organic compounds

4-Bromo-*N*-(4-methoxy-2-nitrophenyl)-benzamide

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In the title compound, $C_{14}H_{11}BrN_2O_4$, the amide segment makes dihedral angles of 23.4 (2) and 20.5 (2)° with the benzene rings, while the dihedral angle between the bezene rings is 2.90 (8)°. The nitro and methoxy groups are almost coplanar with their bound benzene ring, with the r.m.s. deviation for the 11 non-H atoms being 0.0265 (1) Å. An intramolecular N—H⋯O hydrogen bond generates an $S(6)$ ring motif. In the crystal, molecules are linked into [2$\bar{1}$0] chains by weak C—H⋯O and C—H⋯Br interactions, which form an $R_2^2(8)$ motif between pairs of molecules in the chain. A Br⋯O [3.2018 (12) Å] short contact also occurs.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Johnston & Taylor (2011); Li & Cui (2011); Saeed *et al.* (2008). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986). For standard bond lengths, see: Allen *et al.* (1987).
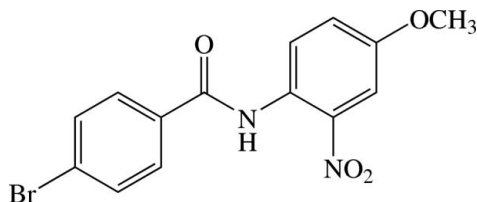
Experimental

Crystal data

C₁₄H₁₁BrN₂O₄   *M*ᵣ = 351.15

Triclinic, *P*$\bar{1}$
a = 6.1219 (2) Å
b = 7.6519 (3) Å
c = 14.3504 (6) Å
α = 89.197 (1)°
β = 84.795 (1)°
γ = 77.983 (1)°
V = 654.78 (4) Å³
Z = 2
Mo *K*α radiation
μ = 3.16 mm⁻¹
T = 100 K
0.54 × 0.27 × 0.17 mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
*T*ₘᵢₙ = 0.281, *T*ₘₐₓ = 0.616
14195 measured reflections
3725 independent reflections
3558 reflections with *I* > 2σ(*I*)
*R*ᵢₙₜ = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.080$
$S = 1.12$
3725 reflections
195 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.93$ e Å⁻³
$\Delta\rho_{min} = -0.48$ e Å⁻³

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1*N*1⋯O2	0.84 (3)	1.99 (3)	2.6318 (19)	132 (2)
C3—H3*A*⋯O4ⁱ	0.95	2.57	3.475 (2)	160
C12—H12*A*⋯O1ⁱⁱ	0.95	2.41	3.358 (2)	172
C10—H10*A*⋯Br1ⁱⁱⁱ	0.95	2.93	3.863 (2)	167

Symmetry codes: (i) $x - 2, y + 1, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $x + 2, y - 1, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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supporting information

4-Bromo-*N*-(4-methoxy-2-nitrophenyl)benzamide

Weerawat Sripet, Suchada Chantrapromma, Pumsak Ruanwas and Hoong-Kun Fun

S1. Comment

As part of our research in medicinal chemistry, the title benzamide derivative (I) was synthesized with a hope that it may exhibit anticancer and/or anti-alzheimer activities. Herein, its crystal structure was reported.

The molecule of the title benzamide derivative (Fig. 1), $C_{14}H_{11}BrN_2O_4$, is not planar as the plane of the middle N-C=O segment makes the dihedral angles of 23.4 (2) and 20.5 (2) ° with the C1–C6 and C8–C13 benzene rings, respectively whereas the dihedral angle between the two benzene rings is 2.90 (8)°. In the 4-methoxy-2-nitrophenyl moiety, the nitro and methoxy groups are co-planar with the bound benzene ring with the r.m.s. deviation of 0.0265 (1) Å for the eleven non-H atoms [C8–C14/N2/O2–O4] and the torsion angles O2–N2–C9–C8 = -3.8 (2)°, O3–N2–C9–C8 = 175.88 (15)° and C14–O4–C11–C12 = 3.5 (2)°. An intramolecular N1—H1N1···O2 hydrogen bond generates a S(6) ring motif (Bernstein *et al.*, 1995). Bond distances are comparable with those in related structures (Johnston & Taylor, 2011; Li & Cui, 2011 and Saeed *et al.*, 2008).

In the crystal (Fig. 2), the molecules are linked into [210] chains by weak C—H···O and C—H···Br interactions forming $R_2^2(8)$ motifs. Br1···O2^{iii}[3.2018 (12) Å; (iii) = -x, 2-y, -z] short contact is presented.

S2. Experimental

A mixture of 4-bromobenzoyl chloride (0.20 g, 0.91 mmol) and 4-metoxy-2-nitroaniline (0.23 g, 1.40 mmol) in anhydrous acetone (20 ml) was refluxed for 4 h. An orange solid was formed, which was filtered and washed with water. Orange blocks of the title compound were recrystallized from ethylacetate by slow evaporation of the solvent at room temperature after a week, Mp. 434-436 K.

S3. Refinement

Amide H atom was located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C-H) = 0.95 Å for aromatic and CH and 0.98 Å for CH_3 atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.75 Å from Br1 and the deepest hole is located at 0.85 Å from Br1.
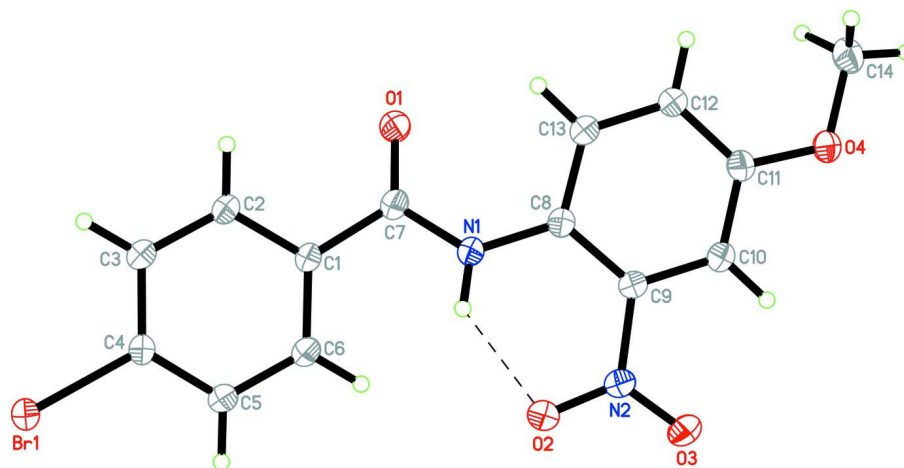
Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids. The N—H···O hydrogen bond is drawn as dash line.
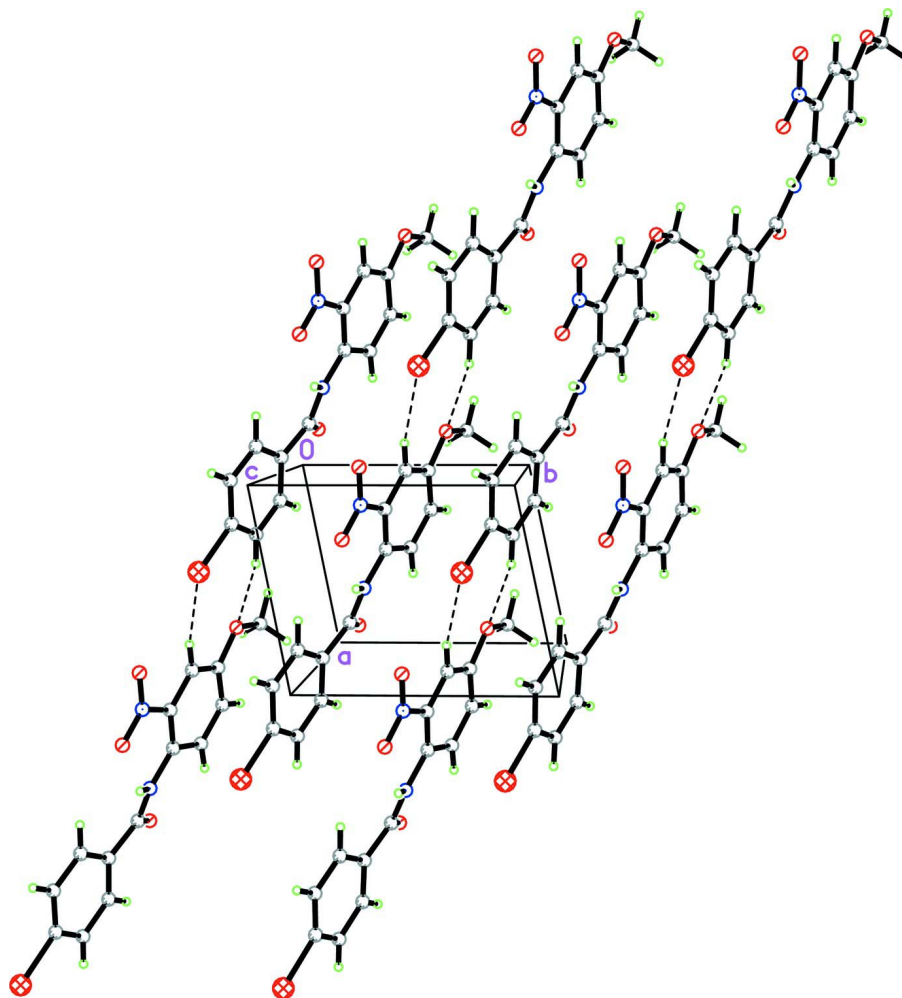
Figure 2

The crystal packing of the title compound viewed along the *c* axis. Hydrogen bonds were drawn as dashed lines.

4-Bromo-*N*-(4-methoxy-2-nitrophenyl)benzamide

Crystal data

$C_{14}H_{11}BrN_2O_4$	$Z = 2$
$M_r = 351.15$	$F(000) = 352$
Triclinic, $P\overline{1}$	$D_x = 1.781$ Mg m^{-3}
Hall symbol: -P 1	Melting point = 434–436 K
$a = 6.1219$ (2) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 7.6519$ (3) Å	Cell parameters from 3725 reflections
$c = 14.3504$ (6) Å	$\theta = 2.9$–30.0°
$\alpha = 89.197$ (1)°	$\mu = 3.16$ mm^{-1}
$\beta = 84.795$ (1)°	$T = 100$ K
$\gamma = 77.983$ (1)°	Block, orange
$V = 654.78$ (4) Å^3	$0.54 \times 0.27 \times 0.17$ mm

Data collection

Bruker APEX DUO CCD area-detector diffractometer	14195 measured reflections
Radiation source: sealed tube	3725 independent reflections
Graphite monochromator	3558 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.022$
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$\theta_{max} = 30.0°$, $\theta_{min} = 2.9°$
	$h = -8 \rightarrow 8$
	$k = -10 \rightarrow 10$
$T_{min} = 0.281$, $T_{max} = 0.616$	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.026$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.080$	
$S = 1.12$	H atoms treated by a mixture of independent and constrained refinement
3725 reflections	
195 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.2246P]$ where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.93$ e Å^{-3}
	$\Delta\rho_{min} = -0.48$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F² against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F², conventional R-factors R are based on F, with F set to zero for negative F². The threshold expression of F² > 2sigma(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² 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_{iso}*/U_{eq}
Br1	−0.43080 (2)	1.27590 (2)	0.081247 (10)	0.02080 (7)
O1	0.2430 (2)	0.77009 (18)	0.38411 (9)	0.0252 (3)
O4	1.2328 (2)	0.25761 (17)	0.36805 (9)	0.0225 (2)
O3	1.0773 (2)	0.5836 (2)	0.08860 (9)	0.0292 (3)
O2	0.7257 (2)	0.70628 (18)	0.09318 (9)	0.0240 (3)
N1	0.4593 (2)	0.69155 (19)	0.24641 (10)	0.0182 (3)
N2	0.8886 (2)	0.61497 (18)	0.12882 (9)	0.0183 (3)
C2	−0.1093 (3)	0.9574 (2)	0.28387 (11)	0.0183 (3)
H2A	−0.1492	0.9142	0.3441	0.022*
C3	−0.2720 (3)	1.0681 (2)	0.23633 (11)	0.0181 (3)
H3A	−0.4229	1.0997	0.2629	0.022*
C4	−0.2085 (3)	1.1314 (2)	0.14887 (11)	0.0165 (3)
C5	0.0114 (3)	1.0909 (2)	0.10918 (11)	0.0178 (3)
H5A	0.0518	1.1394	0.0504	0.021*
C6	0.1719 (3)	0.9777 (2)	0.15721 (11)	0.0179 (3)
H6A	0.3227	0.9473	0.1305	0.021*
C1	0.1126 (3)	0.9086 (2)	0.24441 (11)	0.0163 (3)
C7	0.2764 (3)	0.7849 (2)	0.29928 (11)	0.0176 (3)
C8	0.6493 (3)	0.5785 (2)	0.27797 (11)	0.0164 (3)
C9	0.8567 (3)	0.5414 (2)	0.22300 (10)	0.0166 (3)
C10	1.0464 (3)	0.4333 (2)	0.25465 (11)	0.0175 (3)
H10A	1.1836	0.4113	0.2159	0.021*
C11	1.0359 (3)	0.3573 (2)	0.34300 (11)	0.0173 (3)
C12	0.8314 (3)	0.3867 (2)	0.39815 (11)	0.0187 (3)
H12A	0.8215	0.3326	0.4579	0.022*
C13	0.6427 (3)	0.4951 (2)	0.36556 (11)	0.0184 (3)
H13A	0.5048	0.5132	0.4038	0.022*
C14	1.2322 (3)	0.1852 (2)	0.46014 (13)	0.0249 (3)
H14A	1.3862	0.1315	0.4731	0.037*
H14B	1.1412	0.0937	0.4650	0.037*
H14C	1.1689	0.2807	0.5057	0.037*
H1N1	0.467 (4)	0.710 (4)	0.1886 (19)	0.028 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01483 (10)	0.02630 (10)	0.01952 (10)	−0.00065 (6)	−0.00143 (6)	0.00490 (6)
O1	0.0244 (6)	0.0295 (6)	0.0164 (5)	0.0048 (5)	0.0024 (4)	0.0015 (5)
O4	0.0157 (6)	0.0283 (6)	0.0209 (6)	0.0013 (4)	−0.0021 (4)	0.0057 (4)
O3	0.0174 (6)	0.0415 (7)	0.0236 (6)	0.0013 (5)	0.0065 (5)	0.0085 (5)
O2	0.0190 (6)	0.0306 (6)	0.0189 (5)	0.0019 (5)	0.0001 (4)	0.0063 (5)
N1	0.0159 (6)	0.0215 (6)	0.0146 (6)	0.0012 (5)	0.0004 (5)	0.0015 (5)
N2	0.0187 (7)	0.0196 (6)	0.0156 (6)	−0.0027 (5)	0.0005 (5)	0.0015 (5)
C2	0.0176 (7)	0.0208 (7)	0.0153 (6)	−0.0020 (5)	0.0009 (5)	0.0004 (5)
C3	0.0138 (7)	0.0211 (7)	0.0180 (7)	−0.0015 (5)	0.0016 (5)	0.0001 (5)

C4	0.0136 (7)	0.0180 (6)	0.0171 (7)	−0.0012 (5)	−0.0011 (5)	0.0004 (5)
C5	0.0156 (7)	0.0196 (7)	0.0168 (7)	−0.0017 (5)	0.0018 (5)	0.0012 (5)
C6	0.0146 (7)	0.0195 (6)	0.0180 (7)	−0.0015 (5)	0.0020 (5)	0.0007 (5)
C1	0.0147 (7)	0.0165 (6)	0.0163 (6)	−0.0010 (5)	−0.0001 (5)	−0.0001 (5)
C7	0.0157 (7)	0.0172 (6)	0.0184 (7)	−0.0008 (5)	0.0002 (5)	0.0001 (5)
C8	0.0142 (7)	0.0174 (6)	0.0166 (6)	−0.0009 (5)	−0.0012 (5)	0.0000 (5)
C9	0.0172 (7)	0.0177 (6)	0.0142 (6)	−0.0032 (5)	0.0007 (5)	0.0009 (5)
C10	0.0142 (7)	0.0190 (6)	0.0185 (7)	−0.0025 (5)	0.0010 (5)	−0.0001 (5)
C11	0.0140 (7)	0.0182 (6)	0.0192 (7)	−0.0016 (5)	−0.0027 (5)	0.0005 (5)
C12	0.0191 (7)	0.0196 (6)	0.0160 (7)	−0.0016 (5)	−0.0002 (5)	0.0024 (5)
C13	0.0152 (7)	0.0208 (7)	0.0174 (7)	−0.0012 (5)	0.0013 (5)	0.0012 (5)
C14	0.0237 (9)	0.0254 (8)	0.0233 (8)	0.0014 (6)	−0.0053 (6)	0.0037 (6)

Geometric parameters (Å, º)

Br1—C4	1.8975 (16)	C5—H5A	0.9500
O1—C7	1.224 (2)	C6—C1	1.399 (2)
O4—C11	1.3615 (19)	C6—H6A	0.9500
O4—C14	1.426 (2)	C1—C7	1.499 (2)
O3—N2	1.2220 (19)	C8—C13	1.403 (2)
O2—N2	1.2395 (19)	C8—C9	1.410 (2)
N1—C7	1.367 (2)	C9—C10	1.387 (2)
N1—C8	1.404 (2)	C10—C11	1.389 (2)
N1—H1N1	0.84 (3)	C10—H10A	0.9500
N2—C9	1.4692 (19)	C11—C12	1.397 (2)
C2—C3	1.390 (2)	C12—C13	1.389 (2)
C2—C1	1.400 (2)	C12—H12A	0.9500
C2—H2A	0.9500	C13—H13A	0.9500
C3—C4	1.391 (2)	C14—H14A	0.9800
C3—H3A	0.9500	C14—H14B	0.9800
C4—C5	1.387 (2)	C14—H14C	0.9800
C5—C6	1.394 (2)		
C11—O4—C14	117.04 (13)	O1—C7—C1	121.42 (14)
C7—N1—C8	127.66 (14)	N1—C7—C1	114.35 (13)
C7—N1—H1N1	117.3 (19)	C13—C8—N1	121.83 (14)
C8—N1—H1N1	114.8 (19)	C13—C8—C9	116.34 (14)
O3—N2—O2	122.41 (14)	N1—C8—C9	121.82 (14)
O3—N2—C9	118.06 (14)	C10—C9—C8	122.13 (14)
O2—N2—C9	119.53 (13)	C10—C9—N2	115.12 (13)
C3—C2—C1	121.01 (14)	C8—C9—N2	122.75 (14)
C3—C2—H2A	119.5	C9—C10—C11	120.04 (14)
C1—C2—H2A	119.5	C9—C10—H10A	120.0
C2—C3—C4	118.32 (14)	C11—C10—H10A	120.0
C2—C3—H3A	120.8	O4—C11—C10	115.23 (14)
C4—C3—H3A	120.8	O4—C11—C12	125.42 (14)
C5—C4—C3	122.18 (15)	C10—C11—C12	119.35 (14)
C5—C4—Br1	119.02 (11)	C13—C12—C11	119.98 (14)

C3—C4—Br1	118.80 (12)	C13—C12—H12A	120.0
C4—C5—C6	118.73 (14)	C11—C12—H12A	120.0
C4—C5—H5A	120.6	C12—C13—C8	122.10 (14)
C6—C5—H5A	120.6	C12—C13—H13A	119.0
C5—C6—C1	120.52 (14)	C8—C13—H13A	119.0
C5—C6—H6A	119.7	O4—C14—H14A	109.5
C1—C6—H6A	119.7	O4—C14—H14B	109.5
C6—C1—C2	119.17 (14)	H14A—C14—H14B	109.5
C6—C1—C7	123.21 (14)	O4—C14—H14C	109.5
C2—C1—C7	117.61 (13)	H14A—C14—H14C	109.5
O1—C7—N1	124.23 (15)	H14B—C14—H14C	109.5
C1—C2—C3—C4	−0.9 (2)	N1—C8—C9—C10	178.41 (14)
C2—C3—C4—C5	−1.5 (2)	C13—C8—C9—N2	178.41 (14)
C2—C3—C4—Br1	177.86 (12)	N1—C8—C9—N2	−1.0 (2)
C3—C4—C5—C6	2.4 (2)	O3—N2—C9—C10	−3.6 (2)
Br1—C4—C5—C6	−176.97 (12)	O2—N2—C9—C10	176.71 (14)
C4—C5—C6—C1	−0.9 (2)	O3—N2—C9—C8	175.88 (15)
C5—C6—C1—C2	−1.3 (2)	O2—N2—C9—C8	−3.8 (2)
C5—C6—C1—C7	179.40 (14)	C8—C9—C10—C11	0.2 (2)
C3—C2—C1—C6	2.2 (2)	N2—C9—C10—C11	179.64 (14)
C3—C2—C1—C7	−178.44 (14)	C14—O4—C11—C10	176.68 (14)
C8—N1—C7—O1	−6.9 (3)	C14—O4—C11—C12	−3.5 (2)
C8—N1—C7—C1	173.95 (14)	C9—C10—C11—O4	−178.33 (14)
C6—C1—C7—O1	157.07 (16)	C9—C10—C11—C12	1.8 (2)
C2—C1—C7—O1	−22.2 (2)	O4—C11—C12—C13	178.40 (15)
C6—C1—C7—N1	−23.7 (2)	C10—C11—C12—C13	−1.8 (2)
C2—C1—C7—N1	156.97 (15)	C11—C12—C13—C8	−0.3 (2)
C7—N1—C8—C13	23.9 (2)	N1—C8—C13—C12	−178.36 (15)
C7—N1—C8—C9	−156.70 (16)	C9—C8—C13—C12	2.2 (2)
C13—C8—C9—C10	−2.2 (2)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O2	0.84 (3)	1.99 (3)	2.6318 (19)	132 (2)
C3—H3*A*···O4ⁱ	0.95	2.57	3.475 (2)	160
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C10—H10*A*···Br1ⁱⁱⁱ	0.95	2.93	3.863 (2)	167

Symmetry codes: (i) *x*−2, *y*+1, *z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*+2, *y*−1, *z*.