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## Structure Reports

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# 1,3-Bis(4-bromophenyl)imidazolium chloride dihydrate

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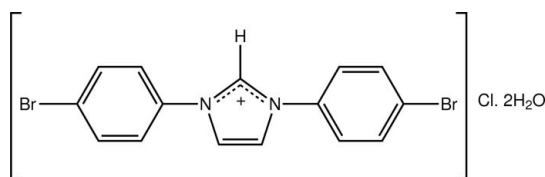
Received 18 May 2010; accepted 19 May 2010

Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.075; data-to-parameter ratio = 17.5.

In the title hydrated salt,  $\text{C}_{15}\text{H}_{11}\text{Br}_2\text{N}_2^+\cdot\text{Cl}^-\cdot 2\text{H}_2\text{O}$ , the complete imidazolium cation is generated by a crystallographic twofold axis, with one C atom lying on the axis. The chloride ion and both water molecules of crystallization also lie on a crystallographic twofold axis of symmetry. The cation is non-planar, the dihedral angle formed between the central imidazolium and benzene rings being  $12.9(3)^\circ$ ; the dihedral angle between the symmetry-related benzene rings is  $25.60(13)^\circ$ . In the crystal,  $\text{O}-\text{H}\cdots\text{Cl}$  hydrogen bonds result in supramolecular chains along  $c$  mediated by eight-membered  $\{\cdots\text{HOH}\cdots\text{Cl}\}_2$  synthons. These are consolidated by  $\text{C}-\text{H}\cdots\text{O}$  and  $\pi-\pi$  [centroid-centroid distance =  $3.687(3)$  Å] interactions.

## Related literature

For the preparation of imidazolylidene carbenes, see: Nolan (2006); Diez-Gonzalez & Nolan (2007); Glorius (2007); Leuthaeusser *et al.* (2007); Alcarazo *et al.* (2010). For related structures, see: Luger & Ruban (1975); Cole & Junk (2004); Wan *et al.* (2008).



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## Experimental

### Crystal data

$\text{C}_{15}\text{H}_{11}\text{Br}_2\text{N}_2^+\cdot\text{Cl}^-\cdot 2\text{H}_2\text{O}$   
 $M_r = 450.56$   
 Tetragonal,  $P4_22_12$   
 $a = 17.8377(7)$  Å  
 $c = 5.1270(1)$  Å  
 $V = 1631.33(10)$  Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 5.14$  mm<sup>-1</sup>  
 $T = 120$  K  
 $0.40 \times 0.03 \times 0.02$  mm

### Data collection

Nonius KappaCCD diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 2007)  
 $T_{\text{min}} = 0.665$ ,  $T_{\text{max}} = 1.000$

13675 measured reflections  
 1885 independent reflections  
 1654 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.075$   
 $S = 1.06$   
 1885 reflections  
 108 parameters  
 2 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.40$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.69$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 742 Friedel pairs  
 Flack parameter: 0.01 (2)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1o}\cdots\text{Cl1}^i$	0.84 (6)	2.28 (6)	3.1116 (19)	170 (8)
$\text{O2}-\text{H2o}\cdots\text{Cl1}$	0.87 (6)	2.40 (6)	3.211 (3)	157 (7)
$\text{C1}-\text{H1}\cdots\text{O1}$	0.95	2.09	3.042 (5)	180
$\text{C2}-\text{H2}\cdots\text{O2}^{ii}$	0.95	2.40	3.302 (7)	159

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5456).

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## supporting information

*Acta Cryst.* (2010). E66, o1438–o1439 [https://doi.org/10.1107/S1600536810018581]

**1,3-Bis(4-bromophenyl)imidazolium chloride dihydrate**

**Simon J. Garden, Paola E. Gama, Edward R. T. Tiekink, James L. Wardell, Solange M. S. V. Wardell and R. Alan Howie**

**S1. Comment**

The deprotonation of *N,N*-disubstituted imidazolium salts has been extensively used to generate imidazolylidene carbenes for use as ligands for metals or their salts in homogeneous catalysis (Nolan, 2006; Glorius, 2007). The structural motif can be readily varied so as to modify the electronic properties of the carbene and their complexes (Alcarazo *et al.*, 2010; Leuthaeusser *et al.*, 2007; Diez-Gonzalez & Nolan, 2007). Whereas structural studies are available for a number of derivatives (Luger & Ruban, 1975; Cole & Junk, 2004; Wan *et al.*, 2008), little is known about simple 1,3-diphenyl derivatives that do not possess substituents in the 2,6-positions of the phenyl rings. As part of a study into structural effects of these carbenes, we have been able to prepare and crystallize for the first time the salt 1,3-di-(4-bromophenyl)-imidazolium chloride, isolated as a dihydrate, (I).

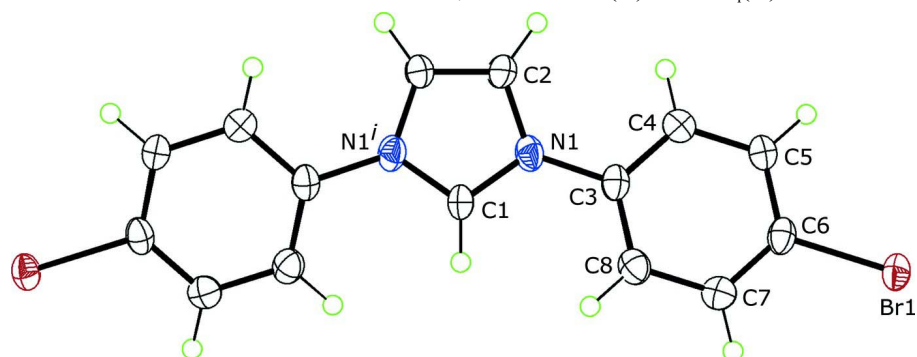
The crystallographic asymmetric unit of (I) comprises half a 1,3-di-(4-bromophenyl)imidazolium cation, Fig. 1, half a chloride, and two half water molecules, as each of the aforementioned species lies on a two-fold axis of symmetry. The cation is non-planar with the dihedral angle formed between the central imidazolium ring [r.m.s. deviation = 0.005 Å] and the benzene ring (C3–C8) being 12.9 (3) °; the dihedral angle formed between the symmetry related benzene rings is 25.60 (13) °. The twists between the rings allows for the close approach of a water molecule allowing the formation of a C1—H···O1 interaction, Table 1. This O1-water molecule also forms O—H···Cl interactions with the chloride which in turn is connected to the second water molecule leading to eight-membered {···HOH···Cl}<sub>2</sub> synthons aligned along the *c* axis, Fig. 2 and Table 1. The three-dimensional packing is consolidated by further C—H···O2 interactions, Fig. 3, as well as  $\pi$ – $\pi$  contacts (along *c*) between the imidazolium and between rings [ring centroid···ring centroid distance = 3.687 (3) Å, angle of inclination = 12.9 (3) ° for *i*: *x*, *y*, 1+*z*].

**S2. Experimental**

*p*-Bromoaniline (50 mmol) was solubilised in AcOH/H<sub>2</sub>O (3:1 V/V, 40 ml). Aqueous formaldehyde (37%, 2 ml) was added to the solution resulting in the precipitation of a solid. Following this, aqueous glyoxal (40%, 3 ml) was added and the reaction mixture was subsequently warmed (333 K) for 30 minutes. Finally, aqueous HCl (3M, 10 ml) was added resulting in the formation of a homogeneous solution. Heating was continued for a further 30 min. The crude product was precipitated from the reaction by diluting with water. The solid was isolated by filtration and allowed to air dry. The product was recrystallized from 2-propanol to generate colourless needles of (I). Melting point 581–583 K; 83% yield. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>/CDCl<sub>3</sub>):  $\delta$  10.48 [1H, s]; 8.55 [2H, s]; 7.90 [4H, s] p.p.m. <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>/CDCl<sub>3</sub>):  $\delta$  122.1; 123.2; 124.3; 133.2; 134.0; 135.0 p.p.m. IR (cm<sup>-1</sup>): 3365, 3092, 3048, 1556, 1488, 1309, 1259, 1075, 1008, 824.

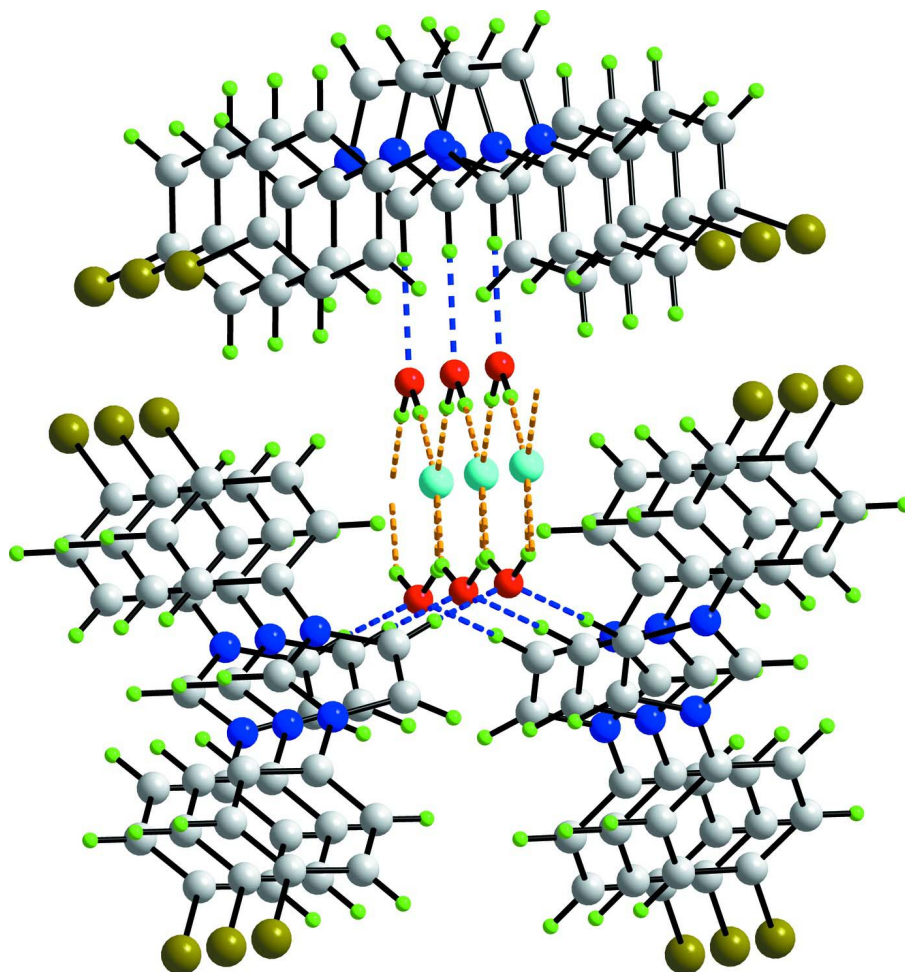
### S3. Refinement

The C-bound H atoms were geometrically placed ( $C-H = 0.95 \text{ \AA}$ ) and refined as riding with  $U_{iso}(H) = 1.2U_{eq}(C)$ . The water-bound H atoms were refined with  $O-H = 0.84 \pm 0.01 \text{ \AA}$ , and with  $U_{iso}(H) = 1.5U_{eq}(O)$ .



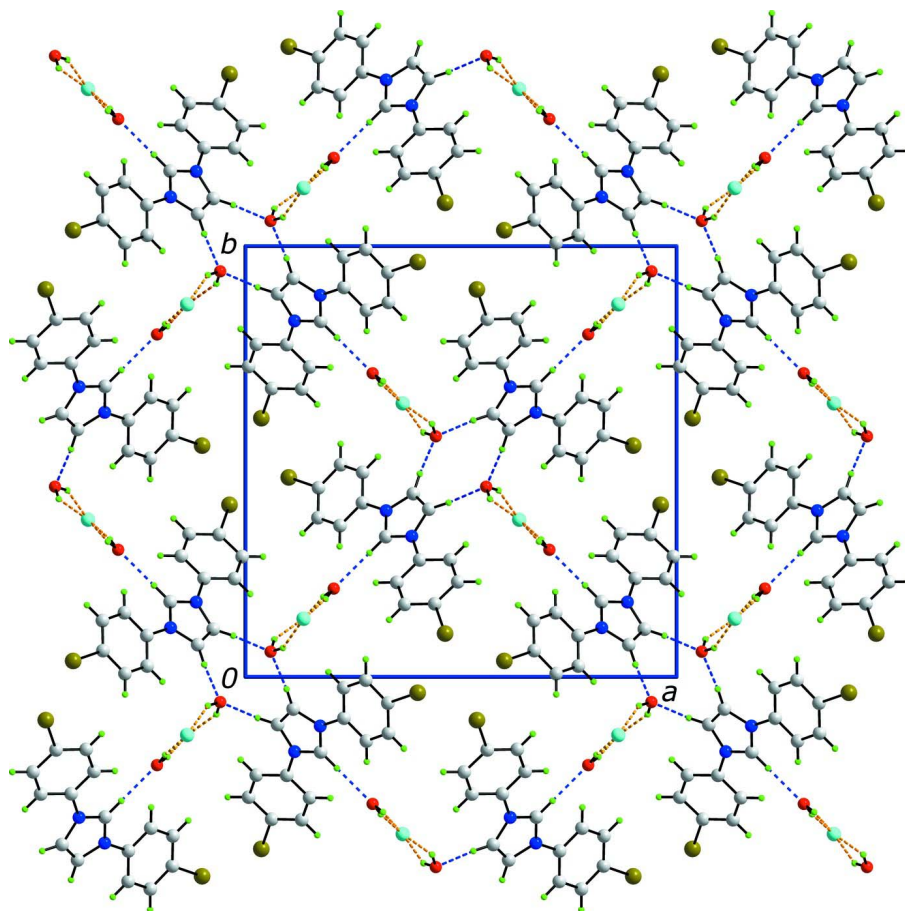
**Figure 1**

The molecular structure of the cation in (I) showing displacement ellipsoids at the 50% probability level. The C1 atom lies on a two-fold axis. Symmetry operation *i*:  $y, x, 2-z$ .



**Figure 2**

A view highlighting the eight-membered  $\{\cdots\text{HOH}\cdots\text{Cl}\}_2$  synthons aligned along the *c* axis in (I). The O–H $\cdots$ O hydrogen bonding and C–H $\cdots$ O interactions are shown as orange and blue dashed lines, respectively. Colour code: Br, olive; O, red; N, blue; C, grey; and H, green.



**Figure 3**

A view in projection down the  $c$  axis of the crystal packing in (I). The O–H $\cdots$ O hydrogen bonding and C–H $\cdots$ O interactions are shown as orange and blue dashed lines, respectively. Colour code: Br, olive; O, red; N, blue; C, grey; and H, green.

### 1,3-Bis(4-bromophenyl)imidazolium chloride dihydrate

#### Crystal data

$C_{15}H_{11}Br_2N_2^+ \cdot Cl^- \cdot 2H_2O$

$M_r = 450.56$

Tetragonal,  $P4_22_12$

Hall symbol:  $P\ 4n\ 2n$

$a = 17.8377\ (7)\ \text{\AA}$

$c = 5.1270\ (1)\ \text{\AA}$

$V = 1631.33\ (10)\ \text{\AA}^3$

$Z = 4$

$F(000) = 888$

$D_x = 1.835\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2084 reflections

$\theta = 2.9\text{--}27.5^\circ$

$\mu = 5.14\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Needle, colourless

$0.40 \times 0.03 \times 0.02\ \text{mm}$

#### Data collection

Nonius KappaCCD  
diffractometer

Radiation source: Enraf Nonius FR591 rotating  
anode

10 cm confocal mirrors monochromator

Detector resolution:  $9.091\ \text{pixels mm}^{-1}$

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 2007)

$T_{\min} = 0.665$ ,  $T_{\max} = 1.000$

13675 measured reflections  
 1885 independent reflections  
 1654 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$

$\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 3.2^\circ$   
 $h = -23 \rightarrow 23$   
 $k = -14 \rightarrow 23$   
 $l = -6 \rightarrow 6$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.075$   
 $S = 1.06$   
 1885 reflections  
 108 parameters  
 2 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 2.7306P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.40 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.69 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 742 Friedel  
 pairs  
 Absolute structure parameter: 0.01 (2)

*Special details*

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.100493 (18)	0.463088 (18)	0.00124 (12)	0.02761 (11)
Cl1	0.13502 (7)	0.13502 (7)	0.5000	0.0552 (4)
O1	0.20493 (17)	0.20493 (17)	1.0000	0.0681 (15)
H1O	0.186 (5)	0.181 (5)	1.125 (8)	0.102*
O2	0.0584 (3)	0.0584 (3)	1.0000	0.0794 (16)
H2O	0.067 (5)	0.087 (4)	0.867 (10)	0.119*
N1	0.32639 (19)	0.38584 (18)	0.8472 (7)	0.0274 (7)
C1	0.32553 (19)	0.32553 (19)	1.0000	0.0270 (9)
H1	0.2879	0.2879	1.0000	0.032*
C2	0.3893 (3)	0.4253 (3)	0.9090 (14)	0.083 (3)
H2	0.4044	0.4714	0.8322	0.100*
C3	0.2714 (2)	0.4041 (2)	0.6525 (7)	0.0252 (8)
C4	0.2720 (2)	0.4748 (2)	0.5411 (11)	0.0382 (13)
H4	0.3075	0.5111	0.5974	0.046*
C5	0.2207 (3)	0.4925 (3)	0.3464 (9)	0.0369 (10)
H5	0.2209	0.5408	0.2685	0.044*
C6	0.1696 (2)	0.4391 (2)	0.2680 (7)	0.0267 (9)
C7	0.1683 (2)	0.3694 (2)	0.3799 (8)	0.0265 (9)
H7	0.1325	0.3334	0.3241	0.032*

C8	0.2194 (2)	0.3512 (2)	0.5749 (8)	0.0289 (10)
H8	0.2185	0.3030	0.6536	0.035*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.02725 (18)	0.0350 (2)	0.02061 (17)	0.00424 (13)	-0.0019 (3)	0.0025 (3)
Cl1	0.0652 (6)	0.0652 (6)	0.0352 (8)	-0.0031 (8)	0.0037 (10)	-0.0037 (10)
O1	0.079 (2)	0.079 (2)	0.046 (3)	-0.049 (3)	-0.009 (4)	0.009 (4)
O2	0.080 (2)	0.080 (2)	0.078 (4)	0.008 (3)	0.018 (4)	-0.018 (4)
N1	0.0252 (17)	0.0274 (18)	0.0296 (19)	0.0075 (14)	-0.0003 (15)	0.0059 (15)
C1	0.0298 (14)	0.0298 (14)	0.021 (2)	0.0017 (19)	-0.005 (3)	0.005 (3)
C2	0.056 (3)	0.061 (3)	0.132 (7)	-0.030 (3)	-0.064 (4)	0.064 (4)
C3	0.0245 (19)	0.030 (2)	0.021 (2)	0.0066 (16)	0.0005 (15)	0.0017 (16)
C4	0.038 (2)	0.033 (2)	0.044 (4)	-0.0073 (16)	-0.011 (2)	0.011 (2)
C5	0.038 (2)	0.033 (2)	0.039 (3)	-0.0040 (19)	-0.013 (2)	0.014 (2)
C6	0.0221 (19)	0.036 (2)	0.0219 (19)	0.0067 (16)	0.0002 (15)	0.0025 (16)
C7	0.0219 (19)	0.027 (2)	0.030 (2)	0.0039 (16)	0.0048 (17)	-0.0021 (17)
C8	0.026 (2)	0.029 (2)	0.032 (3)	0.0052 (16)	0.0026 (15)	0.0037 (15)

*Geometric parameters (Å, °)*

Br1—C6	1.890 (4)	C3—C8	1.383 (6)
O1—H1O	0.84 (6)	C3—C4	1.383 (6)
O2—H2O	0.87 (6)	C4—C5	1.391 (6)
N1—C1	1.331 (4)	C4—H4	0.9500
N1—C2	1.362 (6)	C5—C6	1.378 (6)
N1—C3	1.437 (5)	C5—H5	0.9500
C1—N1 <sup>i</sup>	1.331 (4)	C6—C7	1.370 (6)
C1—H1	0.9500	C7—C8	1.391 (6)
C2—C2 <sup>i</sup>	1.303 (10)	C7—H7	0.9500
C2—H2	0.9500	C8—H8	0.9500
C1—N1—C2	106.9 (4)	C5—C4—H4	120.1
C1—N1—C3	125.8 (4)	C6—C5—C4	119.2 (4)
C2—N1—C3	127.3 (4)	C6—C5—H5	120.4
N1—C1—N1 <sup>i</sup>	109.1 (5)	C4—C5—H5	120.4
N1—C1—H1	125.4	C7—C6—C5	121.1 (4)
N1 <sup>i</sup> —C1—H1	125.4	C7—C6—Br1	119.8 (3)
C2 <sup>i</sup> —C2—N1	108.5 (3)	C5—C6—Br1	119.1 (3)
C2 <sup>i</sup> —C2—H2	125.7	C6—C7—C8	120.2 (4)
N1—C2—H2	125.7	C6—C7—H7	119.9
C8—C3—C4	120.6 (4)	C8—C7—H7	119.9
C8—C3—N1	120.2 (4)	C3—C8—C7	119.1 (4)
C4—C3—N1	119.2 (4)	C3—C8—H8	120.5
C3—C4—C5	119.9 (4)	C7—C8—H8	120.5
C3—C4—H4	120.1		



C2—N1—C1—N1 <sup>i</sup>	-0.2 (4)	N1—C3—C4—C5	177.9 (4)
C3—N1—C1—N1 <sup>i</sup>	178.9 (4)	C3—C4—C5—C6	0.2 (7)
C1—N1—C2—C2 <sup>i</sup>	0.7 (10)	C4—C5—C6—C7	0.5 (7)
C3—N1—C2—C2 <sup>i</sup>	-178.4 (6)	C4—C5—C6—Br1	-179.4 (4)
C1—N1—C3—C8	-12.5 (5)	C5—C6—C7—C8	-0.4 (6)
C2—N1—C3—C8	166.4 (5)	Br1—C6—C7—C8	179.5 (3)
C1—N1—C3—C4	168.5 (4)	C4—C3—C8—C7	1.0 (6)
C2—N1—C3—C4	-12.5 (7)	N1—C3—C8—C7	-177.9 (3)
C8—C3—C4—C5	-1.0 (7)	C6—C7—C8—C3	-0.3 (6)

Symmetry code: (i)  $y, x, -z+2$ .

#### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 <sub>o</sub> $\cdots$ C11 <sup>ii</sup>	0.84 (6)	2.28 (6)	3.1116 (19)	170 (8)
O2—H2 <sub>o</sub> $\cdots$ C11	0.87 (6)	2.40 (6)	3.211 (3)	157 (7)
C1—H1 $\cdots$ O1	0.95	2.09	3.042 (5)	180
C2—H2 $\cdots$ O2 <sup>iii</sup>	0.95	2.40	3.302 (7)	159

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