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2-[(4-Bromophenyl)(4-fluorophenylamino)methyl]cyclohexanone

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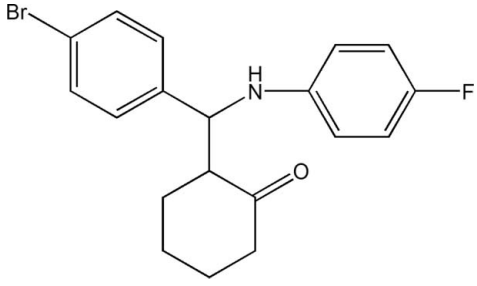
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; R factor = 0.048; wR factor = 0.113; data-to-parameter ratio = 14.7.

In the crystal structure of the title compound, $C_{19}H_{19}BrFNO$, molecules are connected into dimers *via* intermolecular N—H···O hydrogen bonding. The dihedral angle between the two benzene rings is 74.84 (1)°. The cyclohexane ring has the usual chair conformation.

Related literature

For related literature, see: Shou *et al.* (2006).



Experimental

Crystal data

$C_{19}H_{19}BrFNO$
M_r = 376.26
Monoclinic, $P2_1/n$
a = 12.6501 (14) Å
b = 8.2310 (9) Å
c = 17.0111 (18) Å
β = 103.031 (2)°
V = 1725.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.40 mm^{-1}
T = 295 (2) K
0.37 × 0.31 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.450, T_{max} = 0.660
8450 measured reflections
3060 independent reflections
1669 reflections with $I > 2\sigma(I)$
R_{int} = 0.120

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.113
S = 0.82
3060 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.81	2.19	2.978 (4)	166

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2047).

References

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  doi:10.1107/S1600536807036847

supplementary materials

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2-[(4-Bromophenyl)(4-fluorophenylamino)methyl]cyclohexanone

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Comment

β-Amino carbonyl moieties are found as structural units of a number of biologically active natural products. As a part of a project on the synthesis of such compounds the crystal structure analysis was performed. In the crystal structure of the title compound (I) the dihedral angle between the phenyl rings C8—C13 and C14—C19 is 74.84 (1)°. The six-membered ring (C1—C6) is in a chair conformation. A pair of weak intermolecular N—H···O hydrogen bonds link the molecules into dimers (Fig.1 and Table 1).

Experimental

The starting materials were purchased from Acros and used without purification. The title compound (I) was synthesized according to the method described previously (Shou *et al.*, 2006). IR (KBr,cm^{-1}): 1705, 1598, 1515, 1489, 1256. ^{1}H NMR (500 MHz, δ in p.p.m., $CDCl_3$): 7.43–7.40 (m, 2H), 7.25–7.22 (m, 2H), 6.78–6.74 (m, 2H), 6.44–6.41 (m, 2H), 4.75 (s, br, 1H), 4.49 (d, J = 6.6 Hz, 1H), 2.72–2.70 (m, 1H), 2.42–2.39 (m, 1H), 2.34–2.31 (m, 1H), 1.98–1.89 (m, 2H), 1.88–1.85 (m, 1H), 1.75–1.63 (m, 3H). ^{13}C NMR (125 MHz, δ in p.p.m., $CDCl_3$): 212.7, 157.1 (d, $J_{C—F}$ = 234.4 Hz), 143.5, 140.8, 131.8, 129.3, 121.2, 115.7 (d, $J_{C—F}$ = 22.5 Hz), 114.9 (d, $J_{C—F}$ = 7.5 Hz), 58.7, 57.4, 42.4, 31.8, 28.1, 24.3. Melting point: 386–387 K. MS (ESI): m/*z* 399 ([*M*+Na]$^+$). Calculated for $C_{19}H_{19}BrFNO$: C 60.65, H 5.09, N 3.72%; found: C 60.70, H 5.12, N 3.70%. The crystal used for the data collection was obtained by slow evaporation of the solvent from a saturated hexane-dichloromethane solution of I at room temperature.

Refinement

The N—H H atom was located in the difference map, fixed at this position and refined as riding with U_{iso}(H) = 1.2U_{eq}(N). The C—H H atoms were placed in calculated positions, with C—H = 0.93Å (aromatic), 0.97Å (methylene) and 0.98Å (methine) and were refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Figures

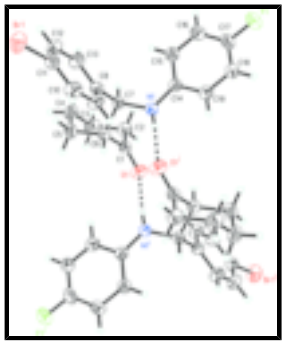


Fig. 1. View of the dimers in the crystal structure of compound I with labeling and displacement ellipsoids drawn at the 50% probability level (Hydrogen bonding is shown as dashed lines. Symmetry code: i) $1-x$, $1-y$, $1-z$.

2-[(4-Bromophenyl)(4-fluorophenylamino)methyl]cyclohexanone

Crystal data

$C_{19}H_{19}BrFNO$	F_{000} = 768
M_r = 376.26	D_x = 1.448 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1759 reflections
a = 12.6501 (14) Å	θ = 4.5–41.2º
b = 8.2310 (9) Å	μ = 2.40 mm^{-1}
c = 17.0111 (18) Å	T = 295 (2) K
β = 103.031 (2)º	Prismatic, colorless
V = 1725.6 (3) Å^3	0.37 × 0.31 × 0.17 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3060 independent reflections
Radiation source: fine-focus sealed tube	1669 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.120
T = 295(2) K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −8→15
T_{min} = 0.450, T_{max} = 0.660	k = −9→9
8450 measured reflections	l = −20→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H-atom parameters constrained

$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.82$	$(\Delta/\sigma)_{max} < 0.001$
3060 reflections	$\Delta\rho_{max} = 0.51$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.04416 (4)	−0.08221 (6)	0.22974 (3)	0.0824 (2)
F1	0.4047 (2)	−0.0544 (3)	0.82439 (15)	0.1046 (10)
N1	0.3694 (2)	0.3266 (4)	0.55354 (17)	0.0530 (8)
H1	0.4227	0.3337	0.5347	0.064*
O1	0.4507 (2)	0.7010 (3)	0.53240 (16)	0.0698 (8)
C1	0.3566 (3)	0.6596 (5)	0.5257 (2)	0.0497 (9)
C2	0.2870 (3)	0.7350 (5)	0.5766 (2)	0.0626 (11)
H2A	0.3290	0.8137	0.6132	0.075*
H2B	0.2620	0.6518	0.6085	0.075*
C3	0.1896 (3)	0.8187 (6)	0.5221 (3)	0.0732 (12)
H3A	0.1414	0.8591	0.5547	0.088*
H3B	0.2145	0.9108	0.4956	0.088*
C4	0.1277 (3)	0.7028 (5)	0.4588 (3)	0.0693 (12)
H4A	0.0967	0.6162	0.4851	0.083*
H4B	0.0687	0.7605	0.4236	0.083*
C5	0.2005 (3)	0.6311 (5)	0.4095 (2)	0.0630 (11)
H5A	0.2255	0.7171	0.3791	0.076*
H5B	0.1592	0.5547	0.3711	0.076*
C6	0.3002 (3)	0.5429 (4)	0.4614 (2)	0.0480 (9)
H6	0.3501	0.5158	0.4269	0.058*
C7	0.2709 (3)	0.3845 (4)	0.5007 (2)	0.0470 (9)
H7	0.2194	0.4120	0.5339	0.056*
C8	0.2161 (3)	0.2645 (4)	0.4365 (2)	0.0477 (9)
C9	0.2742 (3)	0.1833 (5)	0.3891 (2)	0.0598 (11)
H9	0.3489	0.1980	0.3986	0.072*

C10	0.2239 (3)	0.0810 (5)	0.3282 (2)	0.0630 (11)
H10	0.2642	0.0292	0.2961	0.076*
C11	0.1146 (3)	0.0558 (5)	0.3149 (2)	0.0566 (10)
C12	0.0553 (3)	0.1319 (5)	0.3614 (2)	0.0636 (11)
H12	−0.0192	0.1156	0.3519	0.076*
C13	0.1063 (3)	0.2331 (5)	0.4227 (2)	0.0598 (11)
H13	0.0658	0.2816	0.4557	0.072*
C14	0.3719 (3)	0.2214 (4)	0.6170 (2)	0.0466 (9)
C15	0.2835 (3)	0.1887 (5)	0.6498 (2)	0.0568 (10)
H15	0.2157	0.2295	0.6247	0.068*
C16	0.2939 (3)	0.0961 (5)	0.7196 (2)	0.0657 (11)
H16	0.2345	0.0783	0.7422	0.079*
C17	0.3928 (4)	0.0321 (5)	0.7540 (2)	0.0668 (12)
C18	0.4799 (3)	0.0540 (5)	0.7218 (3)	0.0689 (12)
H18	0.5463	0.0074	0.7458	0.083*
C19	0.4693 (3)	0.1465 (5)	0.6528 (2)	0.0621 (11)
H19	0.5288	0.1591	0.6297	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0878 (4)	0.0778 (4)	0.0732 (4)	−0.0049 (3)	0.0003 (3)	−0.0205 (2)
F1	0.1045 (19)	0.124 (2)	0.0783 (18)	−0.0166 (17)	0.0050 (15)	0.0468 (17)
N1	0.0448 (17)	0.063 (2)	0.0512 (18)	0.0024 (16)	0.0108 (15)	0.0142 (17)
O1	0.0537 (17)	0.077 (2)	0.0808 (19)	−0.0162 (15)	0.0184 (15)	−0.0109 (16)
C1	0.053 (2)	0.053 (2)	0.042 (2)	0.000 (2)	0.0104 (19)	0.0009 (18)
C2	0.070 (3)	0.062 (3)	0.058 (2)	−0.009 (2)	0.020 (2)	−0.015 (2)
C3	0.061 (3)	0.062 (3)	0.102 (3)	0.005 (2)	0.028 (3)	−0.008 (3)
C4	0.054 (2)	0.062 (3)	0.085 (3)	0.006 (2)	0.003 (2)	0.006 (2)
C5	0.073 (3)	0.056 (3)	0.051 (2)	−0.007 (2)	−0.003 (2)	0.002 (2)
C6	0.052 (2)	0.046 (2)	0.048 (2)	−0.0002 (18)	0.0161 (18)	0.0005 (18)
C7	0.045 (2)	0.050 (2)	0.046 (2)	0.0038 (18)	0.0112 (18)	0.0047 (18)
C8	0.043 (2)	0.042 (2)	0.058 (2)	0.0014 (18)	0.0113 (19)	0.0044 (18)
C9	0.044 (2)	0.068 (3)	0.071 (3)	−0.006 (2)	0.020 (2)	−0.010 (2)
C10	0.062 (3)	0.062 (3)	0.070 (3)	−0.001 (2)	0.025 (2)	−0.011 (2)
C11	0.059 (3)	0.054 (3)	0.054 (2)	0.000 (2)	0.008 (2)	0.0022 (19)
C12	0.040 (2)	0.069 (3)	0.079 (3)	0.002 (2)	0.008 (2)	−0.008 (2)
C13	0.047 (2)	0.064 (3)	0.071 (3)	0.004 (2)	0.018 (2)	−0.011 (2)
C14	0.046 (2)	0.045 (2)	0.049 (2)	−0.0001 (18)	0.0097 (19)	−0.0003 (18)
C15	0.053 (2)	0.066 (3)	0.052 (2)	0.006 (2)	0.013 (2)	0.002 (2)
C16	0.069 (3)	0.077 (3)	0.055 (2)	−0.005 (2)	0.023 (2)	0.005 (2)
C17	0.086 (3)	0.062 (3)	0.046 (2)	−0.010 (3)	0.001 (2)	0.011 (2)
C18	0.056 (3)	0.074 (3)	0.072 (3)	0.001 (2)	0.006 (2)	0.024 (2)
C19	0.050 (2)	0.072 (3)	0.065 (3)	0.002 (2)	0.016 (2)	0.012 (2)

Geometric parameters (Å, °)

Br1—C11	1.898 (4)	C7—C8	1.519 (5)
F1—C17	1.371 (4)	C7—H7	0.9800

N1—C14	1.378 (4)	C8—C13	1.379 (5)
N1—C7	1.444 (4)	C8—C9	1.380 (5)
N1—H1	0.8111	C9—C10	1.375 (5)
O1—C1	1.218 (4)	C9—H9	0.9300
C1—C2	1.501 (5)	C10—C11	1.366 (5)
C1—C6	1.509 (5)	C10—H10	0.9300
C2—C3	1.529 (5)	C11—C12	1.359 (5)
C2—H2A	0.9700	C12—C13	1.377 (5)
C2—H2B	0.9700	C12—H12	0.9300
C3—C4	1.517 (5)	C13—H13	0.9300
C3—H3A	0.9700	C14—C15	1.385 (5)
C3—H3B	0.9700	C14—C19	1.389 (5)
C4—C5	1.499 (5)	C15—C16	1.391 (5)
C4—H4A	0.9700	C15—H15	0.9300
C4—H4B	0.9700	C16—C17	1.363 (6)
C5—C6	1.548 (5)	C16—H16	0.9300
C5—H5A	0.9700	C17—C18	1.349 (6)
C5—H5B	0.9700	C18—C19	1.380 (5)
C6—C7	1.547 (5)	C18—H18	0.9300
C6—H6	0.9800	C19—H19	0.9300
C14—N1—C7	124.0 (3)	N1—C7—H7	108.0
C14—N1—H1	118.7	C8—C7—H7	108.0
C7—N1—H1	114.1	C6—C7—H7	108.0
O1—C1—C2	121.2 (3)	C13—C8—C9	117.3 (3)
O1—C1—C6	122.5 (3)	C13—C8—C7	121.5 (3)
C2—C1—C6	116.0 (3)	C9—C8—C7	121.2 (3)
C1—C2—C3	109.5 (3)	C10—C9—C8	121.3 (3)
C1—C2—H2A	109.8	C10—C9—H9	119.3
C3—C2—H2A	109.8	C8—C9—H9	119.3
C1—C2—H2B	109.8	C11—C10—C9	119.8 (4)
C3—C2—H2B	109.8	C11—C10—H10	120.1
H2A—C2—H2B	108.2	C9—C10—H10	120.1
C4—C3—C2	111.3 (3)	C12—C11—C10	120.3 (4)
C4—C3—H3A	109.4	C12—C11—Br1	119.6 (3)
C2—C3—H3A	109.4	C10—C11—Br1	120.0 (3)
C4—C3—H3B	109.4	C11—C12—C13	119.6 (4)
C2—C3—H3B	109.4	C11—C12—H12	120.2
H3A—C3—H3B	108.0	C13—C12—H12	120.2
C5—C4—C3	111.2 (3)	C12—C13—C8	121.6 (4)
C5—C4—H4A	109.4	C12—C13—H13	119.2
C3—C4—H4A	109.4	C8—C13—H13	119.2
C5—C4—H4B	109.4	N1—C14—C15	124.0 (3)
C3—C4—H4B	109.4	N1—C14—C19	118.9 (3)
H4A—C4—H4B	108.0	C15—C14—C19	117.0 (3)
C4—C5—C6	112.9 (3)	C14—C15—C16	121.4 (4)
C4—C5—H5A	109.0	C14—C15—H15	119.3
C6—C5—H5A	109.0	C16—C15—H15	119.3
C4—C5—H5B	109.0	C17—C16—C15	118.7 (4)
C6—C5—H5B	109.0	C17—C16—H16	120.7

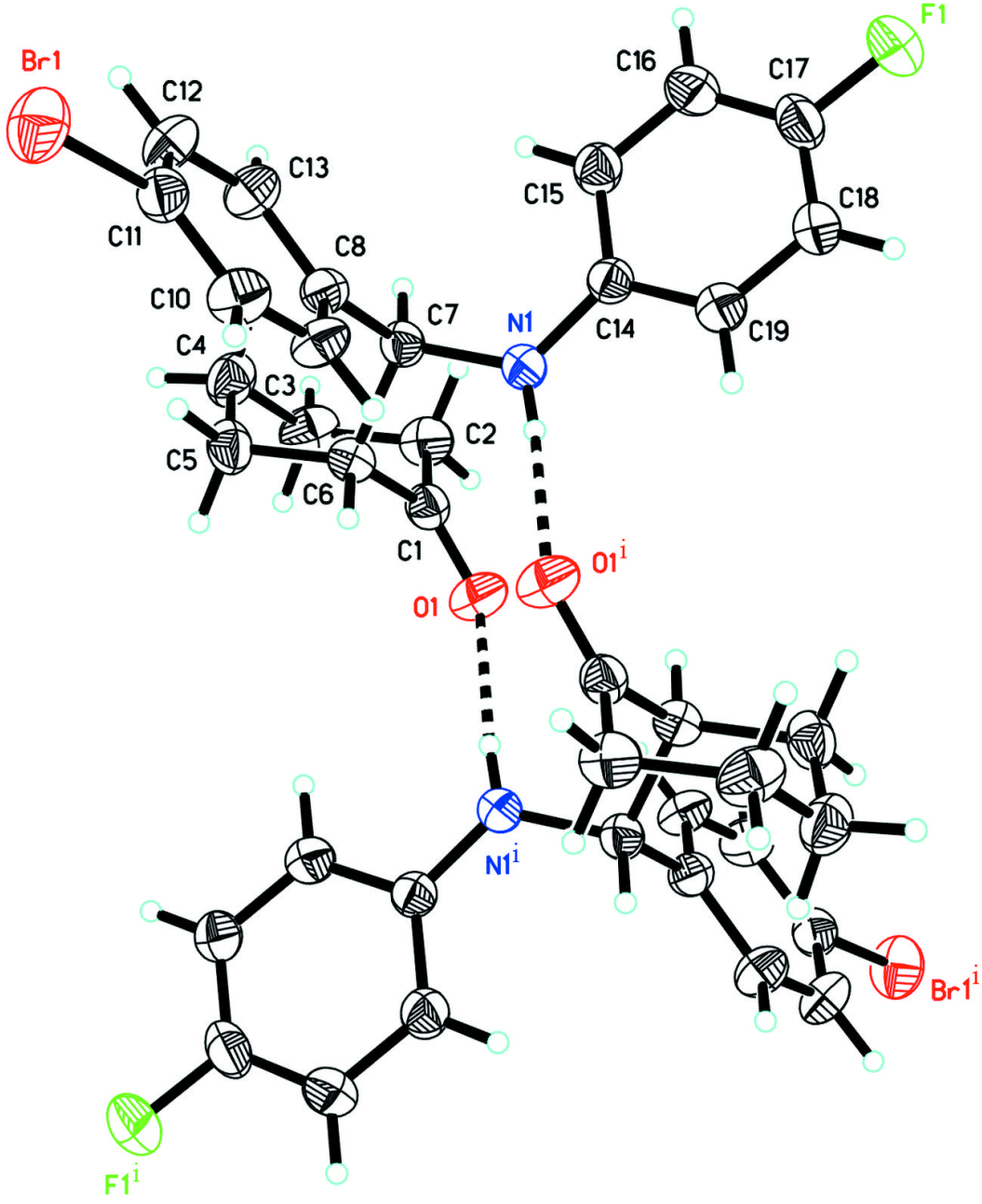
H5A—C5—H5B	107.8	C15—C16—H16	120.7
C1—C6—C7	110.1 (3)	C18—C17—C16	121.9 (4)
C1—C6—C5	107.8 (3)	C18—C17—F1	119.1 (4)
C7—C6—C5	113.5 (3)	C16—C17—F1	119.0 (4)
C1—C6—H6	108.4	C17—C18—C19	119.2 (4)
C7—C6—H6	108.4	C17—C18—H18	120.4
C5—C6—H6	108.4	C19—C18—H18	120.4
N1—C7—C8	115.0 (3)	C18—C19—C14	121.6 (4)
N1—C7—C6	106.9 (3)	C18—C19—H19	119.2
C8—C7—C6	110.7 (3)	C14—C19—H19	119.2
O1—C1—C2—C3	119.2 (4)	C7—C8—C9—C10	−176.5 (3)
C6—C1—C2—C3	−55.2 (4)	C8—C9—C10—C11	−1.5 (6)
C1—C2—C3—C4	54.1 (4)	C9—C10—C11—C12	0.3 (6)
C2—C3—C4—C5	−56.3 (4)	C9—C10—C11—Br1	178.9 (3)
C3—C4—C5—C6	56.5 (4)	C10—C11—C12—C13	−0.8 (6)
O1—C1—C6—C7	115.0 (4)	Br1—C11—C12—C13	−179.4 (3)
C2—C1—C6—C7	−70.6 (4)	C11—C12—C13—C8	2.4 (6)
O1—C1—C6—C5	−120.7 (4)	C9—C8—C13—C12	−3.5 (6)
C2—C1—C6—C5	53.7 (4)	C7—C8—C13—C12	176.1 (3)
C4—C5—C6—C1	−53.2 (4)	C7—N1—C14—C15	−16.0 (5)
C4—C5—C6—C7	69.1 (4)	C7—N1—C14—C19	166.3 (3)
C14—N1—C7—C8	−78.0 (4)	N1—C14—C15—C16	−172.5 (3)
C14—N1—C7—C6	158.8 (3)	C19—C14—C15—C16	5.3 (6)
C1—C6—C7—N1	−52.9 (4)	C14—C15—C16—C17	−2.4 (6)
C5—C6—C7—N1	−173.9 (3)	C15—C16—C17—C18	−1.0 (6)
C1—C6—C7—C8	−178.8 (3)	C15—C16—C17—F1	177.7 (3)
C5—C6—C7—C8	60.2 (4)	C16—C17—C18—C19	1.2 (7)
N1—C7—C8—C13	132.0 (4)	F1—C17—C18—C19	−177.4 (4)
C6—C7—C8—C13	−106.8 (4)	C17—C18—C19—C14	1.9 (7)
N1—C7—C8—C9	−48.4 (5)	N1—C14—C19—C18	172.9 (4)
C6—C7—C8—C9	72.8 (4)	C15—C14—C19—C18	−5.0 (6)
C13—C8—C9—C10	3.0 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.81	2.19	2.978 (4)	166

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1


Br1
C12
C13
C11
C8
C10
C7
N1
C4
C3
C2
C5
C6
C1
O1
O1i
C16
C17
F1
C15
C18
C14
C19
N1i
Br1i
F1i