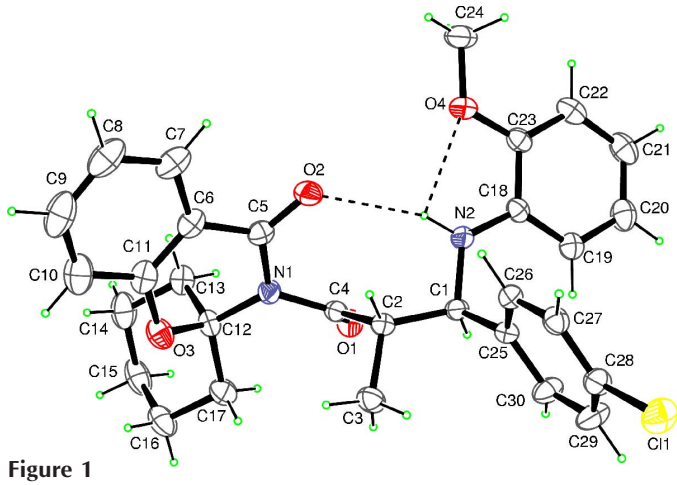


Figure 1
The molecule of compound (3) in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate intramolecular hydrogen bonds.

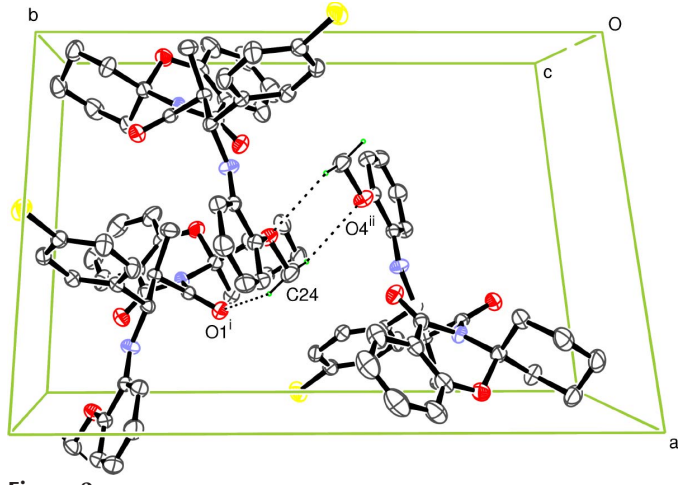


Figure 2
The molecular packing of (3), viewed along the *c* axis. Dashed lines indicate the intermolecular hydrogen-bonding interactions (see Table 2 for symmetry codes). H atoms not involved in hydrogen bonding have been omitted.

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