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{2-[(3-Bromobenzylidene)amino]-5-chlorophenyl}(phenyl)methanone

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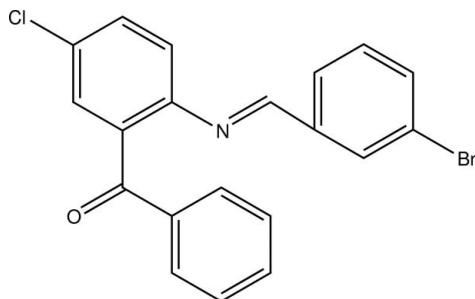
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 Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.045; wR factor = 0.115; data-to-parameter ratio = 14.9.

In the title compound, $\text{C}_{20}\text{H}_{13}\text{BrClNO}$, the azomethine double bond [$\text{C}=\text{N} = 1.246$ (4) Å] adopts an *E* conformation. The bromo- and chlorophenyl rings are inclined to one another by 13.70 (11)°, and form dihedral angles of 76.68 (10) and 74.24 (7)°, respectively, with the phenyl ring. In the crystal, molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds to form double stranded chains propagating along the *b*-axis direction.

Related literature

For background information and preparation of Schiff bases, see: Khan *et al.* (2009); Aslam *et al.* (2011a,b); Zeb & Yousuf (2011). For the crystal structures of related Schiff bases, see: Aslam *et al.* (2011a,b); Cox *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{20}\text{H}_{13}\text{BrClNO}$
 $M_r = 398.67$

 Orthorhombic, *Pbca*
 $a = 16.2068$ (12) Å

 $b = 7.8839$ (6) Å

 $c = 27.262$ (2) Å

 $V = 3483.4$ (5) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 2.52$ mm⁻¹
 $T = 273$ K

 $0.52 \times 0.21 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

 Absorption correction: multi-scan (*SADABS*; Bruker, 2000)

 $T_{\min} = 0.354$, $T_{\max} = 0.704$

19261 measured reflections

3243 independent reflections

 1931 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.115$
 $S = 1.01$

3243 reflections

217 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.64$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> — <i>H</i> ⋯ <i>A</i>
$\text{C12}—\text{H12A}\cdots\text{O1}^{\text{i}}$	0.93	2.44	3.346 (4)	166
$\text{C17}—\text{H17A}\cdots\text{O1}^{\text{ii}}$	0.93	2.51	3.428 (5)	168

 Symmetry codes: (i) $-x, -y - 1, -z + 1$; (ii) $x, y + 1, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2506).

References

- Aslam, M., Anis, I., Afza, N., Nelofar, A. & Yousuf, S. (2011a). *Acta Cryst.* **E67**, o3442–o3443.
 Aslam, M., Anis, I., Afza, N., Nelofar, A. & Yousuf, S. (2011b). *Acta Cryst.* **E67**, o3215.
 Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Cox, P. J., Kechagias, D. & Kelly, O. (2008). *Acta Cryst.* **B64**, 206–216.
 Khan, K. M., Khan, M., Ali, M., Taha, M., Rasheed, S., Perveen, S. & Choudhary, M. I. (2009). *Bioorg. Med. Chem.* **17**, 7795–7801.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Zeb, A. & Yousuf, S. (2011). *Acta Cryst.* **E67**, o2801.

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supporting information

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{2-[(3-Bromobenzylidene)amino]-5-chlorophenyl}(phenyl)methanone

M. Aslam, I. Anis, N. Afza, M. Safder and S. Yousuf

S1. Comment

The title compound was prepared as a part of our ongoing research on schiff bases (Khan *et al.*, 2009; Aslam *et al.*, 2011a,b; Zeb & Yousuf, 2011).

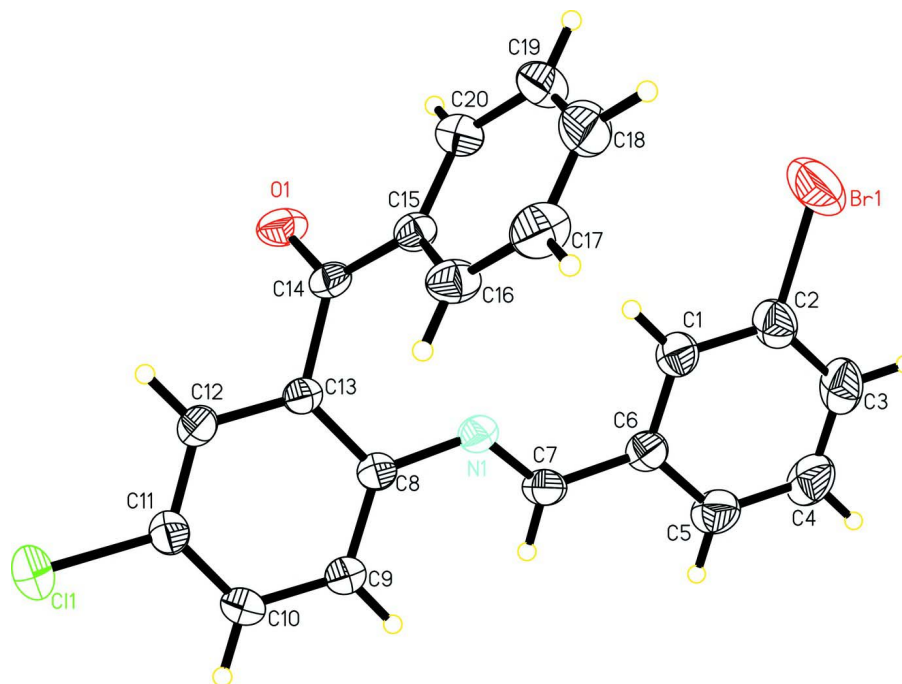
In the title compound (Fig. 1), the azomethine double bond (C=N, 1.246 (4) Å) adopts an E configuration with torsion angle C6—C7—N1—C8 174.9 (3)°. The bond lengths and angle are similar as in other structurally related compounds (Aslam *et al.*, 2011a,b; Cox *et al.*, 2008). In the crystal structure the molecules are arranged in parallel sheets along the *b*-axis via C—H···O type intermolecular hydrogen bonds (Fig. 2).

S2. Experimental

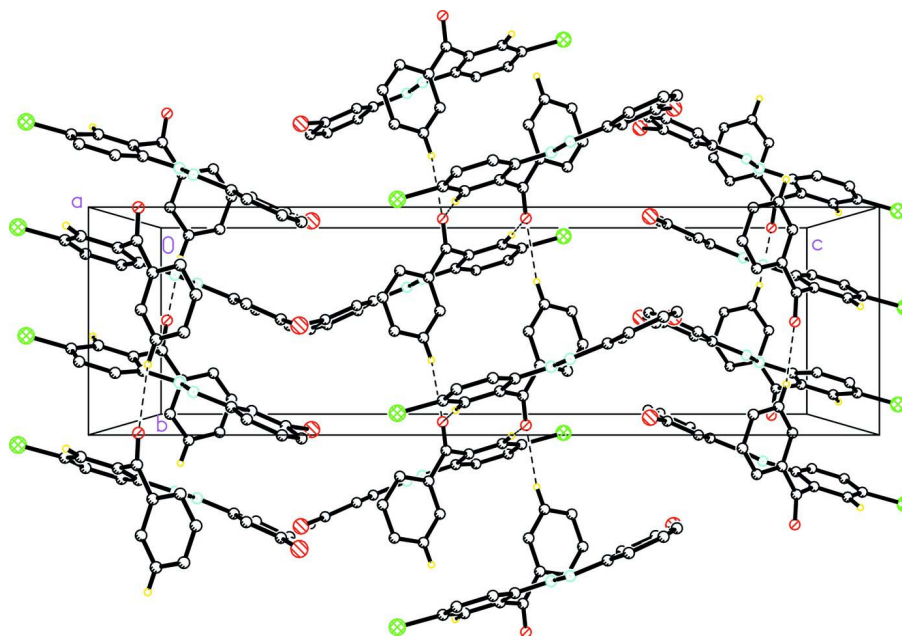
A mixture of 3-bromobenzaldehyde (1 mol) and 2-amino-5-chlorobenzophenone (1 mol) in ethanol (50 ml) along with 3 drops of conc. H₂SO₄ was refluxed for 5 h at 343 K. After cooling, the mixture was concentrated to one third under reduced pressure. The concentrated reaction mixture was kept at room temperature and orange red crystals were obtained after five days. The crystalline product was collected, washed with methanol and dried to afford the title compound in 87% yield. Slow evaporation of a methanol solution afforded yellow crystals suitable for single-crystal X-ray diffraction studies. All chemicals were purchased from Sigma-Aldrich.

S3. Refinement

H atoms were positioned geometrically with C—H = 0.93 Å, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radius.

**Figure 2**

A view of the C—H...O hydrogen bonds (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity.

{2-[(3-Bromobenzylidene)-amino]-5-chlorophenyl}(phenyl)methanone*Crystal data*C₂₀H₁₃BrClNO $M_r = 398.67$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 16.2068$ (12) Å $b = 7.8839$ (6) Å $c = 27.262$ (2) Å $V = 3483.4$ (5) Å³ $Z = 8$ $F(000) = 1600$ $D_x = 1.520$ Mg m⁻³Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 2145 reflections

 $\theta = 2.5$ – 20.5° $\mu = 2.52$ mm⁻¹ $T = 273$ K

Block, yellow

 $0.52 \times 0.21 \times 0.15$ mm*Data collection*Bruker SMART APEX CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

 ω scan

Absorption correction: multi-scan

(SADABS; Bruker, 2000)

 $T_{\min} = 0.354$, $T_{\max} = 0.704$

19261 measured reflections

3243 independent reflections

1931 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.056$ $\theta_{\max} = 25.5^\circ$, $\theta_{\min} = 1.5^\circ$ $h = -19 \rightarrow 19$ $k = -9 \rightarrow 9$ $l = -33 \rightarrow 33$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.115$ $S = 1.01$

3243 reflections

217 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 3.0254P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.40$ e Å⁻³ $\Delta\rho_{\min} = -0.64$ 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.

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.08907 (4)	0.01989 (9)	0.238025 (19)	0.1197 (3)
Cl1	0.20842 (7)	-0.43108 (16)	0.61666 (4)	0.0784 (4)
O1	0.04701 (14)	-0.5034 (3)	0.44146 (10)	0.0608 (7)
N1	0.21723 (16)	-0.2133 (4)	0.40923 (10)	0.0480 (7)
C1	0.1977 (2)	-0.0784 (5)	0.31387 (12)	0.0564 (10)

H1B	0.1496	-0.1092	0.3302	0.068*
C2	0.1939 (3)	-0.0077 (5)	0.26789 (13)	0.0657 (11)
C3	0.2627 (3)	0.0425 (5)	0.24302 (14)	0.0756 (13)
H3A	0.2585	0.0912	0.2121	0.091*
C4	0.3383 (3)	0.0196 (6)	0.26466 (15)	0.0806 (14)
H4A	0.3859	0.0535	0.2484	0.097*
C5	0.3439 (2)	-0.0533 (6)	0.31041 (14)	0.0720 (12)
H5A	0.3955	-0.0691	0.3246	0.086*
C6	0.2739 (2)	-0.1034 (5)	0.33568 (12)	0.0508 (9)
C7	0.2795 (2)	-0.1732 (5)	0.38504 (12)	0.0517 (9)
H7A	0.3314	-0.1883	0.3990	0.062*
C8	0.22119 (19)	-0.2687 (4)	0.45845 (11)	0.0421 (8)
C9	0.2910 (2)	-0.2620 (5)	0.48817 (12)	0.0501 (9)
H9A	0.3406	-0.2225	0.4753	0.060*
C10	0.2873 (2)	-0.3135 (5)	0.53632 (12)	0.0534 (9)
H10A	0.3342	-0.3089	0.5559	0.064*
C11	0.2143 (2)	-0.3715 (5)	0.55527 (12)	0.0508 (9)
C12	0.1447 (2)	-0.3843 (4)	0.52657 (12)	0.0485 (9)
H12A	0.0960	-0.4277	0.5396	0.058*
C13	0.14765 (18)	-0.3321 (4)	0.47808 (11)	0.0400 (8)
C14	0.07325 (18)	-0.3588 (5)	0.44606 (11)	0.0431 (8)
C15	0.03203 (19)	-0.2139 (4)	0.42183 (11)	0.0427 (8)
C16	0.0429 (2)	-0.0504 (5)	0.43778 (14)	0.0581 (10)
H16A	0.0776	-0.0286	0.4642	0.070*
C18	-0.0479 (3)	0.0481 (6)	0.37522 (18)	0.0799 (13)
H18A	-0.0746	0.1369	0.3593	0.096*
C19	-0.0588 (2)	-0.1138 (6)	0.35928 (15)	0.0706 (12)
H19A	-0.0930	-0.1352	0.3326	0.085*
C17	0.0027 (2)	0.0814 (6)	0.41493 (17)	0.0750 (12)
H17A	0.0096	0.1920	0.4261	0.090*
C20	-0.0199 (2)	-0.2447 (5)	0.38228 (13)	0.0553 (9)
H20A	-0.0281	-0.3553	0.3714	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1021 (4)	0.1846 (7)	0.0725 (4)	0.0117 (4)	-0.0213 (3)	0.0344 (4)
Cl1	0.0843 (7)	0.0996 (9)	0.0513 (5)	-0.0078 (6)	-0.0055 (5)	0.0208 (6)
O1	0.0485 (14)	0.0481 (16)	0.0857 (19)	-0.0091 (13)	-0.0129 (13)	-0.0014 (13)
N1	0.0384 (15)	0.059 (2)	0.0470 (16)	-0.0057 (14)	0.0019 (13)	0.0018 (14)
C1	0.060 (2)	0.066 (3)	0.043 (2)	-0.005 (2)	0.0067 (17)	-0.0031 (18)
C2	0.081 (3)	0.072 (3)	0.044 (2)	-0.002 (2)	-0.0013 (19)	-0.003 (2)
C3	0.109 (4)	0.077 (3)	0.041 (2)	-0.012 (3)	0.012 (2)	-0.001 (2)
C4	0.087 (4)	0.100 (4)	0.055 (2)	-0.028 (3)	0.024 (2)	-0.005 (2)
C5	0.060 (2)	0.099 (3)	0.056 (2)	-0.016 (2)	0.0112 (19)	-0.006 (2)
C6	0.054 (2)	0.056 (2)	0.0417 (18)	-0.0070 (18)	0.0043 (16)	-0.0089 (17)
C7	0.042 (2)	0.065 (3)	0.048 (2)	-0.0048 (18)	-0.0032 (17)	-0.0059 (18)
C8	0.0406 (18)	0.041 (2)	0.0442 (18)	-0.0010 (16)	-0.0002 (15)	-0.0022 (15)

C9	0.0437 (19)	0.056 (2)	0.051 (2)	-0.0079 (18)	-0.0024 (17)	0.0021 (17)
C10	0.046 (2)	0.063 (3)	0.051 (2)	-0.0029 (18)	-0.0121 (17)	0.0017 (18)
C11	0.055 (2)	0.051 (2)	0.0462 (19)	-0.0017 (19)	-0.0016 (17)	0.0047 (17)
C12	0.0436 (19)	0.049 (2)	0.053 (2)	-0.0058 (17)	0.0027 (16)	0.0057 (17)
C13	0.0356 (18)	0.0359 (19)	0.0483 (19)	-0.0002 (15)	-0.0017 (14)	0.0002 (15)
C14	0.0329 (17)	0.048 (2)	0.0480 (19)	-0.0035 (17)	0.0048 (14)	-0.0056 (17)
C15	0.0356 (17)	0.042 (2)	0.0503 (19)	0.0025 (16)	0.0022 (15)	-0.0020 (16)
C16	0.046 (2)	0.052 (3)	0.077 (3)	0.0043 (19)	-0.0056 (19)	-0.007 (2)
C18	0.066 (3)	0.076 (3)	0.098 (3)	0.016 (3)	-0.003 (3)	0.026 (3)
C19	0.059 (3)	0.084 (3)	0.069 (3)	0.007 (2)	-0.015 (2)	0.011 (2)
C17	0.065 (3)	0.052 (3)	0.109 (3)	0.006 (2)	0.002 (3)	-0.001 (2)
C20	0.050 (2)	0.061 (2)	0.055 (2)	0.0024 (19)	-0.0054 (18)	-0.0049 (19)

Geometric parameters (Å, °)

Br1—C2	1.896 (4)	C9—H9A	0.9300
C11—C11	1.741 (3)	C10—C11	1.369 (5)
O1—C14	1.223 (4)	C10—H10A	0.9300
N1—C7	1.246 (4)	C11—C12	1.377 (4)
N1—C8	1.412 (4)	C12—C13	1.385 (4)
C1—C2	1.373 (5)	C12—H12A	0.9300
C1—C6	1.384 (5)	C13—C14	1.503 (4)
C1—H1B	0.9300	C14—C15	1.479 (5)
C2—C3	1.365 (6)	C15—C16	1.372 (5)
C3—C4	1.371 (6)	C15—C20	1.389 (4)
C3—H3A	0.9300	C16—C17	1.376 (5)
C4—C5	1.376 (6)	C16—H16A	0.9300
C4—H4A	0.9300	C18—C19	1.360 (6)
C5—C6	1.385 (5)	C18—C17	1.383 (6)
C5—H5A	0.9300	C18—H18A	0.9300
C6—C7	1.457 (5)	C19—C20	1.363 (5)
C7—H7A	0.9300	C19—H19A	0.9300
C8—C9	1.393 (4)	C17—H17A	0.9300
C8—C13	1.399 (4)	C20—H20A	0.9300
C9—C10	1.375 (4)		
C7—N1—C8	123.0 (3)	C10—C11—C12	121.2 (3)
C2—C1—C6	119.4 (4)	C10—C11—C11	120.0 (3)
C2—C1—H1B	120.3	C12—C11—C11	118.8 (3)
C6—C1—H1B	120.3	C11—C12—C13	119.4 (3)
C3—C2—C1	122.3 (4)	C11—C12—H12A	120.3
C3—C2—Br1	119.1 (3)	C13—C12—H12A	120.3
C1—C2—Br1	118.6 (3)	C12—C13—C8	120.1 (3)
C2—C3—C4	118.6 (4)	C12—C13—C14	119.0 (3)
C2—C3—H3A	120.7	C8—C13—C14	120.7 (3)
C4—C3—H3A	120.7	O1—C14—C15	121.1 (3)
C3—C4—C5	120.2 (4)	O1—C14—C13	118.0 (3)
C3—C4—H4A	119.9	C15—C14—C13	120.9 (3)

C5—C4—H4A	119.9	C16—C15—C20	119.3 (3)
C4—C5—C6	121.1 (4)	C16—C15—C14	121.7 (3)
C4—C5—H5A	119.5	C20—C15—C14	119.0 (3)
C6—C5—H5A	119.5	C15—C16—C17	120.4 (4)
C1—C6—C5	118.4 (3)	C15—C16—H16A	119.8
C1—C6—C7	120.4 (3)	C17—C16—H16A	119.8
C5—C6—C7	121.1 (3)	C19—C18—C17	120.4 (4)
N1—C7—C6	122.3 (3)	C19—C18—H18A	119.8
N1—C7—H7A	118.9	C17—C18—H18A	119.8
C6—C7—H7A	118.9	C18—C19—C20	120.2 (4)
C9—C8—C13	118.9 (3)	C18—C19—H19A	119.9
C9—C8—N1	125.3 (3)	C20—C19—H19A	119.9
C13—C8—N1	115.8 (3)	C16—C17—C18	119.4 (4)
C10—C9—C8	120.6 (3)	C16—C17—H17A	120.3
C10—C9—H9A	119.7	C18—C17—H17A	120.3
C8—C9—H9A	119.7	C19—C20—C15	120.3 (4)
C11—C10—C9	119.8 (3)	C19—C20—H20A	119.8
C11—C10—H10A	120.1	C15—C20—H20A	119.8
C9—C10—H10A	120.1		
C6—C1—C2—C3	-1.4 (6)	C11—C12—C13—C8	0.7 (5)
C6—C1—C2—Br1	178.4 (3)	C11—C12—C13—C14	175.4 (3)
C1—C2—C3—C4	0.7 (6)	C9—C8—C13—C12	1.1 (5)
Br1—C2—C3—C4	-179.0 (3)	N1—C8—C13—C12	-177.9 (3)
C2—C3—C4—C5	0.3 (7)	C9—C8—C13—C14	-173.5 (3)
C3—C4—C5—C6	-0.6 (7)	N1—C8—C13—C14	7.5 (4)
C2—C1—C6—C5	1.0 (6)	C12—C13—C14—O1	-57.2 (4)
C2—C1—C6—C7	178.1 (3)	C8—C13—C14—O1	117.5 (4)
C4—C5—C6—C1	0.0 (6)	C12—C13—C14—C15	121.4 (3)
C4—C5—C6—C7	-177.1 (4)	C8—C13—C14—C15	-63.9 (4)
C8—N1—C7—C6	-174.9 (3)	O1—C14—C15—C16	159.3 (3)
C1—C6—C7—N1	0.3 (6)	C13—C14—C15—C16	-19.3 (5)
C5—C6—C7—N1	177.3 (4)	O1—C14—C15—C20	-19.9 (5)
C7—N1—C8—C9	10.0 (5)	C13—C14—C15—C20	161.6 (3)
C7—N1—C8—C13	-171.1 (3)	C20—C15—C16—C17	0.1 (5)
C13—C8—C9—C10	-1.4 (5)	C14—C15—C16—C17	-179.0 (3)
N1—C8—C9—C10	177.5 (3)	C17—C18—C19—C20	-0.1 (6)
C8—C9—C10—C11	-0.1 (5)	C15—C16—C17—C18	-0.9 (6)
C9—C10—C11—C12	2.0 (6)	C19—C18—C17—C16	0.9 (6)
C9—C10—C11—C11	-178.2 (3)	C18—C19—C20—C15	-0.8 (6)
C10—C11—C12—C13	-2.3 (5)	C16—C15—C20—C19	0.7 (5)
C11—C11—C12—C13	177.9 (3)	C14—C15—C20—C19	179.9 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C12—H12A \cdots O1 ⁱ	0.93	2.44	3.346 (4)	166

C17—H17A ⁱⁱ ···O1 ⁱⁱ	0.93	2.51	3.428 (5)	168
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Symmetry codes: (i) $-x, -y-1, -z+1$; (ii) $x, y+1, z$.