

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-(2-Hydroxybenzyl)adamantan-1-aminium bromide

Tao Rong

Ordered Matter Science Research Center, Southeast University, Nanjing 210096, People's Republic of China

Correspondence e-mail: rongtao198806@163.com

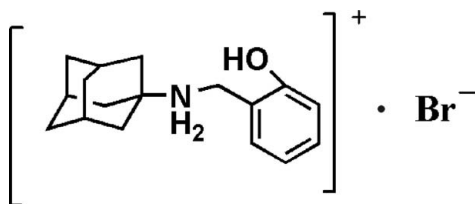
Received 20 April 2011; accepted 5 July 2011

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.045; wR factor = 0.108; data-to-parameter ratio = 19.4.

There are two independent ion pairs in the asymmetric unit of the title compound, $\text{C}_{17}\text{H}_{24}\text{NO}^+\cdot\text{Br}^-$. In the crystal, the ions are linked by intermolecular $\text{N}-\text{H}\cdots\text{Br}$ and $\text{O}-\text{H}\cdots\text{Br}$ hydrogen bonds.

Related literature

The title compound was studied as part of our work to obtain potential ferroelectric phase-change materials. For general background to ferroelectric organic frameworks, see: Fu *et al.* (2009); Ye *et al.* (2006); Zhang *et al.* (2008, 2010). For a related structure of the adamantyl ring, see: Cheng *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{17}\text{H}_{24}\text{NO}^+\cdot\text{Br}^-$
 $M_r = 338.28$

 Triclinic, $P\bar{1}$
 $a = 10.616$ (2) Å

 $b = 12.627$ (3) Å

 $c = 12.896$ (3) Å

 $\alpha = 108.46$ (3)°

 $\beta = 104.69$ (3)°

 $\gamma = 93.88$ (3)°

 $V = 1565.4$ (7) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 2.62$ mm⁻¹
 $T = 293$ K

 $0.20 \times 0.20 \times 0.20$ mm

Data collection

Rigaku SCXmini diffractometer

Absorption correction: multi-scan

 (*CrystalClear*; Rigaku, 2005)

 $T_{\min} = 0.596$, $T_{\max} = 0.598$

16356 measured reflections

7165 independent reflections

 5368 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.108$
 $S = 1.05$

7165 reflections

369 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.55$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2O}\cdots\text{Br1}^{\text{i}}$	0.82 (4)	2.45 (4)	3.255 (2)	168 (4)
$\text{N1}-\text{H1A}\cdots\text{Br2}^{\text{ii}}$	0.90	2.69	3.527 (3)	155
$\text{N1}-\text{H1B}\cdots\text{Br2}^{\text{iii}}$	0.90	2.45	3.337 (2)	167
$\text{N2}-\text{H2A}\cdots\text{Br1}^{\text{iv}}$	0.90	2.40	3.297 (2)	176
$\text{N2}-\text{H2B}\cdots\text{Br2}^{\text{iv}}$	0.90	2.50	3.377 (2)	165

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the starter fund of Southeast University for financial support to buy the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2188).

References

- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Cheng, L., Xu, X. & Xu, Y. (2008). *Acta Cryst.* **E64**, m82.
- Fu, D. W., Ge, J. Z., Dai, J., Ye, H. Y. & Qu, Z. R. (2009). *Inorg. Chem. Commun.* **12**, 994–997.
- Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Ye, Q., Song, Y. M., Wang, G. X., Chen, K. & Fu, D. W. (2006). *J. Am. Chem. Soc.* **128**, 6554–6555.
- Zhang, W., Xiong, R. G. & Huang, S. P. D. (2008). *J. Am. Chem. Soc.* **130**, 10468–10469.
- Zhang, W., Ye, H. Y., Cai, H. L., Ge, J. Z. & Xiong, R. G. (2010). *J. Am. Chem. Soc.* **132**, 7300–7302.

supporting information

Acta Cryst. (2011). E67, o1979 [doi:10.1107/S1600536811026742]

N*-(2-Hydroxybenzyl)adamantan-1-aminium bromide*Tao Rong****S1. Comment**

The study of ferroelectric materials has received much attention and some materials have predominantly dielectric-ferroelectric performance (Fu *et al.*, 2009; Ye *et al.*, 2006; Zhang *et al.*, 2008, 2010), As a part of our work to obtain potential ferroelectric phase-change materials, we report herein on the crystal structure of title compound. Unluckily, the title compound has no dielectric anomalies in the temperature range 93–53 K, suggesting that it might be only a paraelectric.

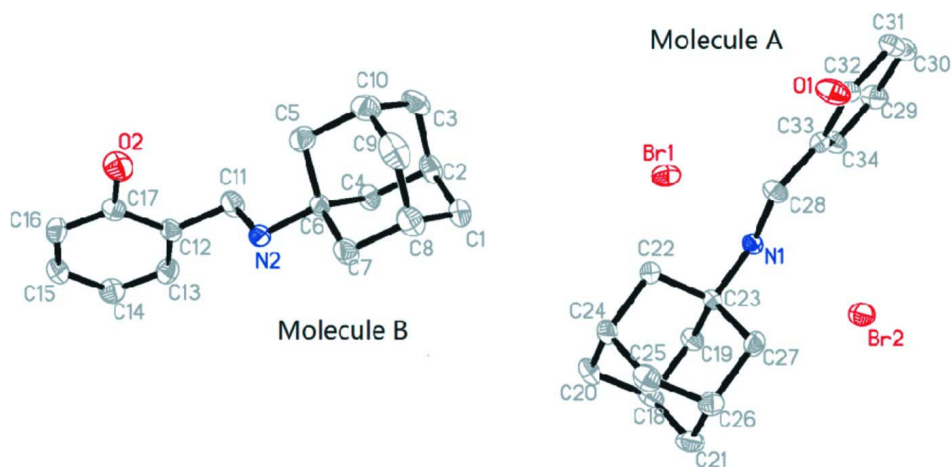
The asymmetric unit of the title compound is shown in Fig. 1. There are two independent molecules [labelled A & B]. The crystal packing (Fig. 2) is stabilized by weak intermolecular N—H \cdots Br and O—H \cdots Br hydrogen bonds between the *N*-(2-hydroxybenzyl)-1-adamantylammonium cations (Cheng *et al.* 2008) and bromide anions (see; Table 1).

S2. Experimental

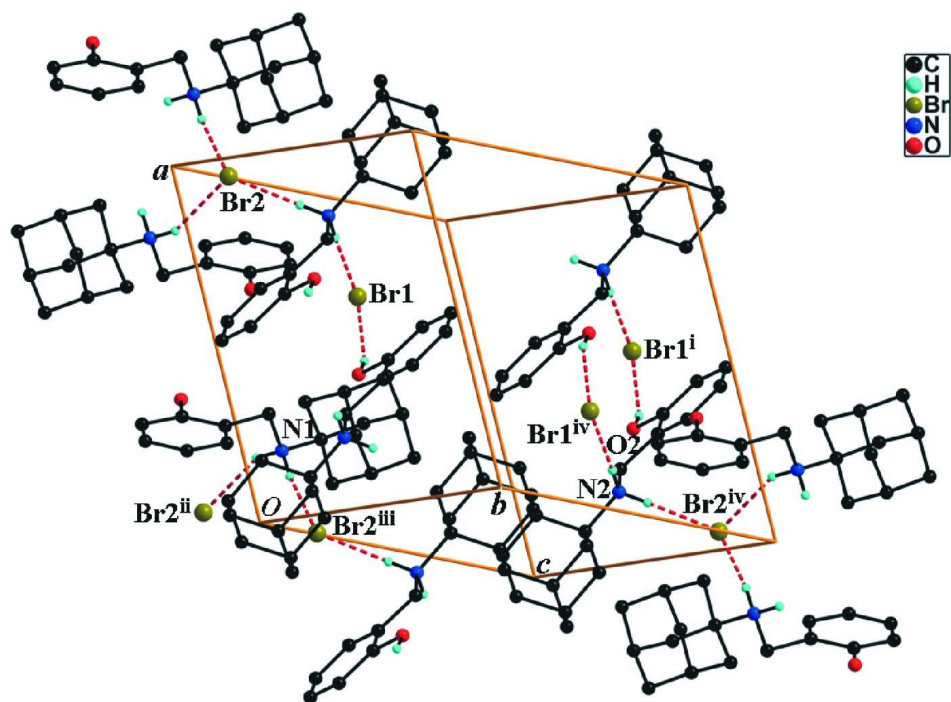
Salicylaldehyde (2.44 g, 20 mmol) and KOH (1.12 g, 20 mmol) were added into a solution of amantadine hydrochloride (3.76 g, 20 mmol) in ethanol. Then a little of anhydrous magnesium sulfate was added into it, after 6 h return the yellow precipitate came out. The yellow solid of amantadine shrink Yang Schiff was obtained by filtration, collection and drying. NaBH₄ (3.78 g, 10 mmol) was added into a solution of amantadine shrink Yang Schiff (6.38 g, 25 mmol) in anhydrous methanol (120 ml). After 5 h reaction, then the white solid, *N*-(2-hydroxybenzyl)-1-adamantylamine was obtained by reduced pressure distillation, extraction and drying. A solution of hydrobromide (0.8 g, 10 mmol) was added to a solution of *N*-(2-hydroxybenzyl)-1-adamantylamine (2.56 g, 10 mmol) in ethanol (20 ml). Single crystals suitable for *X*-ray diffraction were prepared by slow evaporation of the mixture at room temperature.

S3. Refinement

The H atoms of OH group were located in a difference density Fourier map and these H atoms were refined freely with an isotropic displacement parameters $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{O})$. All other H atoms were positioned geometrically and refined using a riding model, with N—H = 0.95Å, C—H = 0.93Å for aryl, 0.98Å for methine and 0.97Å for methylene H atoms, respectively. $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$, and $1.2U_{\text{eq}}(\text{C})$ for aryl, methine and methylene H atoms.


Figure 1

The molecular structure of the title compound, with the atomic numbering scheme. Thermal ellipsoids are shown at the 30% probability level.


Figure 2

A view of the N—H...Br and O—H...Br interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $x, y, z + 1$; (ii) $-x + 1, -y, -z$; (iii) $x - 1, y, z$; (iv) $-x + 1, -y + 1, -z + 1$.]

N-(2-Hydroxybenzyl)adamantan-1-aminium bromide

Crystal data

$C_{17}H_{24}NO^+Br^-$

$M_r = 338.28$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 10.616\ (2)\ \text{\AA}$

$b = 12.627\ (3)\ \text{\AA}$

$c = 12.896\ (3)\ \text{\AA}$

$\alpha = 108.46\ (3)^\circ$

$\beta = 104.69 (3)^\circ$
 $\gamma = 93.88 (3)^\circ$
 $V = 1565.4 (7) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 704$
 $D_x = 1.435 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 7165 reflections
 $\theta = 3.0\text{--}27.5^\circ$
 $\mu = 2.62 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Prism, colourless
 $0.20 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Rigaku SCXmini
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: $13.6612 \text{ pixels mm}^{-1}$
 CCD_Profile_fitting scans
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.596$, $T_{\max} = 0.598$

16356 measured reflections
 7165 independent reflections
 5368 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -13 \rightarrow 13$
 $k = -16 \rightarrow 16$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.108$
 $S = 1.05$
 7165 reflections
 369 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: difference Fourier map
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.2157P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.53 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.55 \text{ e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.63717 (3)	0.35091 (3)	0.25158 (3)	0.04739 (11)
Br2	0.95230 (3)	0.20644 (3)	0.01025 (3)	0.05202 (12)
O1	0.3412 (3)	-0.0956 (2)	-0.0911 (2)	0.0595 (7)
H1O	0.350 (4)	-0.159 (4)	-0.133 (4)	0.089*
N1	0.2065 (2)	0.07541 (18)	0.07930 (19)	0.0354 (5)
H1A	0.1895	-0.0006	0.0484	0.043*
H1B	0.1296	0.1009	0.0614	0.043*
C1	0.2492 (3)	0.0655 (2)	-0.1044 (2)	0.0393 (7)
C2	0.1854 (3)	0.1234 (3)	-0.1706 (3)	0.0481 (8)

H2	0.1656	0.1941	-0.1353	0.058*
C3	0.1503 (3)	0.0787 (3)	-0.2881 (3)	0.0502 (8)
H3	0.1069	0.1187	-0.3319	0.060*
C4	0.1798 (3)	-0.0245 (3)	-0.3394 (3)	0.0473 (7)
H4	0.1568	-0.0544	-0.4188	0.057*
C5	0.2426 (3)	-0.0855 (2)	-0.2770 (3)	0.0450 (7)
H5	0.2613	-0.1562	-0.3135	0.054*
C6	0.2778 (3)	-0.0407 (2)	-0.1589 (3)	0.0409 (7)
C7	0.2971 (3)	0.1177 (3)	0.0236 (3)	0.0487 (8)
H7A	0.3844	0.1005	0.0508	0.058*
H7B	0.3039	0.1993	0.0455	0.058*
C8	0.3704 (3)	0.0488 (3)	0.2401 (3)	0.0485 (8)
H8A	0.4422	0.0723	0.2143	0.058*
H8B	0.3442	-0.0324	0.2036	0.058*
C9	0.2549 (3)	0.1080 (2)	0.2089 (2)	0.0329 (6)
C10	0.4152 (3)	0.0800 (3)	0.3700 (3)	0.0580 (9)
H10	0.4909	0.0430	0.3915	0.070*
C11	0.3040 (4)	0.0408 (3)	0.4091 (3)	0.0658 (10)
H11A	0.3330	0.0588	0.4913	0.079*
H11B	0.2785	-0.0406	0.3733	0.079*
C12	0.4553 (3)	0.2068 (3)	0.4270 (3)	0.0555 (9)
H12A	0.4850	0.2262	0.5093	0.067*
H12B	0.5276	0.2324	0.4029	0.067*
C13	0.3391 (3)	0.2646 (3)	0.3947 (3)	0.0498 (8)
H13	0.3660	0.3467	0.4317	0.060*
C14	0.1411 (3)	0.0686 (3)	0.2471 (3)	0.0492 (8)
H14A	0.1140	-0.0125	0.2102	0.059*
H14B	0.0663	0.1052	0.2256	0.059*
C15	0.2955 (3)	0.2355 (2)	0.2650 (2)	0.0399 (7)
H15A	0.2218	0.2731	0.2431	0.048*
H15B	0.3675	0.2608	0.2404	0.048*
C16	0.1863 (3)	0.0988 (3)	0.3772 (3)	0.0578 (9)
H16	0.1140	0.0733	0.4026	0.069*
C17	0.2266 (3)	0.2272 (3)	0.4337 (3)	0.0583 (9)
H17A	0.1522	0.2644	0.4126	0.070*
H17B	0.2540	0.2477	0.5161	0.070*
O2	0.3888 (2)	0.43144 (18)	1.1055 (2)	0.0513 (6)
H2O	0.445 (4)	0.412 (3)	1.150 (3)	0.077*
N2	0.1643 (2)	0.55623 (17)	0.87142 (17)	0.0301 (5)
H2A	0.2172	0.5849	0.8389	0.036*
H2B	0.1351	0.6146	0.9147	0.036*
C18	0.5517 (3)	0.7260 (3)	1.2349 (3)	0.0489 (8)
H18	0.6185	0.7729	1.2983	0.059*
C19	0.4831 (3)	0.7692 (2)	1.1562 (3)	0.0467 (7)
H19	0.5045	0.8449	1.1656	0.056*
C20	0.3824 (3)	0.6999 (2)	1.0633 (2)	0.0407 (7)
H20	0.3351	0.7301	1.0112	0.049*
C21	0.3507 (3)	0.5867 (2)	1.0463 (2)	0.0327 (6)

C22	0.4228 (3)	0.5436 (2)	1.1257 (2)	0.0348 (6)
C23	0.5217 (3)	0.6137 (3)	1.2199 (3)	0.0445 (7)
H23	0.5681	0.5847	1.2733	0.053*
C24	0.2453 (3)	0.5038 (2)	0.9480 (2)	0.0378 (6)
H24A	0.2862	0.4469	0.9035	0.045*
H24B	0.1877	0.4660	0.9775	0.045*
C25	0.0462 (2)	0.4793 (2)	0.7770 (2)	0.0277 (5)
C26	0.0911 (3)	0.3764 (2)	0.7046 (2)	0.0327 (6)
H26A	0.1287	0.3326	0.7510	0.039*
H26B	0.1580	0.4005	0.6740	0.039*
C27	-0.0289 (3)	0.3042 (2)	0.6069 (2)	0.0370 (6)
H27	-0.0019	0.2375	0.5595	0.044*
C28	-0.1328 (3)	0.2664 (2)	0.6566 (3)	0.0463 (8)
H28A	-0.2083	0.2190	0.5952	0.056*
H28B	-0.0962	0.2224	0.7032	0.056*
C29	-0.0868 (3)	0.3728 (2)	0.5343 (2)	0.0446 (7)
H29A	-0.1617	0.3265	0.4715	0.053*
H29B	-0.0212	0.3970	0.5025	0.053*
C30	-0.1761 (3)	0.3700 (3)	0.7293 (3)	0.0443 (7)
H30	-0.2430	0.3454	0.7608	0.053*
C31	-0.2341 (3)	0.4387 (3)	0.6565 (3)	0.0503 (8)
H31A	-0.2616	0.5046	0.7028	0.060*
H31B	-0.3108	0.3933	0.5952	0.060*
C32	-0.1304 (3)	0.4758 (2)	0.6070 (2)	0.0409 (7)
H32	-0.1676	0.5199	0.5597	0.049*
C33	-0.0113 (3)	0.5488 (2)	0.7043 (2)	0.0359 (6)
H33A	0.0547	0.5739	0.6736	0.043*
H33B	-0.0380	0.6150	0.7509	0.043*
C34	-0.0571 (3)	0.4427 (2)	0.8272 (2)	0.0390 (7)
H34A	-0.0838	0.5086	0.8742	0.047*
H34B	-0.0206	0.3996	0.8748	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03813 (18)	0.0543 (2)	0.0625 (2)	0.01073 (14)	0.01487 (15)	0.03648 (17)
Br2	0.04467 (19)	0.04178 (18)	0.0533 (2)	0.01298 (14)	0.00594 (16)	-0.00015 (14)
O1	0.0695 (16)	0.0559 (14)	0.0533 (15)	0.0148 (13)	0.0061 (13)	0.0273 (12)
N1	0.0318 (12)	0.0330 (12)	0.0376 (13)	0.0032 (10)	0.0085 (11)	0.0086 (10)
C1	0.0353 (16)	0.0419 (16)	0.0349 (15)	-0.0051 (13)	0.0103 (13)	0.0075 (13)
C2	0.054 (2)	0.0377 (16)	0.054 (2)	0.0096 (14)	0.0210 (17)	0.0133 (15)
C3	0.054 (2)	0.0493 (19)	0.0493 (19)	0.0120 (15)	0.0110 (16)	0.0223 (16)
C4	0.0513 (19)	0.0492 (18)	0.0353 (16)	0.0022 (15)	0.0080 (15)	0.0114 (14)
C5	0.0535 (19)	0.0349 (16)	0.0433 (17)	0.0094 (14)	0.0155 (15)	0.0073 (13)
C6	0.0388 (16)	0.0393 (16)	0.0438 (17)	0.0017 (13)	0.0100 (14)	0.0160 (14)
C7	0.0458 (18)	0.0559 (19)	0.0381 (17)	-0.0088 (15)	0.0135 (15)	0.0102 (14)
C8	0.0531 (19)	0.0467 (18)	0.0486 (19)	0.0197 (15)	0.0150 (16)	0.0179 (15)
C9	0.0308 (14)	0.0308 (14)	0.0327 (15)	0.0004 (11)	0.0089 (12)	0.0061 (11)

C10	0.054 (2)	0.067 (2)	0.058 (2)	0.0211 (18)	0.0085 (18)	0.0310 (19)
C11	0.090 (3)	0.051 (2)	0.051 (2)	-0.0070 (19)	0.009 (2)	0.0233 (17)
C12	0.0389 (18)	0.074 (2)	0.0422 (18)	-0.0123 (16)	-0.0001 (15)	0.0184 (17)
C13	0.058 (2)	0.0369 (16)	0.0394 (17)	-0.0080 (15)	0.0072 (16)	0.0021 (13)
C14	0.0389 (17)	0.0560 (19)	0.0426 (18)	-0.0118 (14)	0.0098 (15)	0.0090 (15)
C15	0.0491 (18)	0.0262 (14)	0.0387 (16)	0.0028 (12)	0.0081 (14)	0.0080 (12)
C16	0.049 (2)	0.074 (2)	0.0435 (19)	-0.0169 (18)	0.0161 (16)	0.0153 (17)
C17	0.056 (2)	0.072 (2)	0.0354 (17)	0.0099 (18)	0.0124 (16)	0.0042 (16)
O2	0.0446 (13)	0.0427 (12)	0.0608 (15)	0.0043 (10)	-0.0049 (11)	0.0266 (11)
N2	0.0297 (12)	0.0304 (11)	0.0270 (11)	0.0019 (9)	0.0058 (10)	0.0081 (9)
C18	0.0383 (17)	0.0465 (18)	0.0435 (18)	0.0033 (14)	-0.0022 (14)	0.0029 (14)
C19	0.0441 (18)	0.0358 (16)	0.0521 (19)	0.0048 (13)	0.0064 (15)	0.0107 (14)
C20	0.0386 (16)	0.0420 (16)	0.0378 (16)	0.0079 (13)	0.0034 (13)	0.0145 (13)
C21	0.0283 (14)	0.0378 (15)	0.0315 (14)	0.0071 (11)	0.0099 (12)	0.0098 (12)
C22	0.0283 (14)	0.0390 (15)	0.0370 (15)	0.0078 (12)	0.0084 (12)	0.0134 (12)
C23	0.0360 (16)	0.0545 (19)	0.0365 (16)	0.0092 (14)	-0.0002 (13)	0.0153 (14)
C24	0.0391 (16)	0.0381 (15)	0.0337 (15)	0.0065 (12)	0.0023 (13)	0.0155 (12)
C25	0.0280 (13)	0.0272 (13)	0.0237 (13)	0.0020 (10)	0.0045 (11)	0.0060 (10)
C26	0.0335 (14)	0.0334 (14)	0.0297 (14)	0.0055 (11)	0.0092 (12)	0.0089 (11)
C27	0.0419 (16)	0.0272 (13)	0.0329 (15)	0.0037 (12)	0.0085 (13)	0.0008 (11)
C28	0.0456 (18)	0.0334 (15)	0.0494 (18)	-0.0055 (13)	0.0039 (15)	0.0103 (14)
C29	0.0491 (18)	0.0455 (17)	0.0279 (15)	-0.0019 (14)	0.0009 (14)	0.0077 (13)
C30	0.0342 (16)	0.0470 (17)	0.0484 (18)	-0.0052 (13)	0.0149 (14)	0.0120 (14)
C31	0.0301 (16)	0.0461 (18)	0.058 (2)	0.0037 (13)	0.0014 (15)	0.0053 (15)
C32	0.0381 (16)	0.0380 (16)	0.0375 (16)	0.0040 (13)	-0.0043 (13)	0.0132 (13)
C33	0.0385 (16)	0.0310 (14)	0.0345 (15)	0.0032 (12)	0.0045 (13)	0.0117 (12)
C34	0.0385 (16)	0.0449 (16)	0.0345 (15)	0.0019 (13)	0.0178 (13)	0.0101 (13)

Geometric parameters (Å, °)

O1—C6	1.358 (4)	O2—C22	1.361 (3)
O1—H1O	0.84 (4)	O2—H2O	0.82 (4)
N1—C7	1.500 (4)	N2—C24	1.487 (3)
N1—C9	1.525 (3)	N2—C25	1.520 (3)
N1—H1A	0.9000	N2—H2A	0.9000
N1—H1B	0.9000	N2—H2B	0.9000
C1—C2	1.375 (4)	C18—C19	1.372 (4)
C1—C6	1.396 (4)	C18—C23	1.372 (4)
C1—C7	1.503 (4)	C18—H18	0.9300
C2—C3	1.378 (4)	C19—C20	1.380 (4)
C2—H2	0.9300	C19—H19	0.9300
C3—C4	1.360 (4)	C20—C21	1.380 (4)
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.370 (4)	C21—C22	1.394 (4)
C4—H4	0.9300	C21—C24	1.495 (4)
C5—C6	1.384 (4)	C22—C23	1.381 (4)
C5—H5	0.9300	C23—H23	0.9300
C7—H7A	0.9700	C24—H24A	0.9700

C7—H7B	0.9700	C24—H24B	0.9700
C8—C9	1.512 (4)	C25—C34	1.522 (4)
C8—C10	1.530 (5)	C25—C33	1.524 (3)
C8—H8A	0.9700	C25—C26	1.526 (4)
C8—H8B	0.9700	C26—C27	1.531 (4)
C9—C15	1.521 (3)	C26—H26A	0.9700
C9—C14	1.525 (4)	C26—H26B	0.9700
C10—C11	1.508 (5)	C27—C29	1.516 (4)
C10—C12	1.513 (5)	C27—C28	1.529 (4)
C10—H10	0.9800	C27—H27	0.9800
C11—C16	1.524 (5)	C28—C30	1.524 (4)
C11—H11A	0.9700	C28—H28A	0.9700
C11—H11B	0.9700	C28—H28B	0.9700
C12—C13	1.510 (5)	C29—C32	1.521 (4)
C12—H12A	0.9700	C29—H29A	0.9700
C12—H12B	0.9700	C29—H29B	0.9700
C13—C17	1.511 (5)	C30—C31	1.519 (4)
C13—C15	1.530 (4)	C30—C34	1.527 (4)
C13—H13	0.9800	C30—H30	0.9800
C14—C16	1.533 (4)	C31—C32	1.521 (4)
C14—H14A	0.9700	C31—H31A	0.9700
C14—H14B	0.9700	C31—H31B	0.9700
C15—H15A	0.9700	C32—C33	1.526 (4)
C15—H15B	0.9700	C32—H32	0.9800
C16—C17	1.530 (5)	C33—H33A	0.9700
C16—H16	0.9800	C33—H33B	0.9700
C17—H17A	0.9700	C34—H34A	0.9700
C17—H17B	0.9700	C34—H34B	0.9700
C6—O1—H1O	109 (3)	C22—O2—H2O	108 (3)
C7—N1—C9	116.9 (2)	C24—N2—C25	116.5 (2)
C7—N1—H1A	108.1	C24—N2—H2A	108.2
C9—N1—H1A	108.1	C25—N2—H2A	108.2
C7—N1—H1B	108.1	C24—N2—H2B	108.2
C9—N1—H1B	108.1	C25—N2—H2B	108.2
H1A—N1—H1B	107.3	H2A—N2—H2B	107.3
C2—C1—C6	118.5 (3)	C19—C18—C23	120.0 (3)
C2—C1—C7	121.4 (3)	C19—C18—H18	120.0
C6—C1—C7	119.9 (3)	C23—C18—H18	120.0
C1—C2—C3	121.3 (3)	C18—C19—C20	119.9 (3)
C1—C2—H2	119.3	C18—C19—H19	120.1
C3—C2—H2	119.3	C20—C19—H19	120.1
C4—C3—C2	119.2 (3)	C19—C20—C21	121.1 (3)
C4—C3—H3	120.4	C19—C20—H20	119.5
C2—C3—H3	120.4	C21—C20—H20	119.5
C3—C4—C5	121.5 (3)	C20—C21—C22	118.4 (3)
C3—C4—H4	119.2	C20—C21—C24	125.6 (2)
C5—C4—H4	119.2	C22—C21—C24	116.1 (2)

C4—C5—C6	119.3 (3)	O2—C22—C23	123.2 (3)
C4—C5—H5	120.4	O2—C22—C21	116.5 (2)
C6—C5—H5	120.4	C23—C22—C21	120.3 (3)
O1—C6—C5	122.7 (3)	C18—C23—C22	120.3 (3)
O1—C6—C1	117.1 (3)	C18—C23—H23	119.8
C5—C6—C1	120.1 (3)	C22—C23—H23	119.8
N1—C7—C1	112.0 (2)	N2—C24—C21	113.7 (2)
N1—C7—H7A	109.2	N2—C24—H24A	108.8
C1—C7—H7A	109.2	C21—C24—H24A	108.8
N1—C7—H7B	109.2	N2—C24—H24B	108.8
C1—C7—H7B	109.2	C21—C24—H24B	108.8
H7A—C7—H7B	107.9	H24A—C24—H24B	107.7
C9—C8—C10	108.5 (3)	N2—C25—C34	110.6 (2)
C9—C8—H8A	110.0	N2—C25—C33	106.36 (19)
C10—C8—H8A	110.0	C34—C25—C33	109.5 (2)
C9—C8—H8B	110.0	N2—C25—C26	109.8 (2)
C10—C8—H8B	110.0	C34—C25—C26	110.6 (2)
H8A—C8—H8B	108.4	C33—C25—C26	110.0 (2)
C8—C9—C15	110.3 (2)	C25—C26—C27	108.3 (2)
C8—C9—N1	109.2 (2)	C25—C26—H26A	110.0
C15—C9—N1	110.2 (2)	C27—C26—H26A	110.0
C8—C9—C14	109.8 (2)	C25—C26—H26B	110.0
C15—C9—C14	109.8 (2)	C27—C26—H26B	110.0
N1—C9—C14	107.4 (2)	H26A—C26—H26B	108.4
C11—C10—C12	109.4 (3)	C29—C27—C28	109.5 (2)
C11—C10—C8	109.6 (3)	C29—C27—C26	109.8 (2)
C12—C10—C8	110.0 (3)	C28—C27—C26	109.3 (2)
C11—C10—H10	109.3	C29—C27—H27	109.4
C12—C10—H10	109.3	C28—C27—H27	109.4
C8—C10—H10	109.3	C26—C27—H27	109.4
C10—C11—C16	109.7 (3)	C30—C28—C27	109.5 (2)
C10—C11—H11A	109.7	C30—C28—H28A	109.8
C16—C11—H11A	109.7	C27—C28—H28A	109.8
C10—C11—H11B	109.7	C30—C28—H28B	109.8
C16—C11—H11B	109.7	C27—C28—H28B	109.8
H11A—C11—H11B	108.2	H28A—C28—H28B	108.2
C13—C12—C10	109.6 (3)	C27—C29—C32	109.7 (2)
C13—C12—H12A	109.7	C27—C29—H29A	109.7
C10—C12—H12A	109.7	C32—C29—H29A	109.7
C13—C12—H12B	109.7	C27—C29—H29B	109.7
C10—C12—H12B	109.7	C32—C29—H29B	109.7
H12A—C12—H12B	108.2	H29A—C29—H29B	108.2
C12—C13—C17	110.3 (3)	C31—C30—C28	109.9 (3)
C12—C13—C15	109.1 (3)	C31—C30—C34	109.6 (2)
C17—C13—C15	110.2 (3)	C28—C30—C34	109.3 (2)
C12—C13—H13	109.1	C31—C30—H30	109.3
C17—C13—H13	109.1	C28—C30—H30	109.3
C15—C13—H13	109.1	C34—C30—H30	109.3

C9—C14—C16	109.1 (2)	C30—C31—C32	109.2 (2)
C9—C14—H14A	109.9	C30—C31—H31A	109.8
C16—C14—H14A	109.9	C32—C31—H31A	109.8
C9—C14—H14B	109.9	C30—C31—H31B	109.8
C16—C14—H14B	109.9	C32—C31—H31B	109.8
H14A—C14—H14B	108.3	H31A—C31—H31B	108.3
C9—C15—C13	108.5 (2)	C29—C32—C31	110.1 (2)
C9—C15—H15A	110.0	C29—C32—C33	109.2 (2)
C13—C15—H15A	110.0	C31—C32—C33	109.3 (2)
C9—C15—H15B	110.0	C29—C32—H32	109.4
C13—C15—H15B	110.0	C31—C32—H32	109.4
H15A—C15—H15B	108.4	C33—C32—H32	109.4
C11—C16—C17	109.7 (3)	C25—C33—C32	109.0 (2)
C11—C16—C14	109.0 (3)	C25—C33—H33A	109.9
C17—C16—C14	109.0 (3)	C32—C33—H33A	109.9
C11—C16—H16	109.7	C25—C33—H33B	109.9
C17—C16—H16	109.7	C32—C33—H33B	109.9
C14—C16—H16	109.7	H33A—C33—H33B	108.3
C13—C17—C16	108.9 (3)	C25—C34—C30	108.7 (2)
C13—C17—H17A	109.9	C25—C34—H34A	110.0
C16—C17—H17A	109.9	C30—C34—H34A	110.0
C13—C17—H17B	109.9	C25—C34—H34B	110.0
C16—C17—H17B	109.9	C30—C34—H34B	110.0
H17A—C17—H17B	108.3	H34A—C34—H34B	108.3
C6—C1—C2—C3	-0.2 (4)	C23—C18—C19—C20	1.2 (5)
C7—C1—C2—C3	175.1 (3)	C18—C19—C20—C21	-1.4 (5)
C1—C2—C3—C4	-0.2 (5)	C19—C20—C21—C22	0.2 (4)
C2—C3—C4—C5	0.6 (5)	C19—C20—C21—C24	-179.0 (3)
C3—C4—C5—C6	-0.5 (5)	C20—C21—C22—O2	-179.7 (3)
C4—C5—C6—O1	-179.4 (3)	C24—C21—C22—O2	-0.4 (4)
C4—C5—C6—C1	0.1 (4)	C20—C21—C22—C23	1.1 (4)
C2—C1—C6—O1	179.8 (3)	C24—C21—C22—C23	-179.6 (3)
C7—C1—C6—O1	4.4 (4)	C19—C18—C23—C22	0.1 (5)
C2—C1—C6—C5	0.3 (4)	O2—C22—C23—C18	179.6 (3)
C7—C1—C6—C5	-175.2 (3)	C21—C22—C23—C18	-1.3 (4)
C9—N1—C7—C1	170.8 (2)	C25—N2—C24—C21	-173.7 (2)
C2—C1—C7—N1	101.4 (3)	C20—C21—C24—N2	-5.7 (4)
C6—C1—C7—N1	-83.3 (3)	C22—C21—C24—N2	175.1 (2)
C10—C8—C9—C15	60.2 (3)	C24—N2—C25—C34	66.7 (3)
C10—C8—C9—N1	-178.6 (2)	C24—N2—C25—C33	-174.5 (2)
C10—C8—C9—C14	-61.0 (3)	C24—N2—C25—C26	-55.5 (3)
C7—N1—C9—C8	-67.9 (3)	N2—C25—C26—C27	-177.1 (2)
C7—N1—C9—C15	53.4 (3)	C34—C25—C26—C27	60.7 (3)
C7—N1—C9—C14	173.1 (3)	C33—C25—C26—C27	-60.4 (3)
C9—C8—C10—C11	61.0 (3)	C25—C26—C27—C29	60.0 (3)
C9—C8—C10—C12	-59.3 (4)	C25—C26—C27—C28	-60.0 (3)
C12—C10—C11—C16	59.9 (4)	C29—C27—C28—C30	-59.4 (3)

C8—C10—C11—C16	-60.8 (4)	C26—C27—C28—C30	60.8 (3)
C11—C10—C12—C13	-60.2 (4)	C28—C27—C29—C32	59.4 (3)
C8—C10—C12—C13	60.3 (4)	C26—C27—C29—C32	-60.5 (3)
C10—C12—C13—C17	60.4 (3)	C27—C28—C30—C31	59.7 (3)
C10—C12—C13—C15	-60.7 (3)	C27—C28—C30—C34	-60.6 (3)
C8—C9—C14—C16	60.8 (3)	C28—C30—C31—C32	-59.5 (3)
C15—C9—C14—C16	-60.7 (3)	C34—C30—C31—C32	60.7 (3)
N1—C9—C14—C16	179.4 (3)	C27—C29—C32—C31	-59.8 (3)
C8—C9—C15—C13	-61.1 (3)	C27—C29—C32—C33	60.2 (3)
N1—C9—C15—C13	178.2 (2)	C30—C31—C32—C29	59.5 (3)
C14—C9—C15—C13	60.0 (3)	C30—C31—C32—C33	-60.5 (3)
C12—C13—C15—C9	60.6 (3)	N2—C25—C33—C32	179.6 (2)
C17—C13—C15—C9	-60.6 (3)	C34—C25—C33—C32	-60.9 (3)
C10—C11—C16—C17	-59.4 (4)	C26—C25—C33—C32	60.8 (3)
C10—C11—C16—C14	59.8 (4)	C29—C32—C33—C25	-60.0 (3)
C9—C14—C16—C11	-59.4 (3)	C31—C32—C33—C25	60.5 (3)
C9—C14—C16—C17	60.3 (4)	N2—C25—C34—C30	177.5 (2)
C12—C13—C17—C16	-59.4 (3)	C33—C25—C34—C30	60.7 (3)
C15—C13—C17—C16	61.1 (4)	C26—C25—C34—C30	-60.7 (3)
C11—C16—C17—C13	58.7 (4)	C31—C30—C34—C25	-60.6 (3)
C14—C16—C17—C13	-60.6 (4)	C28—C30—C34—C25	59.9 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2O...Br1 ⁱ	0.82 (4)	2.45 (4)	3.255 (2)	168 (4)
N1—H1A...Br2 ⁱⁱ	0.90	2.69	3.527 (3)	155
N1—H1B...Br2 ⁱⁱⁱ	0.90	2.45	3.337 (2)	167
N2—H2A...Br1 ^{iv}	0.90	2.40	3.297 (2)	176
N2—H2B...Br2 ^{iv}	0.90	2.50	3.377 (2)	165

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