Acta Cryst E
CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of (*E*)-*N*′-(5-bromo-2-hydroxybenzylidene)nicotinohydrazide monohydrate

S. Sravya,[a] S. Sruthy,[a] N. Aiswarya,[b] M. Sithambaresan[c]* and M. R. Prathapachandra Kurup[b]

[a]Department of Chemistry, Amrita Vishwa Vidyapeetham, Clappana PO, Kollam 690 525, India, [b]Department of Applied Chemistry, Cochin University of Science and Technology, Kochi 682 022, India, and [c]Department of Chemistry, Faculty of Science, Eastern University, Chenkalady, Sri Lanka. *Correspondence e-mail: msithambaresan@gmail.com

Received 4 May 2015
Accepted 19 May 2015

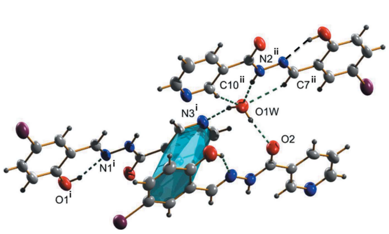
Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

Keywords: crystal structure; aroylhydrazone; carbohydrazide; nicotinohydrazide; hydrogen bonding

CCDC reference: 1401825

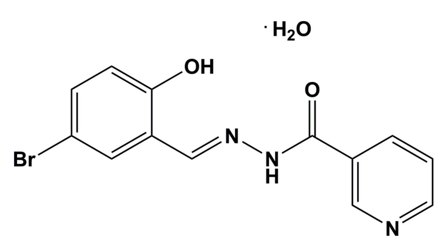
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $C_{13}H_{10}BrN_3O_2·H_2O$, the conformation about the azomethine double bond is *E*. The molecule exists in the amido form with a C=O bond length of 1.229 (2) Å. There is an intramolecular O—H···N hydrogen bond forming an *S*(6) ring motif. The whole molecule is almost planar, with an r.m.s. deviation of 0.021 Å for all non-H atoms, and the dihedral angle between the planes of the pyridine and benzene rings is 0.74 (12)°. In the crystal, the water molecule of crystallization links the organic molecules *via* Ow—H···O, Ow—H···N and N—H···Ow hydrogen bonds and short C—H···Ow contacts, forming sheets lying parallel to (100). Within the sheets there is a weak π–π interaction involving the pyridine and benzene rings [centroid-to-centroid distance = 3.8473 (15) Å]. The sheets are linked *via* C—H···Br interactions, forming a three-dimensional network.



1. Chemical context

Aroylhydrazones can coordinate to transition metals either in the amido form (Bessy Raj & Kurup, 2007) or in the iminolato form (Ghosh *et al.*, 2005; Galić *et al.*, 2011), leading to the formation of two types of complexes. Hydrazones derived from isonicotinoyl hydrazides are potential drugs for the treatment of the iron-overload associated diseases (Macková *et al.*, 2012). They are associated with a broad spectrum of biological activities, and studies have shown that nicotinic acid hydrazones could be considered as anti-inflammatory and analgesic agents (Navidpour *et al.*, 2014; Kheradmand *et al.*, 2013) and as a novel pharmacophore in the design of anti-convulsant drugs (Sinha *et al.*, 2011). Hydrazones have been used in chemical processes, in non-linear optics and as sensors as well as in catalytic processes (Hosseini-Monfared *et al.*, 2013; Du & Hong, 2014). Their potential as analytical reagents (Galić *et al.*, 2011) and their uses as molecular switches, metallo-assemblies and sensors have also been reported (Su & Aprahamian, 2014). Salicylaldehyde isonicotinoylhydrazone has also been used for the spectrophotometric determination of gallium(III) and indium(III) (Reddy *et al.*, 2011).



OPEN ACCESS

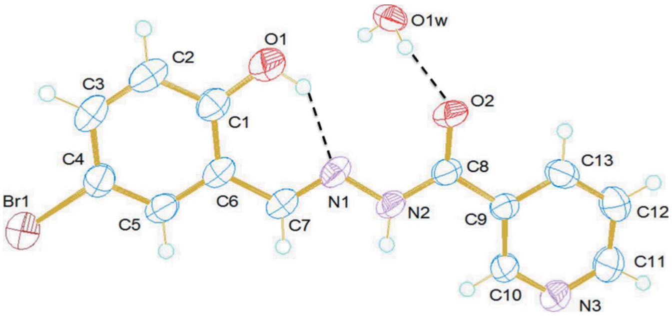


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

The title compound, Fig. 1, exists in the amido form with a C8═O2 bond length of 1.229 (2) Å. The molecule has an *E* conformation with respect to the azomethine bond, which is confirmed by the torsion angle C6—C7═N1—N2 of 179.09 (19)°. The two aromatic rings (C1–C6 and N3/C9–C13), are inclined to the almost planar hydrazone moiety [O2/C8/N2/N1/C7; planar to within 0.006 (2) Å] by 2.12 (9) and 1.40 (8)°, respectively, and to each other by 0.74 (12)°. There is an intramolecular O—H···N hydrogen bond present in the molecule that involves the phenolic oxygen, O1 and the azomethine nitrogen atom, N1, forming an *S*(6) ring motif (Table 1 and Fig. 1).

3. Supramolecular features

In the crystal, the water molecule forms three hydrogen bonds with three different nicotinic hydrazone molecules (Table 1 and Fig. 2). This compound is an example of a system where a single atom acts both as donor and acceptor. There are also C—H···O(water) contacts present enclosing $R_2^1(6)$ and $R_2^1(7)$ ring motifs (Fig. 2). Finally sheets are formed lying parallel to (100). There are weak π–π interactions within the sheets involving the bromine-bearing aromatic ring of one molecule and the pyridine ring of another, with a centroid–centroid distance of 3.8473 (15) Å (Fig. 2). The sheets are linked *via* C—H···Br interactions, forming a three-dimensional network (Table 1 and Fig. 3).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.86 (1)	1.91 (2)	2.641 (2)	143 (3)
O1*W*—H1*A*···O2	0.85 (1)	1.91 (1)	2.756 (2)	172 (3)
O1*W*—H1*B*···N3^{i}	0.85 (1)	2.03 (1)	2.845 (3)	162 (2)
N2—H2′···O1*W*^{ii}	0.87 (1)	1.95 (1)	2.806 (3)	169 (3)
C7—H7···O1*W*^{ii}	0.93	2.49	3.263 (3)	140
C10—H10···O1*W*^{ii}	0.93	2.45	3.362 (3)	165
C11—H11···Br1^{iii}	0.93	2.93	3.825 (3)	162

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

4. Database survey

A search of the Cambridge Structural Database (Version 5.36, update Feb. 2015; Groom & Allen, 2014) yielded 22 hits for the substructure *N*′-(2-hydroxybenzylidene)nicotinohydrazide. The crystal structure of *N*′-(2-hydroxybenzylidene)-nicotinohydrazide itself is reported as a monohydrate (IDASUB; Galić *et al.*, 2001), and the crystal structure of the chloro derivative of the title compound, which crystallized with two independent molecules in the asymmetric unit, has also been reported (MOZPIB; Ren, 2009). In these two

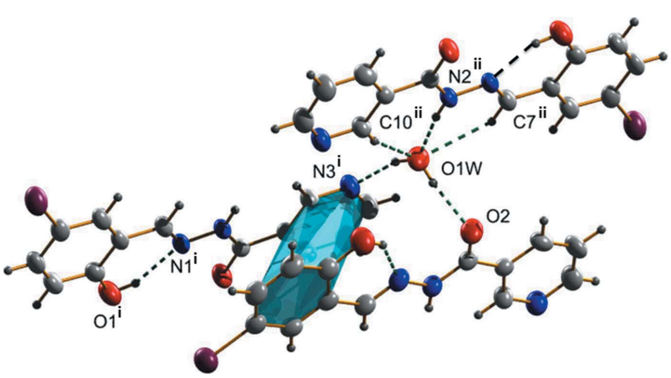


Figure 2
Hydrogen bonds (dashed lines) and a weak π–π interaction (in blue) in the crystal of the title compound [symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$].

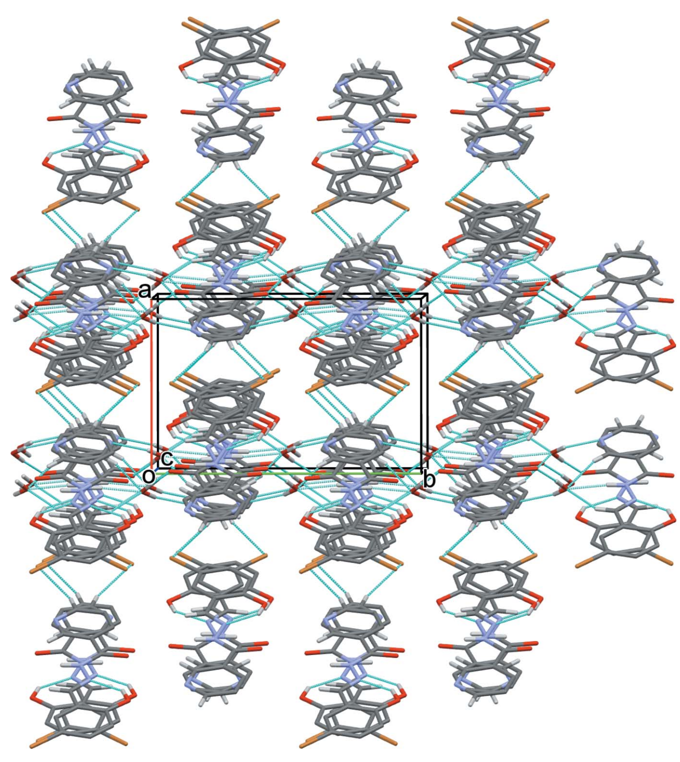


Figure 3
A view along the *c* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1 for details) and H atoms not involved in hydrogen bonding have been omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}BrN_3O_2{\cdot}H_2O$
M_r	338.17
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, *b*, *c* (Å)	8.1623 (7), 12.5953 (9), 13.2510 (8)
β (°)	90.226 (3)
V (Å^3)	1362.28 (17)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.03
Crystal size (mm)	0.42 × 0.12 × 0.11
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.349, 0.356
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7570, 3331, 2233
R_{int}	0.028
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.036, 0.102, 0.99
No. of reflections	3331
No. of parameters	198
No. of restraints	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.35

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2010), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

compounds, an intramolecular O—H···N hydrogen bond is also present. The molecules are also relatively planar, with the benzene and pyridine rings being inclined to one another by *ca* 4.2° in IDASUB, and by *ca* 12.8 and 1.9° in the two independent molecules of MOZPIB. This last dihedral angle is similar to that in the title compound [*cf.* 0.74 (12)°]. In the crystal structure of *N*′-(2-hydroxybenzylidene)nicotinohydrazide monohydrate (IDASUB), the water molecule forms three hydrogen bonds and is another example of a system where a single atom acts both as donor and acceptor.

5. Synthesis and crystallization

The title compound was prepared by adapting a reported procedure (Mathew & Kurup, 2011). A methanolic solution of 5-bromosalicylaldehyde (0.10051 g, 0.5 mmol) and nicotinic hydrazide (0.06857 g, 0.5 mmol) was refluxed for 3 h with two drops of glacial acetic acid. Light-yellow block-shaped crystals of the title compound were obtained by slow evaporation of the solvent. The crystals were filtered, washed with minimum quantity of methanol and dried over P_4O_{10} *in vacuo* (yield: 0.22 g, 68.5%; m.p.: 480 K). Elemental analysis calculated for $C_{13}H_{10}N_3O_2Br{\cdot}H_2O$: C, 46.17, H, 3.58, N, 12.43%; found: C, 46.14, H, 3.57, N, 12.44%. IR FT–IR (KBr, cm^{-1}) 3059 (NH), 3269(OH), 1680 (C=O), 1584 (C=N).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The water, hydroxyl and NH H atoms were located in difference Fourier maps and refined with distances restraints: O—H = 0.86 (1) Å and N—H = 0.88 (1) Å. All C-bound H atoms were placed in calculated positions and refined as riding: C—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(C). Three reflections were omitted owing to bad agreement, *viz*. 100, 110 and 200.

Acknowledgements

NA is grateful to the UGC, New Delhi, India, for the award of a Senior Research Fellowship. MRPK is grateful to the UGC, New Delhi, India, for a UGC–BSR one-time grant to Faculty. We also thank the Sophisticated Analytical Instruments Facility, Cochin University of S&T, Kochi-22, India, for the diffraction measurements.

References

Bessy Raj, B. N. & Kurup, M. R. P. (2007). *Spectrochim. Acta Part A*, **66**, 898–903.
Brandenburg, K. (2010). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2004). *APEX2*, *SAINT*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Du, C. & Hong, Z. (2014). *Synth. React. Inorg. Met. Org. Nano-Met. Chem.* **45**, 507–511.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Galić, N., Perić, B., Kojić-Prodić, B. & Cimerman, Z. (2001). *J. Mol. Struct.* **559**, 187–194.
Galić, N., Rubčić, M., Magdić, K., Cindrić, M. & Tomišić (2011). *Inorg. Chim. Acta*, **366**, 98–104.
Ghosh, T., Bhattacharya, S., Das, A., Mukherjee, G. & Drew, M. G. B. (2005). *Inorg. Chim. Acta*, **358**, 989–996.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Hosseini-Monfared, H., Farrokhi, A., Alavi, S. & Mayer, P. (2013). *Transition Met. Chem.* **38**, 267–273.
Kheradmand, A., Navidpour, L., Shafaroodi, H., Saeedi-Motahar, G. & Shafiee, A. (2013). *Med. Chem. Res.* **22**, 2411–2420.
Macková, E., Hrušková, K., Bendová, P., Vávrová, A., Jansová, H., Hašková, P., Kovaříková, P., Vávrová, K. & Šimůnek, T. (2012). *Chem-Biol. Interact.* **197**, 69–79.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mathew, N. & Kurup, M. R. P. (2011). *Spectrochim. Acta Part A*, **78**, 1424–1428.
Navidpour, L., Shafaroodi, H., Saeedi-Motahar, G. & Shafiee, A. (2014). *Med. Chem. Res.* **23**, 2793–2802.
Reddy, G. C., Devanna, N. & Chandrasekhar, K. B. (2011). *Orbital Elec. J. Chem.* **3**, 24–31.
Ren, C.-G. (2009). *Acta Cryst.* E**65**, o1505–o1506.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Sinha, R., Singh Sara, U. V., Khosa, R. L., Shenoi, V., Stables, J. & Jain, J. (2011). *Med. Chem. Res.* **20**, 1499–1504.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Su, X. & Aprahamian, I. (2014). *Chem. Soc. Rev.* **43**, 1963–1981.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2015). E**71**, 734-736   [doi:10.1107/S2056989015009627]

Crystal structure of (*E*)-*N*′-(5-bromo-2-hydroxybenzylidene)nicotinohydrazide monohydrate

S. Sravya, S. Sruthy, N. Aiswarya, M. Sithambaresan and M. R. Prathapachandra Kurup

Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2010) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

(*E*)-*N*′-(5-Bromo-2-hydroxybenzylidene)nicotinohydrazide monohydrate

Crystal data

$C_{13}H_{10}BrN_3O_2{\cdot}H_2O$
M_r = 338.17
Monoclinic, $P2_1/c$
a = 8.1623 (7) Å
b = 12.5953 (9) Å
c = 13.2510 (8) Å
β = 90.226 (3)°
V = 1362.28 (17) Å^3
Z = 4
F(000) = 680
D_x = 1.649 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2267 reflections
θ = 2.9–25.5°
μ = 3.03 mm^{-1}
T = 296 K
Needle, yellow
0.42 × 0.12 × 0.11 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.349, T_{max} = 0.356
7570 measured reflections
3331 independent reflections
2233 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 28.3°, θ_{min} = 3.0°
$h = -10 \rightarrow 4$
$k = -16 \rightarrow 16$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 0.99
3331 reflections
198 parameters
5 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0129 (13)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3276 (3)	0.35310 (19)	0.45911 (17)	0.0442 (6)
C2	0.4115 (4)	0.3763 (2)	0.54807 (18)	0.0527 (7)
H2	0.4272	0.4467	0.5667	0.063*
C3	0.4712 (3)	0.2969 (2)	0.60856 (16)	0.0499 (6)
H3	0.5276	0.3134	0.6676	0.060*
C4	0.4471 (3)	0.19134 (19)	0.58114 (15)	0.0443 (6)
C5	0.3653 (3)	0.16695 (19)	0.49313 (15)	0.0441 (6)
H5	0.3499	0.0962	0.4753	0.053*
C6	0.3057 (3)	0.24637 (18)	0.43065 (15)	0.0401 (5)
C7	0.2191 (3)	0.21655 (19)	0.33854 (15)	0.0431 (6)
H7	0.2084	0.1452	0.3219	0.052*
C8	0.0128 (3)	0.32662 (17)	0.13365 (15)	0.0385 (5)
C9	−0.0754 (3)	0.28892 (17)	0.04153 (15)	0.0358 (5)
C10	−0.0990 (3)	0.18419 (18)	0.01526 (15)	0.0450 (6)
H10	−0.0591	0.1321	0.0586	0.054*
C11	−0.2332 (4)	0.2286 (2)	−0.12925 (19)	0.0560 (7)
H11	−0.2880	0.2083	−0.1878	0.067*
C12	−0.2160 (3)	0.3349 (2)	−0.10981 (18)	0.0580 (7)
H12	−0.2581	0.3852	−0.1542	0.070*
C13	−0.1355 (3)	0.36555 (19)	−0.02368 (18)	0.0487 (6)
H13	−0.1214	0.4372	−0.0092	0.058*
N1	0.1580 (2)	0.28694 (15)	0.28050 (12)	0.0426 (5)
N2	0.0760 (3)	0.25244 (15)	0.19557 (13)	0.0403 (4)
N3	−0.1758 (3)	0.15309 (16)	−0.06888 (13)	0.0524 (6)
O1	0.2713 (3)	0.43501 (14)	0.40347 (14)	0.0634 (6)
O2	0.0235 (3)	0.42215 (12)	0.15145 (12)	0.0586 (6)
O1W	−0.0975 (3)	0.53043 (13)	0.31578 (13)	0.0641 (6)
Br1	0.52655 (4)	0.08150 (2)	0.66553 (2)	0.06576 (15)
H1	0.218 (4)	0.412 (2)	0.3516 (16)	0.079 (11)*
H2′	0.075 (3)	0.1842 (9)	0.1847 (18)	0.060 (8)*
H1A	−0.053 (4)	0.4947 (19)	0.2684 (15)	0.087 (11)*
H1B	−0.129 (3)	0.4858 (16)	0.3591 (15)	0.065 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0439 (15)	0.0460 (13)	0.0426 (11)	−0.0079 (11)	0.0002 (10)	−0.0046 (10)

C2	0.0600 (18)	0.0505 (14)	0.0477 (13)	−0.0140 (13)	0.0000 (12)	−0.0154 (11)
C3	0.0470 (16)	0.0603 (15)	0.0423 (12)	−0.0117 (12)	−0.0076 (11)	−0.0122 (11)
C4	0.0385 (14)	0.0545 (14)	0.0399 (11)	0.0016 (11)	−0.0018 (10)	−0.0070 (10)
C5	0.0429 (15)	0.0446 (13)	0.0446 (12)	−0.0009 (11)	−0.0008 (10)	−0.0131 (10)
C6	0.0364 (13)	0.0462 (13)	0.0378 (11)	−0.0056 (10)	0.0010 (9)	−0.0113 (9)
C7	0.0467 (15)	0.0435 (13)	0.0391 (11)	−0.0050 (11)	−0.0024 (10)	−0.0104 (10)
C8	0.0475 (15)	0.0335 (12)	0.0346 (10)	−0.0053 (10)	0.0043 (9)	−0.0044 (8)
C9	0.0387 (13)	0.0339 (11)	0.0349 (10)	−0.0006 (9)	0.0038 (9)	−0.0037 (8)
C10	0.0599 (17)	0.0366 (12)	0.0384 (11)	−0.0042 (11)	−0.0085 (11)	−0.0012 (9)
C11	0.0588 (18)	0.0624 (17)	0.0469 (13)	−0.0008 (13)	−0.0150 (12)	−0.0026 (11)
C12	0.0626 (19)	0.0557 (17)	0.0556 (14)	0.0138 (14)	−0.0201 (13)	0.0040 (11)
C13	0.0533 (17)	0.0363 (13)	0.0564 (14)	0.0067 (11)	−0.0034 (12)	0.0000 (10)
N1	0.0479 (13)	0.0449 (11)	0.0348 (9)	−0.0106 (9)	−0.0022 (8)	−0.0090 (8)
N2	0.0510 (13)	0.0350 (11)	0.0348 (9)	−0.0064 (9)	−0.0039 (8)	−0.0069 (7)
N3	0.0667 (16)	0.0464 (12)	0.0439 (11)	−0.0042 (11)	−0.0110 (10)	−0.0066 (9)
O1	0.0871 (16)	0.0443 (11)	0.0587 (11)	−0.0099 (9)	−0.0178 (11)	−0.0052 (8)
O2	0.0956 (17)	0.0328 (9)	0.0472 (9)	−0.0063 (8)	−0.0065 (10)	−0.0094 (6)
O1W	0.1112 (19)	0.0316 (9)	0.0494 (10)	0.0065 (10)	0.0025 (11)	0.0023 (8)
Br1	0.0773 (3)	0.0634 (2)	0.0564 (2)	0.00894 (15)	−0.01868 (14)	−0.00546 (12)

Geometric parameters (Å, º)

C1—O1	1.348 (3)	C8—C9	1.492 (3)
C1—C2	1.392 (3)	C9—C10	1.378 (3)
C1—C6	1.407 (3)	C9—C13	1.384 (3)
C2—C3	1.370 (4)	C10—N3	1.336 (3)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.392 (3)	C11—N3	1.327 (3)
C3—H3	0.9300	C11—C12	1.370 (4)
C4—C5	1.376 (3)	C11—H11	0.9300
C4—Br1	1.892 (2)	C12—C13	1.370 (3)
C5—C6	1.386 (3)	C12—H12	0.9300
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.457 (3)	N1—N2	1.378 (2)
C7—N1	1.274 (3)	N2—H2′	0.872 (10)
C7—H7	0.9300	O1—H1	0.858 (10)
C8—O2	1.229 (2)	O1W—H1A	0.854 (10)
C8—N2	1.345 (3)	O1W—H1B	0.845 (9)
O1—C1—C2	117.9 (2)	N2—C8—C9	117.42 (18)
O1—C1—C6	122.8 (2)	C10—C9—C13	117.5 (2)
C2—C1—C6	119.3 (2)	C10—C9—C8	125.3 (2)
C3—C2—C1	121.0 (2)	C13—C9—C8	117.22 (19)
C3—C2—H2	119.5	N3—C10—C9	123.8 (2)
C1—C2—H2	119.5	N3—C10—H10	118.1
C2—C3—C4	119.6 (2)	C9—C10—H10	118.1
C2—C3—H3	120.2	N3—C11—C12	123.4 (2)
C4—C3—H3	120.2	N3—C11—H11	118.3

C5—C4—C3	120.1 (2)	C12—C11—H11	118.3
C5—C4—Br1	120.11 (18)	C13—C12—C11	118.7 (2)
C3—C4—Br1	119.75 (17)	C13—C12—H12	120.6
C4—C5—C6	120.9 (2)	C11—C12—H12	120.6
C4—C5—H5	119.6	C12—C13—C9	119.4 (2)
C6—C5—H5	119.6	C12—C13—H13	120.3
C5—C6—C1	119.1 (2)	C9—C13—H13	120.3
C5—C6—C7	118.8 (2)	C7—N1—N2	117.49 (18)
C1—C6—C7	122.1 (2)	C8—N2—N1	117.59 (18)
N1—C7—C6	120.9 (2)	C8—N2—H2′	125.4 (19)
N1—C7—H7	119.5	N1—N2—H2′	116.9 (19)
C6—C7—H7	119.5	C11—N3—C10	117.2 (2)
O2—C8—N2	122.4 (2)	C1—O1—H1	111 (2)
O2—C8—C9	120.1 (2)	H1A—O1W—H1B	106 (2)

O1—C1—C2—C3	179.6 (2)	N2—C8—C9—C10	0.7 (3)
C6—C1—C2—C3	−0.7 (4)	O2—C8—C9—C13	3.2 (3)
C1—C2—C3—C4	−0.4 (4)	N2—C8—C9—C13	−177.9 (2)
C2—C3—C4—C5	0.8 (4)	C13—C9—C10—N3	0.1 (4)
C2—C3—C4—Br1	−179.05 (19)	C8—C9—C10—N3	−178.6 (2)
C3—C4—C5—C6	−0.1 (4)	N3—C11—C12—C13	0.0 (5)
Br1—C4—C5—C6	179.72 (18)	C11—C12—C13—C9	−0.6 (4)
C4—C5—C6—C1	−1.0 (3)	C10—C9—C13—C12	0.6 (4)
C4—C5—C6—C7	−179.7 (2)	C8—C9—C13—C12	179.4 (2)
O1—C1—C6—C5	−179.0 (2)	C6—C7—N1—N2	−179.09 (19)
C2—C1—C6—C5	1.4 (4)	O2—C8—N2—N1	−1.4 (3)
O1—C1—C6—C7	−0.3 (4)	C9—C8—N2—N1	179.77 (18)
C2—C1—C6—C7	−180.0 (2)	C7—N1—N2—C8	−179.3 (2)
C5—C6—C7—N1	177.9 (2)	C12—C11—N3—C10	0.7 (4)
C1—C6—C7—N1	−0.8 (3)	C9—C10—N3—C11	−0.7 (4)
O2—C8—C9—C10	−178.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.86 (1)	1.91 (2)	2.641 (2)	143 (3)
O1*W*—H1*A*···O2	0.85 (1)	1.91 (1)	2.756 (2)	172 (3)
O1*W*—H1*B*···N3^{i}	0.85 (1)	2.03 (1)	2.845 (3)	162 (2)
N2—H2′···O1*W*^{ii}	0.87 (1)	1.95 (1)	2.806 (3)	169 (3)
C7—H7···O1*W*^{ii}	0.93	2.49	3.263 (3)	140
C10—H10···O1*W*^{ii}	0.93	2.45	3.362 (3)	165
C11—H11···Br1^{iii}	0.93	2.93	3.825 (3)	162

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