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3-(3-Bromobenzyl)isoquinolin-1(2*H*)-one

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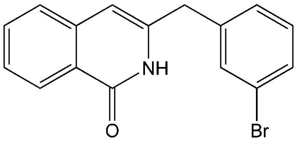
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.026; wR factor = 0.068; data-to-parameter ratio = 15.0.

In the title compound, $C_{16}H_{12}BrNO$, the ring systems subtend an interplanar dihedral angle of 75.95 (3)°. In the crystal packing, molecules are linked to form centrosymmetric pairs by pairs of classical N—H⋯O hydrogen bonds.

Related literature

For the biological and pharmaceutical properties of isoquinolin-1(2*H*)-one derivatives, see: Chern & Li (2004); Coelho *et al.* (2003); Jayaraman *et al.* (2000); Thompson & Kallmerten (1990); Ukita *et al.* (2001). For the structure of a related isochromene derivative, see: Ali *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{12}BrNO$
M_r = 314.18
Triclinic, $P\bar{1}$
a = 4.5858 (4) Å
b = 9.4976 (7) Å
c = 14.8296 (11) Å
α = 88.698 (6)°
β = 83.829 (6)°
γ = 86.529 (6)°
V = 640.88 (9) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 4.28 mm^{-1}
T = 100 K
0.16 × 0.07 × 0.07 mm

Data collection

Oxford Diffraction Nova A diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.817, T_{max} = 1.000
9267 measured reflections
2642 independent reflections
2586 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.068
S = 0.90
2642 reflections
176 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N—H01⋯O^i	0.85 (3)	1.96 (3)	2.8036 (19)	176 (2)

Symmetry code: (i) $-x, -y+1, -z+1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2396).

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supporting information

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3-(3-Bromobenzyl)isoquinolin-1(2*H*)-one

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S1. Comment

Isoquinolin-1(2*H*)-one derivatives are an important class of heterocyclic compounds with substantial biological activities (Jayaraman *et al.*, 2000) that can be found in naturally occurring products of medicinal interest (Ukita *et al.*, 2001) and synthetic pharmaceuticals such as thalifoline (Chern & Li, 2004), pancratistain and lycoricidine (Thompson & Kallmerten, 1990). In addition, isoquinolin-1(2*H*)-ones are versatile building blocks for the total synthesis of natural isocarbostyril alkaloids (Coelho *et al.*, 2003). Bearing in mind the pharmaceutical importance of this class of compounds, the title compound, an isoquinolinone derivative containing a 3-bromobenzyl substituent, has been synthesized and its crystal structure is reported here. We have also determined the structure of the analogous isochromene derivative with an oxygen atom replacing the NH group (Ali *et al.*, 2009).

The molecule of the title compound is shown in Fig. 1. Bond lengths and angles may be regarded as normal. The atom sequence N—C2—C10—C11 displays a *trans* geometry, with a torsion angle of -178.69 (14). The two planar ring systems (including all non-hydrogen substituents) are both planar to within r.m.s. deviations of 0.01 Å and subtend an interplanar angle of 75.95 (3)°. As in the analogous isochromene derivative (Ali *et al.*, 2009), several bond angles depart substantially from ideal values, *e.g.* C1—N—C2 125.36 (14), N—C2—C10 113.15 (14), C2—C2—C10 126.87 (15), C2—C10—C11 114.88 (14)°.

The packing diagram (Fig. 2) shows the molecules to be linked by classical hydrogen bonds N—H···OC across inversion centres.

S2. Experimental

3-(3′-Bromobenzyl)isocoumarin (1 g, 0.0032 mol) in 2-ethoxyethanol was saturated with ammonia gas for 2 h, forming a pale yellow solution that was refluxed for 2 h. The solvent was evaporated under reduced pressure, yielding a fluffy solid. This that was purified by column chromatography using 50% ethyl acetate/petroleum ether as an eluent to afford the title compound (yield 61%; m.p. 228–230 °C). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

S3. Refinement

The H atom bount to the nitrogen atom was refined freely. Other H atoms were placed in calculated positions and refined using a riding model with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 U_{eq}(C).

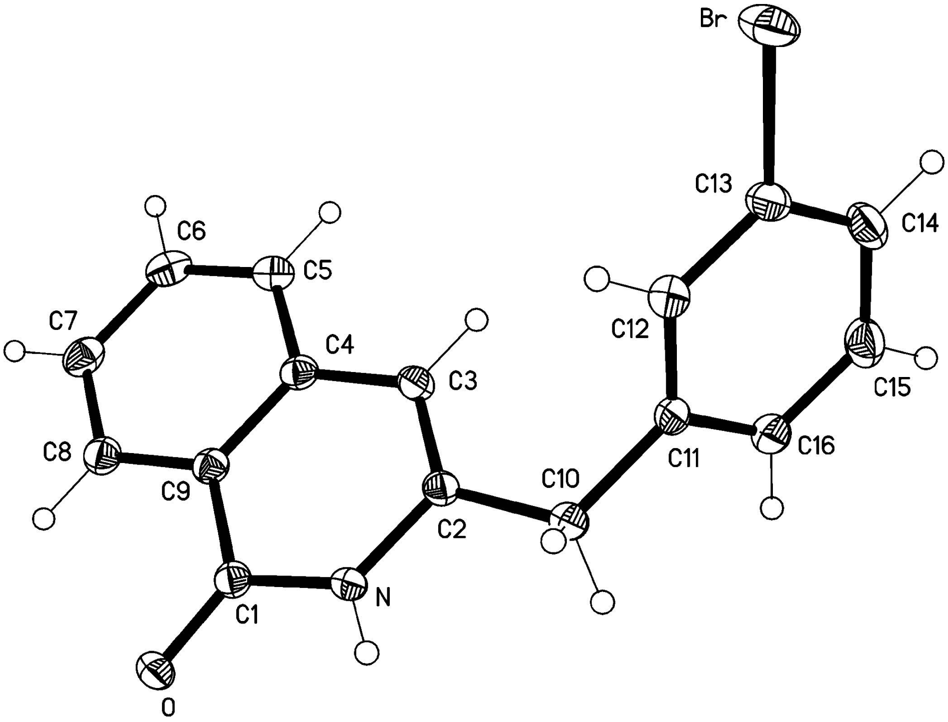

Figure 1

Molecular structure of the title compound showing the atom labelling scheme and displacement ellipsoids at the 50% probability level.

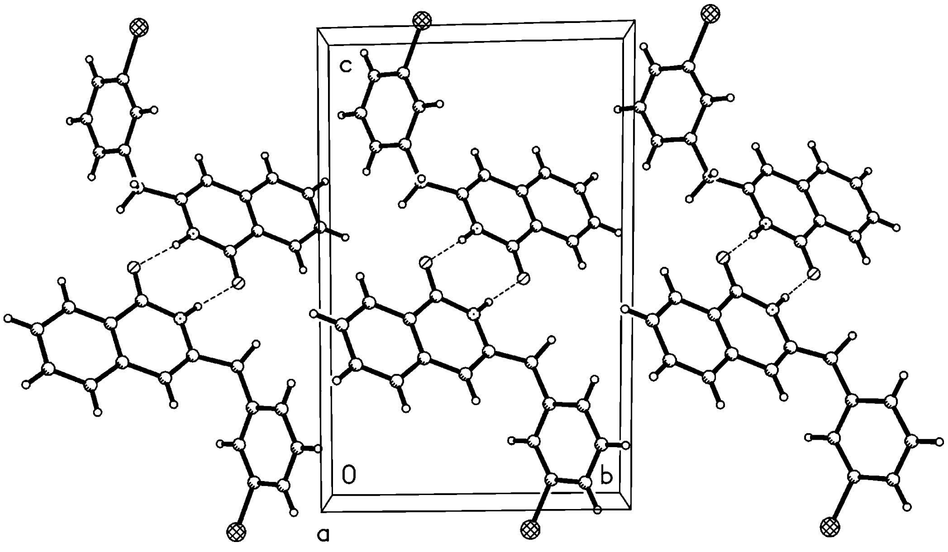

Figure 2

Packing diagram viewed parallel to the *x* axis. Hydrogen bonds are indicated by dashed lines.

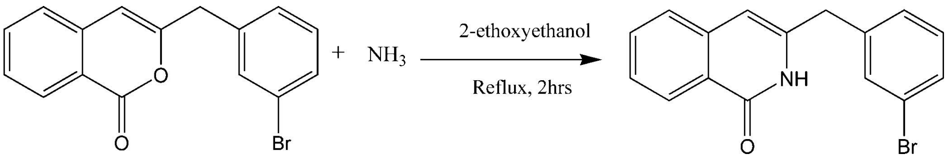

Figure 3

The formation of the title compound.

3-(3-Bromobenzyl)isoquinolin-1(2*H*)-one

Crystal data

$C_{16}H_{12}BrNO$
M_r = 314.18
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 4.5858 (4) Å
b = 9.4976 (7) Å
c = 14.8296 (11) Å
α = 88.698 (6)°
β = 83.829 (6)°
γ = 86.529 (6)°
V = 640.88 (9) Å^3
Z = 2
F(000) = 316
D_x = 1.628 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 9315 reflections
θ = 3.0–75.6°
μ = 4.28 mm^{-1}
T = 100 K
Prism, colourless
0.16 × 0.07 × 0.07 mm

Data collection

Oxford Diffraction Nova A diffractometer
Radiation source: Nova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3543 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.817, T_{max} = 1.000
9267 measured reflections
2642 independent reflections
2586 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 75.8°, θ_{min} = 3.0°
$h = -5 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.068
S = 0.90
2642 reflections
176 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.8338P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.016
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.65 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)
- 3.3521 (0.0017) *x* + 6.0480 (0.0040) *y* - 1.1834 (0.0042) *z* = 2.8164 (0.0038)
* 0.0154 (0.0011) C10 * -0.0101 (0.0014) C11 * -0.0109 (0.0014) C12 * -0.0098 (0.0015) C13 * 0.0036 (0.0015) C14 * 0.0074 (0.0013) C15 * -0.0053 (0.0014) C16 * 0.0097 (0.0008) Br
Rms deviation of fitted atoms = 0.0096
3.3793 (0.0010) *x* + 4.6226 (0.0023) *y* + 8.8098 (0.0043) *z* = 6.7949 (0.0012)
Angle to previous plane (with approximate e.s.d.) = 75.95 (0.03)
* 0.0161 (0.0013) C10 * -0.0154 (0.0013) N * 0.0007 (0.0011) O * -0.0018 (0.0014) C1 * -0.0080 (0.0015) C2 * -0.0054 (0.0015) C3 * 0.0009 (0.0015) C4 * -0.0003 (0.0014) C5 * -0.0016 (0.0014) C6 * -0.0046 (0.0015) C7 * 0.0104 (0.0015) C8 * 0.0089 (0.0015) C9
Rms deviation of fitted atoms = 0.0082

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
Br	0.41731 (5)	0.68583 (2)	−0.065128 (12)	0.03136 (9)
N	0.2730 (3)	0.49455 (15)	0.40535 (10)	0.0155 (3)
H01	0.131 (5)	0.545 (3)	0.4306 (17)	0.025 (6)*
O	0.2033 (3)	0.33201 (12)	0.51915 (8)	0.0187 (2)
C1	0.3423 (4)	0.36850 (17)	0.44644 (11)	0.0159 (3)
C2	0.4140 (4)	0.54534 (17)	0.32544 (11)	0.0155 (3)
C3	0.6381 (4)	0.46744 (18)	0.28064 (11)	0.0168 (3)
H3	0.7347	0.5018	0.2253	0.020*
C4	0.7296 (4)	0.33229 (17)	0.31716 (11)	0.0162 (3)
C5	0.9617 (4)	0.24635 (19)	0.27310 (12)	0.0194 (3)
H5	1.0630	0.2778	0.2178	0.023*
C6	1.0421 (4)	0.11699 (19)	0.30999 (12)	0.0216 (4)
H6	1.1988	0.0603	0.2798	0.026*
C7	0.8955 (4)	0.06803 (18)	0.39159 (12)	0.0216 (4)
H7	0.9509	−0.0217	0.4160	0.026*
C8	0.6701 (4)	0.15123 (18)	0.43607 (12)	0.0190 (3)
H8	0.5721	0.1192	0.4918	0.023*
C9	0.5853 (4)	0.28290 (17)	0.39932 (11)	0.0159 (3)
C10	0.2940 (4)	0.68958 (18)	0.29851 (12)	0.0194 (3)
H10A	0.3169	0.7560	0.3474	0.023*
H10B	0.0811	0.6853	0.2937	0.023*
C11	0.4378 (4)	0.74776 (17)	0.21028 (12)	0.0170 (3)
C12	0.3752 (4)	0.69677 (18)	0.12761 (12)	0.0201 (3)
H12	0.2438	0.6235	0.1261	0.024*
C13	0.5061 (4)	0.75386 (19)	0.04767 (12)	0.0211 (3)
C14	0.6969 (4)	0.8617 (2)	0.04693 (13)	0.0237 (4)

H14	0.7836	0.9000	−0.0086	0.028*
C15	0.7580 (4)	0.91238 (19)	0.12966 (14)	0.0247 (4)
H15	0.8878	0.9864	0.1309	0.030*
C16	0.6308 (4)	0.85559 (18)	0.21038 (12)	0.0195 (3)
H16	0.6758	0.8906	0.2665	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.04174 (14)	0.03719 (14)	0.01477 (12)	0.00054 (9)	−0.00234 (8)	−0.00284 (8)
N	0.0173 (7)	0.0153 (6)	0.0130 (6)	0.0004 (5)	0.0017 (5)	−0.0002 (5)
O	0.0218 (6)	0.0182 (6)	0.0152 (6)	−0.0004 (4)	0.0016 (5)	0.0024 (4)
C1	0.0177 (8)	0.0156 (7)	0.0148 (8)	−0.0021 (6)	−0.0031 (6)	−0.0007 (6)
C2	0.0184 (8)	0.0156 (7)	0.0130 (7)	−0.0029 (6)	−0.0020 (6)	−0.0008 (6)
C3	0.0186 (8)	0.0182 (8)	0.0134 (8)	−0.0027 (6)	−0.0002 (6)	0.0001 (6)
C4	0.0166 (7)	0.0173 (8)	0.0152 (8)	−0.0021 (6)	−0.0031 (6)	−0.0027 (6)
C5	0.0186 (8)	0.0226 (8)	0.0168 (8)	−0.0003 (6)	−0.0013 (6)	−0.0030 (6)
C6	0.0211 (8)	0.0228 (8)	0.0212 (9)	0.0041 (7)	−0.0050 (7)	−0.0066 (7)
C7	0.0270 (9)	0.0175 (8)	0.0211 (9)	0.0031 (7)	−0.0083 (7)	−0.0022 (6)
C8	0.0231 (8)	0.0175 (8)	0.0170 (8)	−0.0015 (6)	−0.0042 (6)	−0.0003 (6)
C9	0.0172 (8)	0.0158 (7)	0.0151 (8)	−0.0013 (6)	−0.0036 (6)	−0.0014 (6)
C10	0.0243 (8)	0.0172 (8)	0.0156 (8)	0.0019 (6)	0.0015 (7)	0.0007 (6)
C11	0.0193 (8)	0.0136 (7)	0.0166 (8)	0.0037 (6)	0.0013 (6)	0.0014 (6)
C12	0.0229 (8)	0.0175 (8)	0.0193 (8)	−0.0007 (6)	−0.0004 (7)	0.0001 (6)
C13	0.0250 (9)	0.0220 (8)	0.0151 (8)	0.0051 (7)	−0.0005 (7)	−0.0001 (6)
C14	0.0246 (9)	0.0243 (9)	0.0202 (9)	0.0016 (7)	0.0045 (7)	0.0073 (7)
C15	0.0250 (9)	0.0205 (8)	0.0280 (10)	−0.0043 (7)	0.0009 (7)	0.0044 (7)
C16	0.0213 (8)	0.0179 (8)	0.0189 (8)	0.0012 (6)	−0.0016 (6)	−0.0003 (6)

Geometric parameters (Å, º)

Br—C13	1.8997 (18)	C12—C13	1.384 (2)
N—C1	1.367 (2)	C13—C14	1.386 (3)
N—C2	1.379 (2)	C14—C15	1.390 (3)
O—C1	1.245 (2)	C15—C16	1.387 (3)
C1—C9	1.463 (2)	N—H01	0.85 (3)
C2—C3	1.352 (2)	C3—H3	0.9500
C2—C10	1.507 (2)	C5—H5	0.9500
C3—C4	1.438 (2)	C6—H6	0.9500
C4—C9	1.407 (2)	C7—H7	0.9500
C4—C5	1.413 (2)	C8—H8	0.9500
C5—C6	1.379 (3)	C10—H10A	0.9900
C6—C7	1.404 (3)	C10—H10B	0.9900
C7—C8	1.380 (3)	C12—H12	0.9500
C8—C9	1.403 (2)	C14—H14	0.9500
C10—C11	1.509 (2)	C15—H15	0.9500
C11—C16	1.394 (2)	C16—H16	0.9500
C11—C12	1.393 (2)		

C1—N—C2	125.36 (14)	C14—C15—C16	120.44 (17)
O—C1—N	120.74 (15)	C15—C16—C11	120.83 (17)
O—C1—C9	123.72 (15)	C1—N—H01	117.5 (16)
N—C1—C9	115.54 (15)	C2—N—H01	117.1 (16)
C3—C2—N	119.97 (15)	C2—C3—H3	120.2
C3—C2—C10	126.87 (15)	C4—C3—H3	120.2
N—C2—C10	113.15 (14)	C6—C5—H5	119.8
C2—C3—C4	119.66 (15)	C4—C5—H5	119.8
C9—C4—C5	118.38 (15)	C5—C6—H6	119.6
C9—C4—C3	119.57 (15)	C7—C6—H6	119.6
C5—C4—C3	122.06 (15)	C8—C7—H7	120.2
C6—C5—C4	120.36 (16)	C6—C7—H7	120.2
C5—C6—C7	120.90 (16)	C7—C8—H8	119.9
C8—C7—C6	119.53 (16)	C9—C8—H8	119.9
C7—C8—C9	120.25 (17)	C2—C10—H10A	108.5
C8—C9—C4	120.57 (16)	C11—C10—H10A	108.5
C8—C9—C1	119.53 (15)	C2—C10—H10B	108.5
C4—C9—C1	119.89 (15)	C11—C10—H10B	108.5
C2—C10—C11	114.88 (14)	H10A—C10—H10B	107.5
C16—C11—C12	119.00 (16)	C13—C12—H12	120.3
C16—C11—C10	120.36 (16)	C11—C12—H12	120.3
C12—C11—C10	120.63 (15)	C13—C14—H14	120.9
C13—C12—C11	119.41 (16)	C15—C14—H14	120.9
C12—C13—C14	122.10 (17)	C16—C15—H15	119.8
C12—C13—Br	119.42 (14)	C14—C15—H15	119.8
C14—C13—Br	118.46 (13)	C15—C16—H16	119.6
C13—C14—C15	118.22 (16)	C11—C16—H16	119.6

C2—N—C1—O	179.43 (15)	O—C1—C9—C8	0.6 (3)
C2—N—C1—C9	−0.6 (2)	N—C1—C9—C8	−179.41 (15)
C1—N—C2—C3	1.0 (3)	O—C1—C9—C4	179.80 (15)
C1—N—C2—C10	−178.22 (15)	N—C1—C9—C4	−0.2 (2)
N—C2—C3—C4	−0.5 (2)	C3—C2—C10—C11	2.2 (3)
C10—C2—C3—C4	178.55 (16)	N—C2—C10—C11	−178.69 (14)
C2—C3—C4—C9	−0.2 (2)	C2—C10—C11—C16	−106.28 (18)
C2—C3—C4—C5	179.80 (16)	C2—C10—C11—C12	75.1 (2)
C9—C4—C5—C6	0.4 (2)	C16—C11—C12—C13	0.1 (2)
C3—C4—C5—C6	−179.64 (16)	C10—C11—C12—C13	178.82 (15)
C4—C5—C6—C7	0.1 (3)	C11—C12—C13—C14	−0.6 (3)
C5—C6—C7—C8	−0.8 (3)	C11—C12—C13—Br	−179.29 (12)
C6—C7—C8—C9	0.9 (3)	C12—C13—C14—C15	0.4 (3)
C7—C8—C9—C4	−0.4 (3)	Br—C13—C14—C15	179.16 (13)
C7—C8—C9—C1	178.80 (15)	C13—C14—C15—C16	0.1 (3)
C5—C4—C9—C8	−0.2 (2)	C14—C15—C16—C11	−0.6 (3)
C3—C4—C9—C8	179.78 (15)	C12—C11—C16—C15	0.4 (3)
C5—C4—C9—C1	−179.46 (15)	C10—C11—C16—C15	−178.27 (16)
C3—C4—C9—C1	0.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H01···O^{i}	0.85 (3)	1.96 (3)	2.8036 (19)	176 (2)

Symmetry code: (i) $-x$, $-y+1$, $-z+1$.