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N-(2-Iodophenylmethyl)hexamethylenetetraminium bromide dihydrate

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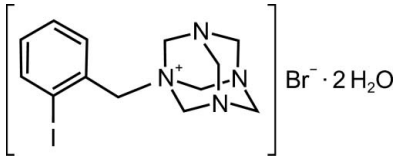
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.061; data-to-parameter ratio = 18.6.

The title compound, $C_{13}H_{18}IN_4^+\cdot Br^-\cdot 2H_2O$, is a quaternary ammonium salt derived from 2-iodobenzyl bromide and hexamethylenetetramine. Two water molecules per asymmetric unit act as hydrogen-bond donors to the bromide anion. Direct cation–anion contacts are established *via* C—H donors, mainly from methylene groups in the α position to the ammonium centre.

Related literature

For synthesis of the title compound, see Angyal *et al.* (1949). For the crystal structure of a related compound, see Shao *et al.* (1982).



Experimental

Crystal data

$C_{13}H_{18}IN_4^+\cdot Br^-\cdot 2H_2O$
M_r = 473.15
Triclinic, $P\overline{1}$
a = 9.084 (4) Å
b = 9.500 (5) Å
c = 10.495 (7) Å
α = 69.28 (5)°
β = 75.47 (5)°
γ = 83.76 (4)°
V = 819.8 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.40 mm^{-1}
T = 200 (2) K
0.28 × 0.15 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: numerical [crystal faces optimized with *X-SHAPE* (Stoe & Cie, 1997) and absorption correction with *X-RED* (Stoe & Cie, 1997)]
T_{min} = 0.409, T_{max} = 0.610
10405 measured reflections
3820 independent reflections
2865 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.061
S = 0.95
3820 reflections
205 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −1.07 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Br	0.77 (3)	2.53 (3)	3.297 (4)	172 (3)
O1—H12···N3^{i}	0.77 (3)	2.72 (3)	3.390 (5)	146 (4)
O2—H21···Br	0.78 (3)	2.61 (3)	3.381 (4)	168 (4)
O2—H22···Br^{ii}	0.79 (3)	2.65 (3)	3.433 (4)	174 (3)
C3—H3···I^{iii}	0.95	3.31	4.237 (4)	166
C5—H5···Br^{iv}	0.95	3.05	3.924 (4)	153
C6—H6···N4^{v}	0.95	2.73	3.558 (5)	146
C7—H72···Br	0.99	2.99	3.872 (4)	149
C8—H82···O1^{v}	0.99	2.78	3.640 (5)	145
C9—H91···O2^{vi}	0.99	2.40	3.326 (5)	156
C10—H101···Br	0.99	2.97	3.875 (4)	153
C10—H102···O2^{vii}	0.99	2.47	3.459 (5)	174
C12—H121···O1^{v}	0.99	2.78	3.643 (5)	146
C12—H122···Br	0.99	3.07	3.952 (4)	149
C13—H131···O1^{v}	0.99	2.56	3.481 (5)	154

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, -y, -z+1$; (iii) $-x+1, -y+2, -z+1$; (iv) $x+1, y, z$; (v) $-x+1, -y+1, -z$; (vi) $x, y+1, z-1$; (vii) $-x, -y+1, -z+1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 1990).

The authors thank Dr Peter Mayer and Sandra Albrecht for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2048).

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supplementary materials

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N-(2-Iodophenylmethyl)hexamethylenetetraminium bromide dihydrate

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Comment

In the quaternary ammonium salt, hexamethylenetetramine is *N*–alkylated by an *ortho*–iodobenzyl moiety. The positive charge of the ammonium centre atom is counterbalanced by a bromide ion (Fig. 1). Intramolecular bond lengths and angles are normal.

The bromide ions are embedded into an environment of 7 hydrogen–bond donors (CIF: the _geom_special_details item for the applied criteria). One hemisphere around the anion contains three O—H donors, the other hemisphere is established by four C—H···Br contacts. Of these, three bonds have methylene functions as donors which are in α–position to the quaternary ammonium centre (Fig. 1), the fourth stemming from a phenyl–CH. The O2 donors are part of $OH_2Br_2H_2O$ rhombs (Fig. 2). All the water molecules are close to the boundary planes of the chosen unit cell. There are no hydrogen–bonded contacts between water molecules. Instead, the acceptor functions of the O atoms are occupied by C—H donors. Again, there is a predominance of α–methylene donors. The acceptor capability of the amine–N atoms is less pronounced than that of the anion and the water–O atoms. There are relatively long bonds from water (N3) and phenyl–CH donors (N4), whereas N2 is not found as an acceptor within the applied criteria.

Experimental

The title compound was obtained as an intermediate in the synthesis of *ortho*–iodomandelic acid according to a published procedure (Angyal *et al.*, 1949) upon addition of *ortho*–iodobenzyl bromide to a solution of hexamethylenetetramine in chloroform. Crystals suitable for *X*–ray analysis were obtained upon the cooling of a saturated aqueous solution to 277 K.

Refinement

The H atoms on C atoms were located in a difference map and refined as riding on their parent atoms with a common U_{iso} value. The H atoms on the water molecules were located in a difference map and refined with a common O—H distance and a constant 105° bond angle. A common U_{iso} value was refined for the O–bonded H atoms. Position of the residual peak - deepest hole, −1.07 e $Å^{-3}$ at 0.3169 0.8397 0.5303 with the distance 0.82Å from I.

Figures

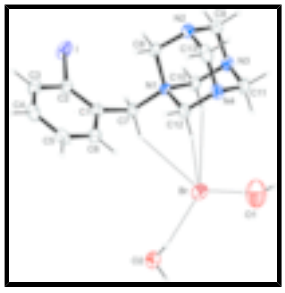

Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at 50% probability level. Hydrogen atoms are presented as a spheres of arbitrary radius. Hydrogen bonds are indicated as dashed lines.

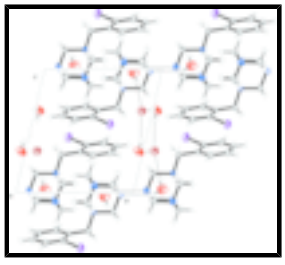

Fig. 2. The packing of title compound, viewed along [0 1 0]. Some of the hydrogen bonds with water donors are drawn as dashed lines.

N-(2-Iodophenylmethyl)hexamethylenetetraminium bromide dihydrate

Crystal data

$C_{13}H_{18}IN_4^+ \cdot Br^- \cdot 2H_2O$	$V = 819.8\ (8)\ Å^3$
$M_r = 473.15$	$Z = 2$
Triclinic, $P\overline{1}$	$F_{000} = 464$
Hall symbol: -P 1	$D_x = 1.917\ Mg\ m^{-3}$
$a = 9.084\ (4)\ Å$	Mo $K\alpha$ radiation $\lambda = 0.71073\ Å$
$b = 9.500\ (5)\ Å$	$\theta = 3.8–27.5°$
$c = 10.495\ (7)\ Å$	$\mu = 4.40\ mm^{-1}$
$\alpha = 69.28\ (5)°$	$T = 200\ (2)\ K$
$\beta = 75.47\ (5)°$	Block, colourless'
$\gamma = 83.76\ (4)°$	$0.28 \times 0.15 \times 0.14\ mm$

Data collection

Nonius KappaCCD diffractometer	3820 independent reflections
Radiation source: Fine-focus sealed tube	2865 reflections with $I > 2\sigma(I)$
Monochromator: Graphite	$R_{int} = 0.032$
$T = 200(2)\ K$	$\theta_{max} = 27.6°$
ω scans	$\theta_{min} = 3.8°$
Absorption correction: numerical [crystal faces optimized with X-SHAPE (Stoe & Cie, 1997) and absorption correction with X-RED (Stoe & Cie, 1997)]	$h = -11 \rightarrow 11$
$T_{min} = 0.409$, $T_{max} = 0.610$	$k = -12 \rightarrow 12$

10405 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.061$	$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} < 0.001$
3820 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
205 parameters	$\Delta\rho_{min} = -1.07$ e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. The hydrogen–bond list was generated with the *PLATON* command "hbond 1.. 4 140", *i.e.*, D···A is less than the sum of the vdW radii + 1 Å, H···A is less than the sum of the vdW radii + 0.4 Å, the D—H···A angle is greater than 140°.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I	0.33565 (2)	0.89354 (2)	0.44815 (2)	0.03398 (8)
Br	0.09573 (4)	0.28677 (3)	0.33816 (3)	0.03467 (9)
O1	0.2401 (3)	0.2543 (4)	0.0283 (3)	0.0676 (9)
H11	0.199 (4)	0.258 (5)	0.101 (3)	0.063 (7)*
H12	0.177 (3)	0.274 (5)	−0.013 (4)	0.063 (7)*
O2	0.0335 (3)	0.0367 (3)	0.6679 (3)	0.0435 (6)
H21	0.037 (5)	0.088 (4)	0.590 (3)	0.063 (7)*
H22	−0.001 (4)	−0.038 (3)	0.672 (4)	0.063 (7)*
N1	0.2986 (2)	0.7033 (2)	0.1572 (2)	0.0182 (5)
N2	0.3446 (3)	0.9209 (3)	−0.0563 (3)	0.0279 (5)
N3	0.1101 (3)	0.7836 (3)	0.0132 (3)	0.0250 (5)
N4	0.3522 (3)	0.6716 (3)	−0.0710 (2)	0.0268 (5)
C1	0.4668 (3)	0.6402 (3)	0.3306 (3)	0.0206 (6)
C2	0.5020 (3)	0.7436 (3)	0.3847 (3)	0.0216 (6)
C3	0.6450 (3)	0.7475 (3)	0.4062 (3)	0.0265 (6)
H3	0.6666	0.8194	0.4427	0.0245 (18)*
C4	0.7556 (3)	0.6472 (4)	0.3748 (3)	0.0289 (7)
H4	0.8546	0.6511	0.3880	0.0245 (18)*
C5	0.7247 (3)	0.5399 (3)	0.3240 (3)	0.0275 (6)
H5	0.8014	0.4696	0.3036	0.0245 (18)*
C6	0.5811 (3)	0.5368 (3)	0.3035 (3)	0.0244 (6)
H6	0.5594	0.4623	0.2702	0.0245 (18)*

C7	0.3114 (3)	0.6314 (3)	0.3075 (3)	0.0220 (6)
H71	0.2364	0.6808	0.3655	0.0245 (18)*
H72	0.2844	0.5241	0.3404	0.0245 (18)*
C8	0.3672 (3)	0.8595 (3)	0.0848 (3)	0.0242 (6)
H81	0.3181	0.9267	0.1377	0.0245 (18)*
H82	0.4773	0.8525	0.0824	0.0245 (18)*
C9	0.1816 (3)	0.9313 (3)	−0.0520 (3)	0.0299 (7)
H91	0.1664	0.9774	−0.1485	0.0245 (18)*
H92	0.1312	0.9979	0.0013	0.0245 (18)*
C10	0.1299 (3)	0.7186 (3)	0.1554 (3)	0.0217 (6)
H101	0.0833	0.6181	0.2001	0.0245 (18)*
H102	0.0775	0.7833	0.2099	0.0245 (18)*
C11	0.1891 (3)	0.6848 (3)	−0.0663 (3)	0.0275 (6)
H111	0.1444	0.5836	−0.0218	0.0245 (18)*
H112	0.1736	0.7264	−0.1629	0.0245 (18)*
C12	0.3752 (3)	0.6060 (3)	0.0696 (3)	0.0230 (6)
H121	0.4855	0.5965	0.0666	0.0245 (18)*
H122	0.3323	0.5039	0.1134	0.0245 (18)*
C13	0.4181 (3)	0.8223 (3)	−0.1354 (3)	0.0307 (7)
H131	0.5284	0.8137	−0.1383	0.0245 (18)*
H132	0.4053	0.8669	−0.2327	0.0245 (18)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I	0.02658 (11)	0.03900 (13)	0.04832 (15)	0.00253 (8)	−0.00951 (9)	−0.02950 (11)
Br	0.03848 (18)	0.02750 (17)	0.0393 (2)	−0.00699 (13)	−0.01011 (14)	−0.01016 (14)
O1	0.0583 (19)	0.099 (3)	0.053 (2)	−0.0062 (18)	−0.0106 (14)	−0.0341 (19)
O2	0.0603 (16)	0.0328 (13)	0.0465 (16)	−0.0065 (12)	−0.0284 (14)	−0.0117 (12)
N1	0.0171 (11)	0.0205 (11)	0.0178 (12)	−0.0020 (9)	−0.0049 (9)	−0.0062 (9)
N2	0.0337 (14)	0.0234 (13)	0.0271 (14)	−0.0051 (11)	−0.0131 (11)	−0.0036 (11)
N3	0.0250 (12)	0.0236 (12)	0.0328 (14)	0.0027 (10)	−0.0137 (11)	−0.0132 (11)
N4	0.0303 (13)	0.0309 (14)	0.0244 (13)	0.0048 (11)	−0.0106 (11)	−0.0141 (11)
C1	0.0197 (13)	0.0240 (14)	0.0157 (14)	−0.0038 (11)	−0.0027 (11)	−0.0039 (11)
C2	0.0194 (13)	0.0235 (14)	0.0219 (15)	−0.0032 (11)	−0.0028 (11)	−0.0083 (12)
C3	0.0235 (14)	0.0319 (16)	0.0268 (16)	−0.0071 (12)	−0.0045 (12)	−0.0124 (13)
C4	0.0192 (14)	0.0404 (17)	0.0244 (16)	−0.0029 (13)	−0.0054 (12)	−0.0066 (13)
C5	0.0277 (15)	0.0308 (16)	0.0217 (16)	0.0086 (13)	−0.0065 (12)	−0.0082 (13)
C6	0.0313 (15)	0.0227 (14)	0.0217 (15)	0.0006 (12)	−0.0073 (12)	−0.0100 (12)
C7	0.0231 (14)	0.0263 (14)	0.0182 (14)	−0.0067 (11)	−0.0057 (11)	−0.0073 (12)
C8	0.0302 (15)	0.0172 (13)	0.0273 (16)	−0.0069 (12)	−0.0096 (12)	−0.0063 (12)
C9	0.0378 (17)	0.0227 (15)	0.0321 (18)	0.0041 (13)	−0.0164 (14)	−0.0085 (13)
C10	0.0172 (13)	0.0216 (14)	0.0301 (16)	0.0003 (11)	−0.0070 (11)	−0.0124 (12)
C11	0.0326 (16)	0.0302 (16)	0.0275 (16)	0.0027 (13)	−0.0135 (13)	−0.0157 (13)
C12	0.0264 (14)	0.0239 (15)	0.0227 (15)	0.0062 (12)	−0.0066 (12)	−0.0141 (12)
C13	0.0287 (15)	0.0400 (18)	0.0196 (16)	−0.0036 (13)	−0.0053 (12)	−0.0050 (13)

Geometric parameters (Å, °)

I—C2	2.111 (3)	C3—C4	1.369 (4)
O1—H11	0.78 (2)	C3—H3	0.9500
O1—H12	0.78 (2)	C4—C5	1.387 (4)
O2—H21	0.78 (2)	C4—H4	0.9500
O2—H22	0.78 (2)	C5—C6	1.379 (4)
N1—C7	1.510 (3)	C5—H5	0.9500
N1—C12	1.525 (3)	C6—H6	0.9500
N1—C8	1.527 (4)	C7—H71	0.9900
N1—C10	1.528 (3)	C7—H72	0.9900
N2—C8	1.446 (4)	C8—H81	0.9900
N2—C9	1.464 (4)	C8—H82	0.9900
N2—C13	1.466 (4)	C9—H91	0.9900
N3—C10	1.449 (4)	C9—H92	0.9900
N3—C9	1.468 (4)	C10—H101	0.9900
N3—C11	1.483 (3)	C10—H102	0.9900
N4—C12	1.444 (4)	C11—H111	0.9900
N4—C11	1.463 (4)	C11—H112	0.9900
N4—C13	1.470 (4)	C12—H121	0.9900
C1—C2	1.393 (4)	C12—H122	0.9900
C1—C6	1.397 (4)	C13—H131	0.9900
C1—C7	1.507 (4)	C13—H132	0.9900
C2—C3	1.380 (4)		
H11—O1—H12	105 (2)	N1—C7—H72	108.7
H21—O2—H22	103 (2)	H71—C7—H72	107.6
C7—N1—C12	111.5 (2)	N2—C8—N1	109.7 (2)
C7—N1—C8	113.9 (2)	N2—C8—H81	109.7
C12—N1—C8	107.5 (2)	N1—C8—H81	109.7
C7—N1—C10	108.0 (2)	N2—C8—H82	109.7
C12—N1—C10	107.67 (19)	N1—C8—H82	109.7
C8—N1—C10	108.0 (2)	H81—C8—H82	108.2
C8—N2—C9	109.4 (2)	N2—C9—N3	112.2 (2)
C8—N2—C13	109.9 (2)	N2—C9—H91	109.2
C9—N2—C13	108.7 (2)	N3—C9—H91	109.2
C10—N3—C9	108.8 (2)	N2—C9—H92	109.2
C10—N3—C11	108.6 (2)	N3—C9—H92	109.2
C9—N3—C11	108.3 (2)	H91—C9—H92	107.9
C12—N4—C11	109.4 (2)	N3—C10—N1	110.7 (2)
C12—N4—C13	109.0 (2)	N3—C10—H101	109.5
C11—N4—C13	109.3 (2)	N1—C10—H101	109.5
C2—C1—C6	117.2 (2)	N3—C10—H102	109.5
C2—C1—C7	123.1 (2)	N1—C10—H102	109.5
C6—C1—C7	119.7 (2)	H101—C10—H102	108.1
C3—C2—C1	121.6 (2)	N4—C11—N3	111.5 (2)
C3—C2—I	116.74 (19)	N4—C11—H111	109.3
C1—C2—I	121.55 (19)	N3—C11—H111	109.3
C4—C3—C2	119.7 (3)	N4—C11—H112	109.3

C4—C3—H3	120.2	N3—C11—H112	109.3
C2—C3—H3	120.2	H111—C11—H112	108.0
C3—C4—C5	120.7 (3)	N4—C12—N1	110.6 (2)
C3—C4—H4	119.6	N4—C12—H121	109.5
C5—C4—H4	119.6	N1—C12—H121	109.5
C6—C5—C4	119.0 (3)	N4—C12—H122	109.5
C6—C5—H5	120.5	N1—C12—H122	109.5
C4—C5—H5	120.5	H121—C12—H122	108.1
C5—C6—C1	121.7 (2)	N2—C13—N4	111.0 (2)
C5—C6—H6	119.1	N2—C13—H131	109.4
C1—C6—H6	119.1	N4—C13—H131	109.4
C1—C7—N1	114.4 (2)	N2—C13—H132	109.4
C1—C7—H71	108.7	N4—C13—H132	109.4
N1—C7—H71	108.7	H131—C13—H132	108.0
C1—C7—H72	108.7		
C6—C1—C2—C3	2.2 (4)	C13—N2—C9—N3	58.9 (3)
C7—C1—C2—C3	179.2 (2)	C10—N3—C9—N2	60.1 (3)
C6—C1—C2—I	−174.31 (19)	C11—N3—C9—N2	−57.7 (3)
C7—C1—C2—I	2.6 (4)	C9—N3—C10—N1	−58.4 (3)
C1—C2—C3—C4	−0.4 (4)	C11—N3—C10—N1	59.3 (3)
I—C2—C3—C4	176.3 (2)	C7—N1—C10—N3	−178.5 (2)
C2—C3—C4—C5	−1.2 (4)	C12—N1—C10—N3	−57.9 (3)
C3—C4—C5—C6	0.9 (4)	C8—N1—C10—N3	57.9 (3)
C4—C5—C6—C1	1.0 (4)	C12—N4—C11—N3	61.0 (3)
C2—C1—C6—C5	−2.5 (4)	C13—N4—C11—N3	−58.2 (3)
C7—C1—C6—C5	−179.6 (3)	C10—N3—C11—N4	−60.8 (3)
C2—C1—C7—N1	101.8 (3)	C9—N3—C11—N4	57.2 (3)
C6—C1—C7—N1	−81.3 (3)	C11—N4—C12—N1	−59.5 (3)
C12—N1—C7—C1	75.0 (3)	C13—N4—C12—N1	59.9 (3)
C8—N1—C7—C1	−46.9 (3)	C7—N1—C12—N4	175.9 (2)
C10—N1—C7—C1	−166.8 (2)	C8—N1—C12—N4	−58.6 (3)
C9—N2—C8—N1	59.6 (3)	C10—N1—C12—N4	57.6 (3)
C13—N2—C8—N1	−59.8 (3)	C8—N2—C13—N4	61.1 (3)
C7—N1—C8—N2	−178.0 (2)	C9—N2—C13—N4	−58.7 (3)
C12—N1—C8—N2	57.9 (3)	C12—N4—C13—N2	−60.6 (3)
C10—N1—C8—N2	−58.0 (3)	C11—N4—C13—N2	58.9 (3)
C8—N2—C9—N3	−61.2 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···Br	0.77 (3)	2.53 (3)	3.297 (4)	172 (3)
O1—H12···N3^{i}	0.77 (3)	2.72 (3)	3.390 (5)	146 (4)
O2—H21···Br	0.78 (3)	2.61 (3)	3.381 (4)	168 (4)
O2—H22···Br^{ii}	0.79 (3)	2.65 (3)	3.433 (4)	174 (3)
C3—H3···I^{iii}	0.95	3.31	4.237 (4)	166
C5—H5···Br^{iv}	0.95	3.05	3.924 (4)	153
C6—H6···N4^{v}	0.95	2.73	3.558 (5)	146

C7—H72···Br	0.99	2.99	3.872 (4)	149
C8—H82···O1^{v}	0.99	2.78	3.640 (5)	145
C9—H91···O2^{vi}	0.99	2.40	3.326 (5)	156
C10—H101···Br	0.99	2.97	3.875 (4)	153
C10—H102···O2^{vii}	0.99	2.47	3.459 (5)	174
C12—H121···O1^{v}	0.99	2.78	3.643 (5)	146
C12—H122···Br	0.99	3.07	3.952 (4)	149
C13—H131···O1^{v}	0.99	2.56	3.481 (5)	154

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+2, −*z*+1; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*; (vi) *x*, *y*+1, *z*−1; (vii) −*x*, −*y*+1, −*z*+1.

Fig. 1

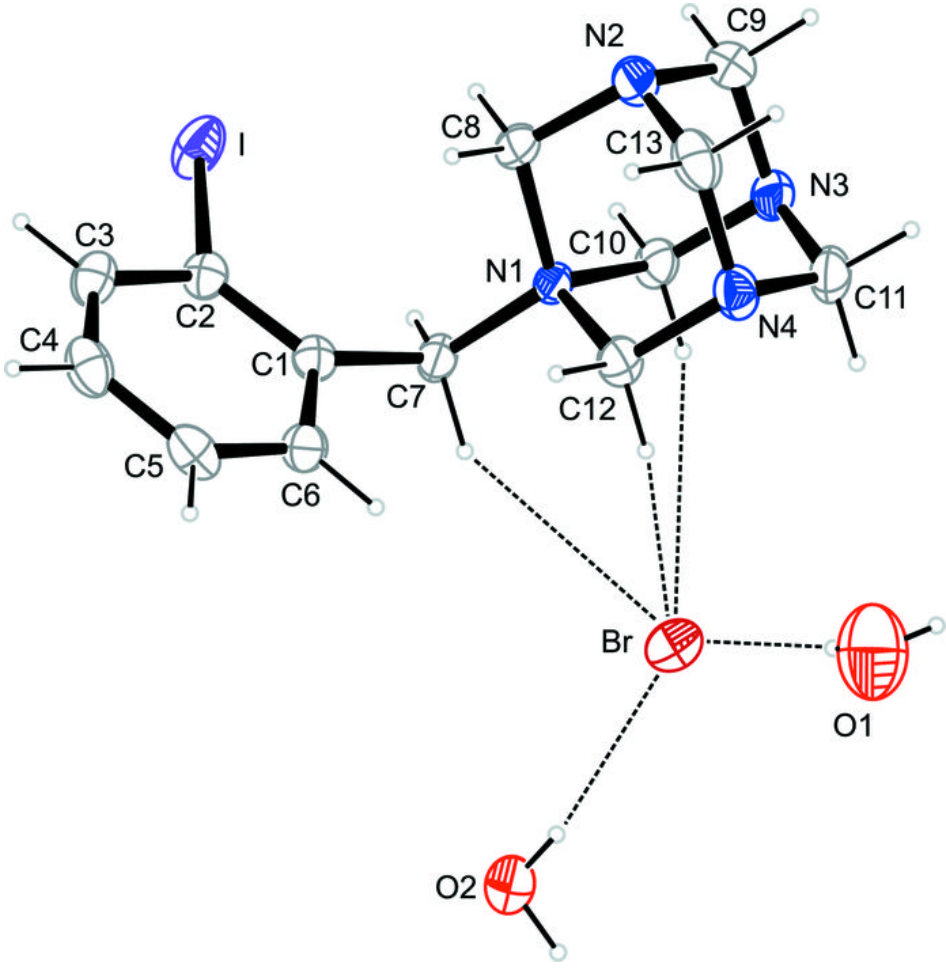

Fig. 2