Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N′-[(*E*)-1-(5-Bromo-2-hydroxyphenyl)-ethylidene]-4-nitrobenzohydrazide

Chang-Zheng Zheng, Liang Wang,* Juan Liu and Yu-Jie Wang

College of Environment and Chemical Engineering, Xi'an Polytechnic University, 710048 Xi'an,Shaanxi, People's Republic of China
Correspondence e-mail: wllily315668256@yahoo.com.cn

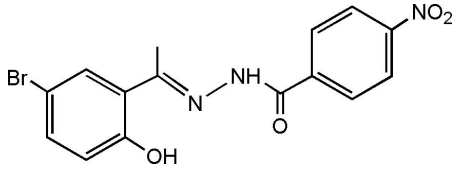
Received 25 May 2011; accepted 16 June 2011

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.049; wR factor = 0.157; data-to-parameter ratio = 12.0.

The title compound, $C_{15}H_{12}BrN_3O_4$, displays a *trans* conformation with respect to the C=N double bond. The central atoms around the C=N double bond are not coplanar, in contrast to the aromatic rings, which exhibit a dihedral angle of 1.80 (4)° between their mean planes. An intramolecular O—H···N hydrogen bond occurs. In the crystal, molecules are connected *via* intermolecular N—H···O hydrogen bonding into chains along the *a* axis.

Related literature

For the coordination properties of aroylhydrazones, see: Ali *et al.* (2004); Carcelli *et al.* (1995); Zhang *et al.* (2011); Zheng *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{12}BrN_3O_4$
M_r = 378.19
Orthorhombic, $Pna2_1$
a = 40.381 (13) Å
b = 5.0598 (16) Å
c = 7.168 (2) Å
V = 1464.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.83 mm^{-1}
T = 298 K
0.35 × 0.23 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.441, T_{max} = 0.689
6753 measured reflections
2520 independent reflections
2074 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.157
S = 0.95
2520 reflections
210 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}
Absolute structure: Flack (1983), 1075 Friedel pairs
Flack parameter: 0.01 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.23	2.981 (6)	146
O1—H1···N1	0.82	1.81	2.531 (7)	145

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

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1996); 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*.

The authors thank the National Natural Science Foundation of Shaanxi Province, China (2009JM2012) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2106).

References

Ali, H. M., Khamis, N. A. & Yamin, B. M. (2004). *Acta Cryst.* E**60**, m1708–m1709.
Bruker (1996). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carcelli, M., Mazza, P., Pelizzi, G. & Zani, F. (1995). *J. Inorg. Biochem.* **57**, 43–62.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhang, J.-M., Wang, L., Liu, J., Li, Y.-C. & Li, H.-J. (2011). *Acta Cryst.* E**67**, m537.
Zheng, C.-Z., Ji, C.-Y., Chang, X.-L. & Zhang, L. (2008). *Acta Cryst.* E**64**, o2487.

supporting information

Acta Cryst. (2011). E**67**, o1809 [doi:10.1107/S1600536811023609]

N′-[(*E*)-1-(5-Bromo-2-hydroxyphenyl)ethylidene]-4-nitrobenzohydrazide

Chang-Zheng Zheng, Liang Wang, Juan Liu and Yu-Jie Wang

S1. Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Zhang *et al.*, 2011; Zheng *et al.*, 2008; Ali *et al.*, 2004) and their biological activity (Carcelli *et al.*, 1995). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound, $C_{15}H_{12}N_3O_4Br$, was synthesized and its crystal structure is reported here.

The title compound, $C_{15}H_{12}N_3O_4Br$, displays a *trans* conformation with respect to the C=N double bond (Fig. 1). The central atoms around the C=N double bond are not coplanar since the dihedral angle C7—N1—N2—C9 is 154.7 (5)° in contrast to the aromatic rings which exhibit a dihedral angle of 1.80 (4)° between their mean planes. In the crystal structure, one intramolecular O—H···N hydrogen bond occurs (Table 1). The molecules are connected via intermolecular N—H···O into one-dimensional linear chains along the *a* axis (Table 1; Fig. 2).

S2. Experimental

Ethyl 4-nitrobenzoate (9.76 g, 0.05 mol) was dissolved in ethanol (40 ml) at room temperature and heated at 363 K, followed by the addition of hydrazine hydrate (2.50 g, 0.05 mol). Subsequently, the mixture was refluxed for 10 h, and then cooled to room temperature. The crystals were precipitated and collected by filtration. The product was recrystallized from ethanol and dried under reduced pressure to give compound of 4-nitrobenzhydrazide. 4-Nitrobenzhydrazide (4.53 g, 0.025 mol) was dissolved in ethanol (20 ml) at room temperature and heated at 363 K, followed by the addition of 5-bromo-2-hydroxyphenyl ethyl ketone (5.38 g, 0.025 mol). Subsequently, the mixture was refluxed for 9 h, and then cooled to room temperature. The crystals were precipitated and collected by filtration. The product was recrystallized from ethanol and dried under reduced pressure to the title compound.

S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with *C*—*H* (methyl) = 0.96 Å, C—H (aromatic) = 0.93 Å, O—H = 0.82 Å, N—H = 0.86 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, O) and 1.2U_{eq}($C_{aromatic}$, N).

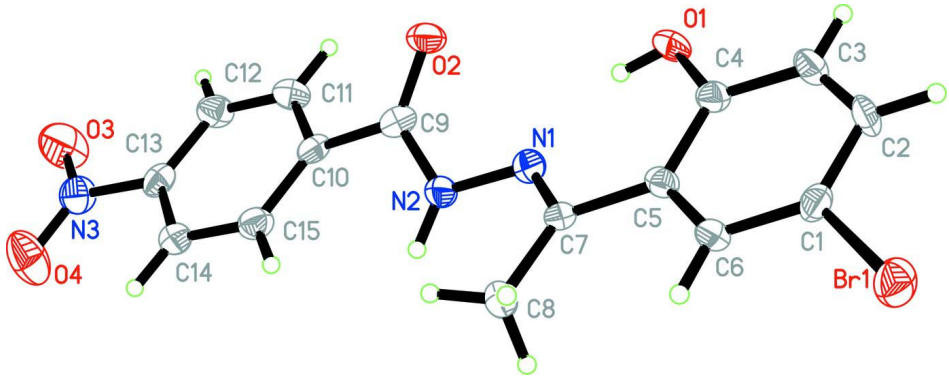


Figure 1

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

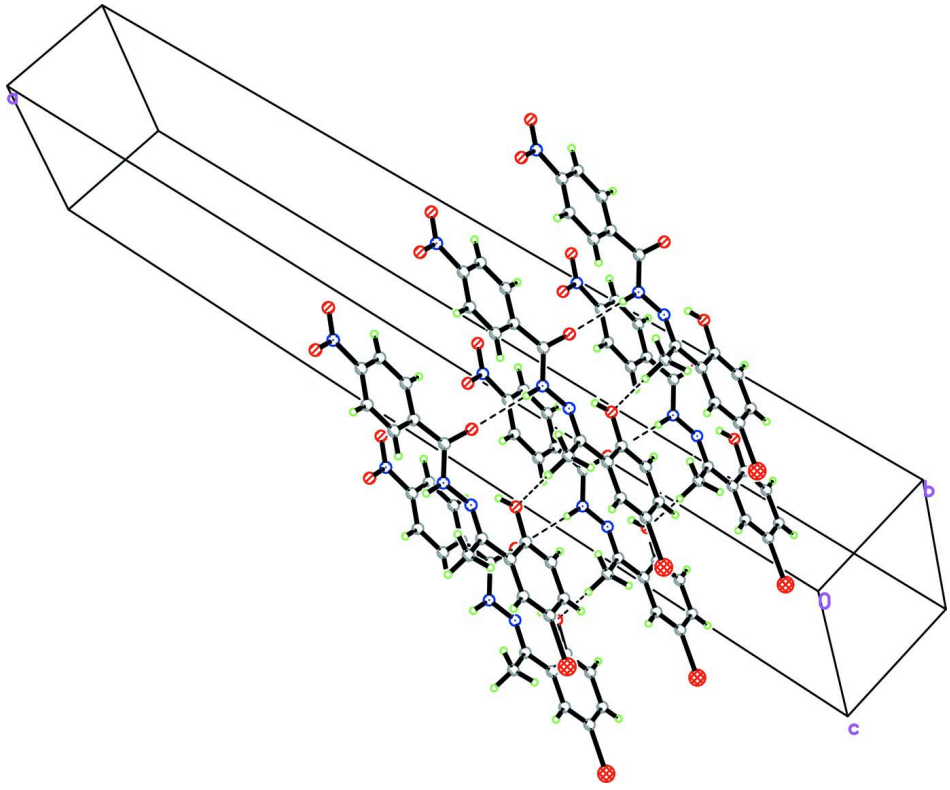


Figure 2

The crystal packing of the title compound viewed along the *a* axis. Dashed lines show intra- and intermolecular hydrogen bonds.

N′-[(*E*)-1-(5-Bromo-2-hydroxyphenyl)ethylidene]-4- nitrobenzohydrazide

Crystal data

$C_{15}H_{12}BrN_3O_4$	a = 40.381 (13) Å
M_r = 378.19	b = 5.0598 (16) Å
Orthorhombic, $Pna2_1$	c = 7.168 (2) Å
Hall symbol: P 2c -2n	V = 1464.5 (8) Å^3

$Z = 4$
$F(000) = 760$
$D_x = 1.715$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2077 reflections

$\theta = 3.0$–$23.4°$
$\mu = 2.83$ mm^{-1}
$T = 298$ K
Block, red
0.35 × 0.23 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.441$, $T_{max} = 0.689$

6753 measured reflections
2520 independent reflections
2074 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.0°$
$h = -39 \rightarrow 48$
$k = -6 \rightarrow 6$
$l = -7 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.157$
$S = 0.95$
2520 reflections
210 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.120P)^2 + 0.2524P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.95$ e Å^{-3}
$\Delta\rho_{min} = -0.75$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0113 (15)
Absolute structure: Flack (1983), 1075 Friedel pairs
Absolute structure parameter: 0.01 (2)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.748669 (14)	0.63795 (13)	0.8107 (3)	0.0527 (3)
N1	0.63083 (12)	0.3604 (9)	0.2325 (8)	0.0407 (12)
N2	0.61516 (12)	0.4290 (9)	0.0655 (7)	0.0398 (12)
H2A	0.6165	0.5855	0.0194	0.048*
N3	0.53035 (13)	0.4393 (12)	−0.7014 (9)	0.0537 (14)
O1	0.63214 (10)	0.0383 (8)	0.5025 (7)	0.0454 (10)
H1	0.6246	0.1078	0.4084	0.068*
O2	0.59928 (11)	0.0029 (8)	0.0369 (7)	0.0534 (12)

O3	0.50841 (18)	0.2902 (13)	−0.7491 (10)	0.094 (2)
O4	0.53936 (16)	0.6248 (14)	−0.7978 (10)	0.087 (2)
C1	0.71150 (13)	0.4542 (11)	0.7103 (8)	0.0383 (13)
C2	0.69852 (14)	0.2403 (12)	0.8090 (10)	0.0460 (13)
H2	0.7078	0.1873	0.9219	0.055*
C3	0.67161 (18)	0.1098 (12)	0.7345 (10)	0.0477 (16)
H3	0.6625	−0.0310	0.8000	0.057*
C4	0.65783 (15)	0.1816 (11)	0.5655 (9)	0.0363 (13)
C5	0.67081 (14)	0.4039 (10)	0.4651 (8)	0.0330 (12)
C6	0.69784 (13)	0.5350 (10)	0.5410 (8)	0.0352 (12)
H6	0.7070	0.6784	0.4781	0.042*
C7	0.65665 (13)	0.4916 (10)	0.2846 (7)	0.0329 (12)
C8	0.67260 (16)	0.7035 (12)	0.1733 (9)	0.0434 (15)
H8A	0.6657	0.6895	0.0455	0.065*
H8B	0.6962	0.6853	0.1804	0.065*
H8C	0.6662	0.8728	0.2218	0.065*
C9	0.59767 (14)	0.2338 (11)	−0.0194 (9)	0.0405 (14)
C10	0.57928 (12)	0.3074 (10)	−0.1896 (10)	0.0353 (11)
C11	0.55293 (16)	0.1454 (12)	−0.2431 (10)	0.0451 (17)
H11	0.5465	0.0060	−0.1667	0.054*
C12	0.53649 (15)	0.1907 (12)	−0.4076 (10)	0.0462 (15)
H12	0.5186	0.0856	−0.4425	0.055*
C13	0.54701 (15)	0.3955 (11)	−0.5200 (9)	0.0411 (14)
C14	0.57239 (14)	0.5633 (11)	−0.4717 (9)	0.0410 (13)
H14	0.5786	0.7022	−0.5492	0.049*
C15	0.58848 (15)	0.5181 (11)	−0.3029 (9)	0.0432 (14)
H15	0.6055	0.6298	−0.2655	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0513 (4)	0.0578 (4)	0.0490 (4)	0.0046 (3)	−0.0137 (3)	−0.0030 (5)
N1	0.050 (3)	0.036 (3)	0.035 (3)	0.004 (2)	−0.007 (2)	0.000 (2)
N2	0.053 (3)	0.032 (2)	0.034 (3)	−0.004 (2)	−0.011 (2)	0.002 (2)
N3	0.055 (3)	0.057 (3)	0.050 (4)	0.001 (2)	−0.015 (3)	−0.001 (3)
O1	0.060 (3)	0.030 (2)	0.046 (3)	−0.0088 (19)	0.002 (2)	0.013 (2)
O2	0.077 (3)	0.030 (2)	0.054 (3)	−0.003 (2)	−0.018 (2)	0.001 (2)
O3	0.112 (4)	0.084 (4)	0.086 (6)	−0.029 (4)	−0.056 (4)	0.015 (3)
O4	0.079 (4)	0.115 (5)	0.067 (4)	−0.022 (3)	−0.021 (3)	0.032 (4)
C1	0.046 (3)	0.039 (3)	0.031 (3)	0.007 (2)	−0.004 (2)	−0.010 (3)
C2	0.066 (3)	0.047 (3)	0.025 (3)	0.011 (3)	−0.004 (3)	0.013 (3)
C3	0.072 (4)	0.038 (3)	0.033 (3)	0.005 (3)	0.006 (3)	0.012 (3)
C4	0.044 (3)	0.029 (3)	0.035 (3)	0.005 (2)	0.005 (2)	0.001 (2)
C5	0.037 (3)	0.028 (3)	0.034 (3)	0.005 (2)	0.006 (2)	0.004 (2)
C6	0.044 (3)	0.028 (3)	0.034 (3)	0.000 (2)	0.002 (2)	0.006 (2)
C7	0.044 (3)	0.023 (2)	0.031 (3)	0.003 (2)	0.001 (2)	−0.002 (2)
C8	0.056 (4)	0.041 (3)	0.033 (4)	−0.004 (3)	−0.005 (3)	0.005 (3)
C9	0.039 (3)	0.034 (3)	0.048 (4)	0.000 (3)	0.000 (3)	−0.009 (3)

C10	0.037 (2)	0.030 (2)	0.039 (3)	0.0031 (19)	−0.005 (3)	−0.009 (3)
C11	0.045 (3)	0.035 (3)	0.055 (5)	−0.004 (2)	0.001 (3)	0.004 (3)
C12	0.044 (3)	0.041 (3)	0.053 (4)	−0.007 (3)	−0.009 (3)	0.002 (3)
C13	0.040 (3)	0.044 (3)	0.040 (4)	0.003 (2)	−0.007 (2)	−0.012 (3)
C14	0.048 (3)	0.036 (3)	0.039 (4)	−0.002 (3)	−0.002 (3)	−0.002 (3)
C15	0.047 (3)	0.036 (3)	0.047 (4)	−0.004 (3)	−0.003 (3)	−0.005 (3)

Geometric parameters (Å, º)

Br1—C1	1.906 (6)	C5—C6	1.388 (8)
N1—C7	1.291 (7)	C5—C7	1.483 (8)
N1—N2	1.397 (7)	C6—H6	0.9300
N2—C9	1.358 (7)	C7—C8	1.484 (8)
N2—H2A	0.8600	C8—H8A	0.9600
N3—O3	1.213 (8)	C8—H8B	0.9600
N3—O4	1.221 (8)	C8—H8C	0.9600
N3—C13	1.480 (8)	C9—C10	1.476 (9)
O1—C4	1.344 (8)	C10—C15	1.391 (9)
O1—H1	0.8200	C10—C11	1.396 (8)
O2—C9	1.238 (7)	C11—C12	1.373 (10)
C1—C6	1.394 (8)	C11—H11	0.9300
C1—C2	1.395 (9)	C12—C13	1.380 (9)
C2—C3	1.380 (10)	C12—H12	0.9300
C2—H2	0.9300	C13—C14	1.375 (8)
C3—C4	1.381 (10)	C14—C15	1.393 (9)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.435 (8)	C15—H15	0.9300
C7—N1—N2	119.1 (5)	C5—C7—C8	121.2 (5)
C9—N2—N1	116.0 (5)	C7—C8—H8A	109.5
C9—N2—H2A	122.0	C7—C8—H8B	109.5
N1—N2—H2A	122.0	H8A—C8—H8B	109.5
O3—N3—O4	122.4 (7)	C7—C8—H8C	109.5
O3—N3—C13	119.1 (6)	H8A—C8—H8C	109.5
O4—N3—C13	118.5 (5)	H8B—C8—H8C	109.5
C4—O1—H1	109.5	O2—C9—N2	120.8 (6)
C6—C1—C2	121.3 (5)	O2—C9—C10	122.3 (5)
C6—C1—Br1	119.8 (4)	N2—C9—C10	116.7 (5)
C2—C1—Br1	118.9 (5)	C15—C10—C11	119.6 (6)
C3—C2—C1	118.1 (6)	C15—C10—C9	122.8 (5)
C3—C2—H2	121.0	C11—C10—C9	117.5 (5)
C1—C2—H2	121.0	C12—C11—C10	120.4 (6)
C2—C3—C4	122.1 (6)	C12—C11—H11	119.8
C2—C3—H3	119.0	C10—C11—H11	119.8
C4—C3—H3	119.0	C11—C12—C13	118.6 (6)
O1—C4—C3	117.6 (6)	C11—C12—H12	120.7
O1—C4—C5	122.4 (5)	C13—C12—H12	120.7
C3—C4—C5	119.9 (6)	C14—C13—C12	123.1 (6)

C6—C5—C4	117.7 (5)	C14—C13—N3	117.8 (6)
C6—C5—C7	120.1 (5)	C12—C13—N3	119.0 (5)
C4—C5—C7	122.1 (5)	C13—C14—C15	117.7 (6)
C5—C6—C1	120.8 (5)	C13—C14—H14	121.1
C5—C6—H6	119.6	C15—C14—H14	121.1
C1—C6—H6	119.6	C10—C15—C14	120.6 (5)
N1—C7—C5	114.2 (5)	C10—C15—H15	119.7
N1—C7—C8	124.5 (5)	C14—C15—H15	119.7
C7—N1—N2—C9	154.7 (5)	N1—N2—C9—O2	−8.8 (8)
C6—C1—C2—C3	−0.1 (9)	N1—N2—C9—C10	176.3 (5)
Br1—C1—C2—C3	179.9 (5)	O2—C9—C10—C15	−149.2 (6)
C1—C2—C3—C4	1.2 (10)	N2—C9—C10—C15	25.6 (8)
C2—C3—C4—O1	178.5 (6)	O2—C9—C10—C11	26.5 (8)
C2—C3—C4—C5	−2.2 (9)	N2—C9—C10—C11	−158.7 (5)
O1—C4—C5—C6	−178.7 (5)	C15—C10—C11—C12	1.0 (9)
C3—C4—C5—C6	1.9 (8)	C9—C10—C11—C12	−174.9 (5)
O1—C4—C5—C7	0.2 (8)	C10—C11—C12—C13	1.3 (10)
C3—C4—C5—C7	−179.2 (5)	C11—C12—C13—C14	−2.6 (10)
C4—C5—C6—C1	−0.8 (8)	C11—C12—C13—N3	177.5 (6)
C7—C5—C6—C1	−179.8 (5)	O3—N3—C13—C14	178.3 (7)
C2—C1—C6—C5	−0.1 (8)	O4—N3—C13—C14	−1.5 (9)
Br1—C1—C6—C5	179.9 (4)	O3—N3—C13—C12	−1.8 (10)
N2—N1—C7—C5	179.1 (4)	O4—N3—C13—C12	178.4 (7)
N2—N1—C7—C8	−4.0 (8)	C12—C13—C14—C15	1.5 (9)
C6—C5—C7—N1	−177.5 (5)	N3—C13—C14—C15	−178.6 (5)
C4—C5—C7—N1	3.6 (7)	C11—C10—C15—C14	−2.2 (9)
C6—C5—C7—C8	5.5 (8)	C9—C10—C15—C14	173.5 (5)
C4—C5—C7—C8	−173.4 (6)	C13—C14—C15—C10	0.9 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{i}	0.86	2.23	2.981 (6)	146
O1—H1···N1	0.82	1.81	2.531 (7)	145

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