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(*E*)-4-Amino-*N*′-(5-bromo-2-hydroxybenzylidene)benzohydrazide monohydrate

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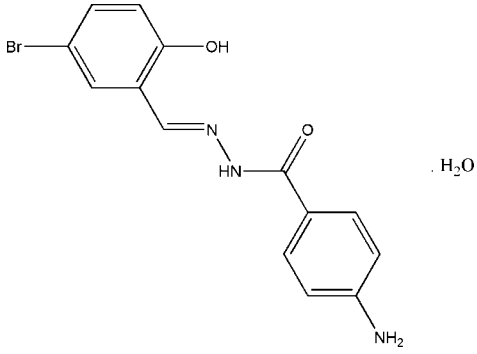
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.077; data-to-parameter ratio = 16.5.

In t the title compound, $C_{14}H_{12}BrN_3O_2.H_2O$, the conformation of the C=N double bond in the hydrazide Schiff base molecule is *E*. The dihedral angle between the benzene rings is 48.01 (11) °. An intramolecular O—H···N hydrogen bond makes an *S*(6) ring motif. In the crystal, molecules are linked through N—H···O (bifurcated acceptor) and O—H···O hydrogen bonds, forming two-dimensional networks lying parallel to (100).

Related literature

For the coordination chemistry of Schiff base and hydrazone derivatives, see: Kucukguzel *et al.* (2006); Karthikeyan *et al.* (2006). For 4-aminobenzohydrazide-derived Schiff base structures, see: Xu (2012); Shi & Li (2012); Bakir & Green (2002). For standard bond lengths, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{12}BrN_3O_2.H_2O$
M_r = 352.19
Monoclinic, $P2_1/c$
a = 15.8435 (11) Å
b = 7.1718 (6) Å
c = 12.6462 (8) Å
β = 101.391 (3)°
V = 1408.64 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.93 mm^{-1}
T = 291 K
0.32 × 0.26 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.454, T_{max} = 0.565
11798 measured reflections
3138 independent reflections
2543 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.04
3138 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*O*···N3	0.93	1.84	2.660 (2)	145
N2—H2*N*···O1*W*^{i}	0.90	1.93	2.823 (2)	171
N1—H2*N*1···O1*W*^{ii}	0.89	2.57	3.276 (3)	137
O1*W*—H2*W*1···O1	0.90	1.77	2.653 (2)	167

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2450).

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supplementary materials

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(*E*)-4-Amino-*N*′-(5-bromo-2-hydroxybenzylidene)benzohydrazide monohydrate

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Comment

Schiff bases are one of the most prevalent mixed-donor ligands in the field of coordination chemistry. They play an important role in the development of coordination chemistry related to catalysis and magnetism, and supramolecular architectures (Karthikeyan *et al.*, 2006; Kucukguzel *et al.*, 2006). Structures of Schiff bases derived from substituted 4-aminobenzohydrazide have been reported earlier (Xu, 2012; Shi & Li, 2012; Bakir & Green, 2002). In order to explore the structure of the new Schiff base derivatives, the title compound was prepared and characterized crystallographically.

The asymmetric unit of the title compound, Fig. 1, comprises a molecule of the title hydrazide Schiff base and a water molecule of crystallization. The hydrazide Schiff base shows an *E* conformation around the C═N double bond. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to those reported for related structures (Xu, 2012; Shi & Li, 2012; Bakir & Green, 2002). An intramolecular O—H···N hydrogen bond makes an *S(6)* ring motif (Table 1; Bernstein *et al.*, 1995). The dihedral angle between the benzene rings is 48.01 (11)Å.

In the crystal, molecules are linked through N—H···O [bifurcated acceptor] and O—H···O hydrogen bonds forming two-dimensional networks lying parallel to (100) [Table 1 and Fig. 2].

Experimental

The title compound was synthesized by adding 1 mmol of methyl 4-aminobenzoate to a solution of 5-Bromosalicyl-aldehyde (1 mmol) in methanol (30 ml). The mixture was refluxed with stirring for 50 min and after cooling to room temperature a light-yellow precipitate was filtered and washed with diethylether and dried in air. Light-yellow prismatic crystals of the title compound, suitable for *X*-ray structure analysis, were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

The N- and O-bound H-atoms were located in a difference Fourier map and were constrained to ride on the parent atom with U_{iso}(H) = 1.2U_{eq}(N) and 1.5U_{eq}(O). The C-bound H atoms were included in calculated positions and treated by the riding model approximation: C—H = 0.93 Å with U_{iso} (H) = 1.2U_{eq}(C).

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); 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* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

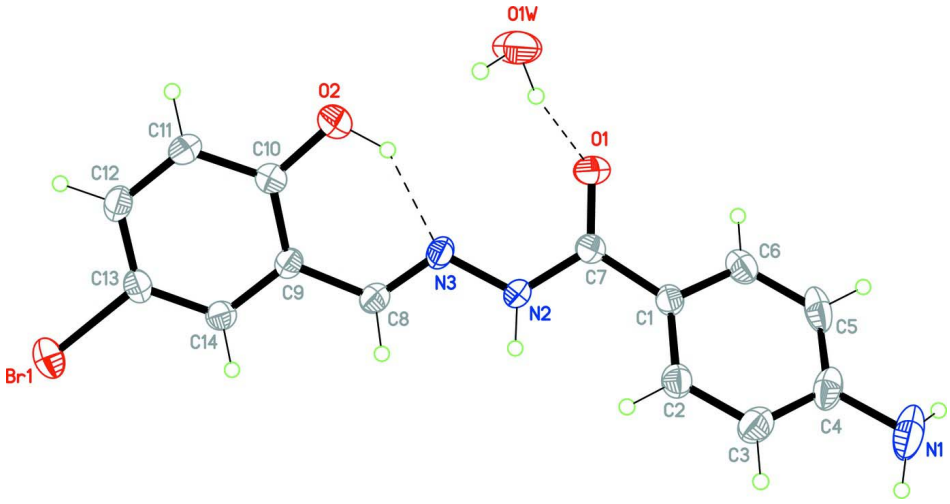

Figure 1

A view of the molecular structure of the title compound, showing 40% probability displacement ellipsoids and the atom numbering. The dashed lines show the intramolecular N-H···O hydrogen bonds (see Table 1 for details).

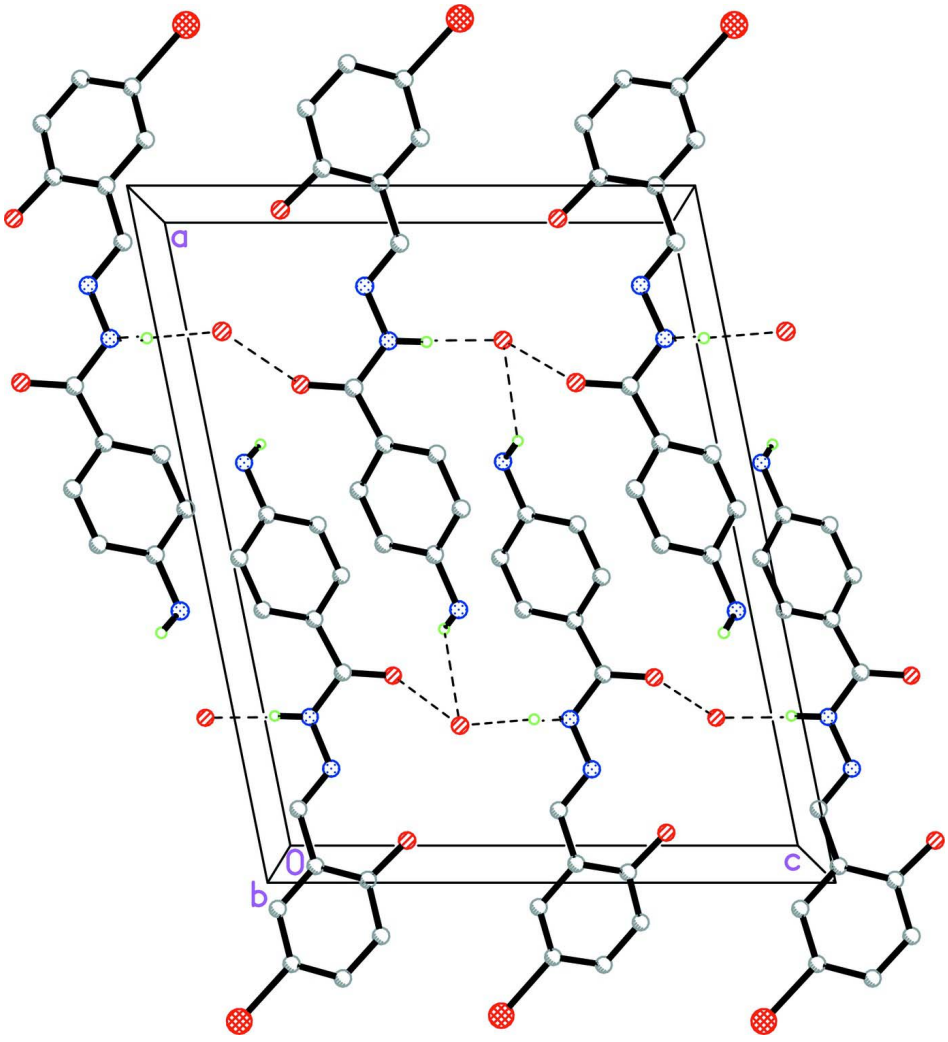

Figure 2

A view along the *b* axis of crystal packing of the title compound, showing the linking of molecules through N—H···O and O—H···O hydrogen bonds (dashed lines; see Table 1 for details; only the NH H atom is shown).

(*E*)-4-Amino-*N*′-(5-bromo-2-hydroxybenzylidene)benzohydrazide monohydrate

Crystal data

$C_{14}H_{12}BrN_3O_2·H_2O$
$M_r = 352.19$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.8435\ (11)$ Å
$b = 7.1718\ (6)$ Å
$c = 12.6462\ (8)$ Å
$\beta = 101.391\ (3)°$
$V = 1408.64\ (18)$ Å^3
$Z = 4$
$F(000) = 712$
$D_x = 1.661$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2225 reflections
$\theta = 2.5–27.5°$
$\mu = 2.93$ mm^{-1}
$T = 291$ K
Prism, white
0.32 × 0.26 × 0.22 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.454, T_{max} = 0.565
11798 measured reflections
3138 independent reflections
2543 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.3°, θ_{min} = 2.6°
h = −20→20
k = −9→8
l = −9→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.077
S = 1.04
3138 reflections
190 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 0.4505P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

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 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.256908 (14)	0.19559 (3)	0.351278 (19)	0.04528 (10)
O1	0.27754 (10)	0.3064 (2)	0.77430 (12)	0.0463 (4)
O2	0.02746 (10)	0.1315 (3)	0.74256 (12)	0.0490 (4)
H2O	0.0786	0.1768	0.7273	0.073*
O1W	0.20712 (12)	0.0602 (3)	0.88744 (12)	0.0555 (5)
H1W1	0.1801	−0.0294	0.8452	0.083*
H2W1	0.2246	0.1388	0.8402	0.083*
N1	0.61249 (13)	0.3236 (4)	0.5692 (2)	0.0675 (7)
H1N1	0.6101	0.3120	0.4985	0.081*
H2N1	0.6429	0.4174	0.6041	0.081*
N2	0.21269 (10)	0.3032 (2)	0.59806 (14)	0.0322 (4)
H2N	0.2133	0.3342	0.5294	0.039*
N3	0.13313 (11)	0.2630 (2)	0.62093 (15)	0.0321 (4)
C1	0.36621 (12)	0.3254 (3)	0.64417 (16)	0.0297 (4)
C2	0.38195 (13)	0.2378 (3)	0.55174 (17)	0.0344 (5)
H2	0.3372	0.1774	0.5059	0.041*
C3	0.46277 (15)	0.2394 (3)	0.5272 (2)	0.0425 (5)

H3	0.4721	0.1783	0.4657	0.051*
C4	0.53040 (14)	0.3309 (3)	0.5931 (2)	0.0428 (6)
C5	0.51473 (13)	0.4213 (4)	0.6841 (2)	0.0475 (6)
H5	0.5592	0.4855	0.7283	0.057*
C6	0.43430 (13)	0.4174 (3)	0.70993 (17)	0.0384 (5)
H6	0.4254	0.4771	0.7721	0.046*
C7	0.28256 (13)	0.3116 (3)	0.67866 (16)	0.0304 (4)
C8	0.06943 (13)	0.2690 (3)	0.54186 (17)	0.0321 (4)
H7	0.0781	0.3071	0.4745	0.039*
C9	−0.01679 (12)	0.2173 (3)	0.55511 (16)	0.0284 (4)
C10	−0.03425 (13)	0.1483 (3)	0.65254 (17)	0.0328 (4)
C11	−0.11712 (13)	0.0932 (3)	0.65825 (17)	0.0369 (5)
H11	−0.1282	0.0459	0.7226	0.044*
C12	−0.18330 (12)	0.1076 (3)	0.56950 (17)	0.0352 (5)
H12	−0.2389	0.0715	0.5740	0.042*
C13	−0.16619 (12)	0.1761 (3)	0.47410 (17)	0.0317 (4)
C14	−0.08416 (13)	0.2293 (3)	0.46589 (17)	0.0304 (4)
H14	−0.0737	0.2735	0.4005	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03422 (13)	0.04959 (16)	0.04628 (16)	−0.00074 (10)	−0.00607 (10)	−0.00069 (10)
O1	0.0456 (9)	0.0669 (12)	0.0280 (8)	−0.0112 (8)	0.0110 (7)	−0.0009 (7)
O2	0.0392 (8)	0.0719 (12)	0.0331 (8)	−0.0078 (8)	0.0005 (6)	0.0088 (8)
O1W	0.0755 (12)	0.0603 (12)	0.0319 (9)	−0.0168 (9)	0.0136 (8)	−0.0011 (8)
N1	0.0289 (10)	0.0796 (18)	0.0962 (19)	0.0066 (10)	0.0174 (11)	0.0289 (14)
N2	0.0246 (8)	0.0455 (11)	0.0279 (9)	−0.0050 (7)	0.0084 (7)	0.0004 (7)
N3	0.0251 (8)	0.0364 (9)	0.0369 (10)	−0.0046 (7)	0.0109 (7)	−0.0024 (7)
C1	0.0260 (9)	0.0326 (11)	0.0294 (10)	−0.0018 (8)	0.0029 (8)	0.0037 (8)
C2	0.0288 (10)	0.0368 (12)	0.0368 (12)	−0.0025 (8)	0.0047 (9)	−0.0015 (9)
C3	0.0394 (12)	0.0462 (13)	0.0447 (14)	0.0069 (10)	0.0156 (10)	0.0046 (10)
C4	0.0267 (10)	0.0430 (13)	0.0593 (15)	0.0052 (9)	0.0101 (10)	0.0219 (11)
C5	0.0277 (11)	0.0473 (14)	0.0599 (16)	−0.0101 (10)	−0.0096 (10)	0.0089 (12)
C6	0.0363 (11)	0.0407 (12)	0.0347 (12)	−0.0045 (9)	−0.0014 (9)	−0.0004 (9)
C7	0.0301 (10)	0.0335 (11)	0.0274 (10)	−0.0026 (8)	0.0049 (8)	0.0004 (8)
C8	0.0294 (10)	0.0361 (11)	0.0326 (11)	−0.0005 (8)	0.0104 (8)	−0.0012 (8)
C9	0.0259 (9)	0.0288 (10)	0.0317 (11)	−0.0011 (7)	0.0086 (8)	−0.0016 (8)
C10	0.0320 (10)	0.0372 (11)	0.0287 (10)	−0.0008 (8)	0.0051 (8)	−0.0008 (8)
C11	0.0362 (11)	0.0447 (13)	0.0327 (11)	−0.0062 (10)	0.0135 (9)	0.0005 (9)
C12	0.0267 (10)	0.0376 (12)	0.0427 (12)	−0.0050 (9)	0.0105 (8)	−0.0052 (10)
C13	0.0267 (9)	0.0319 (11)	0.0347 (11)	0.0006 (8)	0.0021 (8)	−0.0060 (8)
C14	0.0312 (10)	0.0318 (11)	0.0290 (11)	−0.0002 (8)	0.0077 (8)	−0.0003 (8)

Geometric parameters (Å, º)

Br1—C13	1.902 (2)	C3—C4	1.386 (4)
O1—C7	1.228 (2)	C3—H3	0.9300
O2—C10	1.352 (3)	C4—C5	1.384 (4)
O2—H2O	0.9276	C5—C6	1.377 (3)

O1W—H1W1	0.8893	C5—H5	0.9300
O1W—H2W1	0.9036	C6—H6	0.9300
N1—C4	1.393 (3)	C8—C9	1.457 (3)
N1—H1N1	0.8915	C8—H7	0.9300
N1—H2N1	0.8923	C9—C14	1.394 (3)
N2—C7	1.350 (3)	C9—C10	1.405 (3)
N2—N3	1.378 (2)	C10—C11	1.387 (3)
N2—H2N	0.8985	C11—C12	1.380 (3)
N3—C8	1.273 (3)	C11—H11	0.9300
C1—C6	1.392 (3)	C12—C13	1.378 (3)
C1—C2	1.392 (3)	C12—H12	0.9300
C1—C7	1.478 (3)	C13—C14	1.378 (3)
C2—C3	1.376 (3)	C14—H14	0.9300
C2—H2	0.9300		

C10—O2—H2O	108.0	C1—C6—H6	119.6
H1W1—O1W—H2W1	103.2	O1—C7—N2	122.64 (19)
C4—N1—H1N1	111.3	O1—C7—C1	121.92 (19)
C4—N1—H2N1	107.5	N2—C7—C1	115.44 (18)
H1N1—N1—H2N1	118.5	N3—C8—C9	121.11 (19)
C7—N2—N3	119.90 (17)	N3—C8—H7	119.4
C7—N2—H2N	123.7	C9—C8—H7	119.4
N3—N2—H2N	116.1	C14—C9—C10	118.67 (18)
C8—N3—N2	116.35 (17)	C14—C9—C8	118.45 (18)
C6—C1—C2	118.00 (19)	C10—C9—C8	122.82 (18)
C6—C1—C7	119.32 (19)	O2—C10—C11	117.74 (19)
C2—C1—C7	122.52 (18)	O2—C10—C9	122.36 (18)
C3—C2—C1	121.0 (2)	C11—C10—C9	119.90 (19)
C3—C2—H2	119.5	C12—C11—C10	120.8 (2)
C1—C2—H2	119.5	C12—C11—H11	119.6
C2—C3—C4	120.8 (2)	C10—C11—H11	119.6
C2—C3—H3	119.6	C13—C12—C11	119.30 (18)
C4—C3—H3	119.6	C13—C12—H12	120.3
C5—C4—C3	118.5 (2)	C11—C12—H12	120.3
C5—C4—N1	121.8 (2)	C12—C13—C14	121.08 (19)
C3—C4—N1	119.7 (3)	C12—C13—Br1	119.59 (15)
C6—C5—C4	120.9 (2)	C14—C13—Br1	119.33 (16)
C6—C5—H5	119.5	C13—C14—C9	120.29 (19)
C4—C5—H5	119.5	C13—C14—H14	119.9
C5—C6—C1	120.8 (2)	C9—C14—H14	119.9
C5—C6—H6	119.6		

C7—N2—N3—C8	175.72 (19)	N2—N3—C8—C9	175.98 (18)
C6—C1—C2—C3	1.2 (3)	N3—C8—C9—C14	179.08 (19)
C7—C1—C2—C3	−174.1 (2)	N3—C8—C9—C10	−3.7 (3)
C1—C2—C3—C4	−1.1 (3)	C14—C9—C10—O2	179.73 (19)
C2—C3—C4—C5	−0.2 (3)	C8—C9—C10—O2	2.5 (3)
C2—C3—C4—N1	177.1 (2)	C14—C9—C10—C11	0.3 (3)
C3—C4—C5—C6	1.3 (3)	C8—C9—C10—C11	−176.9 (2)

N1—C4—C5—C6	−176.0 (2)	O2—C10—C11—C12	179.6 (2)
C4—C5—C6—C1	−1.2 (4)	C9—C10—C11—C12	−0.9 (3)
C2—C1—C6—C5	−0.1 (3)	C10—C11—C12—C13	0.6 (3)
C7—C1—C6—C5	175.3 (2)	C11—C12—C13—C14	0.3 (3)
N3—N2—C7—O1	−8.8 (3)	C11—C12—C13—Br1	179.78 (16)
N3—N2—C7—C1	170.52 (17)	C12—C13—C14—C9	−0.9 (3)
C6—C1—C7—O1	−30.2 (3)	Br1—C13—C14—C9	179.60 (15)
C2—C1—C7—O1	145.0 (2)	C10—C9—C14—C13	0.6 (3)
C6—C1—C7—N2	150.5 (2)	C8—C9—C14—C13	177.93 (19)
C2—C1—C7—N2	−34.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···N3	0.93	1.84	2.660 (2)	145
N2—H2*N*···O1*W*^{i}	0.90	1.93	2.823 (2)	171
N1—H2*N*1···O1*W*^{ii}	0.89	2.57	3.276 (3)	137
O1*W*—H2*W*1···O1	0.90	1.77	2.653 (2)	167

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2.