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Synthesis and crystal structures of 2-bromo-1,3-dimethylimidazolium iodides

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Attempts at direct bromination of 1,3-dimethylimidazolium salts were futile. The title compounds, 2-bromo-1,3-dimethylimidazolium iodide chloroform 0.33-solvate, $C_5H_8BrN_2^+ \cdot I^- \cdot 0.33CHCl_3$, 2-bromo-1,3-dimethylimidazolium iodide dichloromethane hemisolvate, $C_5H_8BrN_2^+ \cdot I^- \cdot 0.5CH_2Cl_2$, and 2-bromo-1,3-dimethylimidazolium iodide hemi(diiodide), $C_5H_8BrN_2^+ \cdot I^- \cdot 0.5I_2$, were obtained by methylation of 2-bromo-1-methylimidazole. They crystallized as $CHCl_3$, CH_2Cl_2 or I_2 solvates/adducts. The Br atom acts as a σ -hole to accept short $C-Br \cdots I$ interactions. $C-H \cdots I$ hydrogen bonds are observed in each structure.

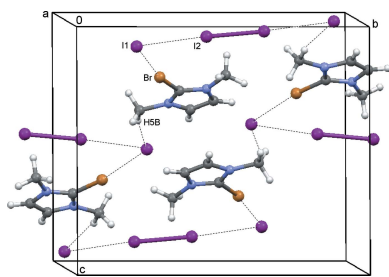
1. Chemical context

Salts containing 2-bromo-1,3-dimethylimidazolium ($C_5H_8N_2Br^+$) cations are the objective of this work. They are presumed to be valuable precursors for substitution reactions. This cation, despite its simplicity, has not yet been described. Since brominations in the 1,3-dimethoxyimidazolium series (Laus *et al.*, 2007) and also bromination of 1-hydroxyimidazole-3-oxide (Laus *et al.*, 2012) gave the respective 2-bromo derivatives, we hoped that in the present case bromination would also yield the desired 2-bromoimidazolium salts. However, on attempted bromination of 1,3-dimethylimidazolium hexafluoridophosphate (Holbrey *et al.*, 2002), no substitution occurred in the 2-position as indicated by NMR. The absence of P–F vibrations in the infrared spectra suggested the formation of a different anion, which was confirmed by X-ray diffraction. Though direct bromination of the quaternary salt did not yield the desired product, it was discovered that an altered sequence of reaction was successful. Thus, the reaction between the 2-lithio derivative of 1-methylimidazole and an equimolar amount of CBr_4 (Boga *et al.*, 2000) or Br_2 (El Borai *et al.*, 1981) gave 2-bromo-1-methylimidazole in good yield, followed by methylation using MeI to afford the desired quaternary salt as an iodide.

Now that the elusive title cation has been secured, further modifications are envisioned, giving access to a plethora of new 2-substituted imidazolium derivatives.

2. Structural commentary

The 2-bromo-1,3-dimethylimidazolium cations and iodide counter-ions crystallize as a $CHCl_3$ 1/3-solvate (**1**) (Fig. 1), a CH_2Cl_2 monosolvate (**2**) (Fig. 2) and an I_2 adduct (**3**) (Fig. 3).



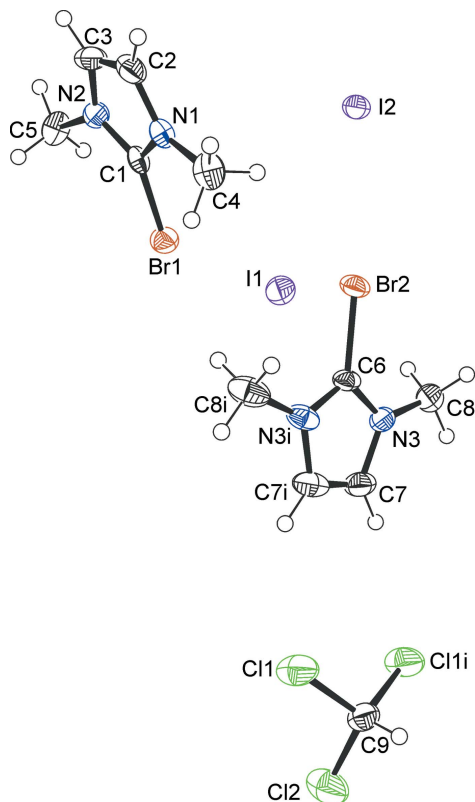


Figure 1
The molecular structure of the chloroform solvate **1**, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (i) $x, 1 - y, z$.]

In every case, the cation is almost planar. In the asymmetric unit of **1**, there are one and a half ion pairs, which are completed by mirror symmetry; the chloroform molecule also lies on a crystallographic mirror plane. In **2**, there are two

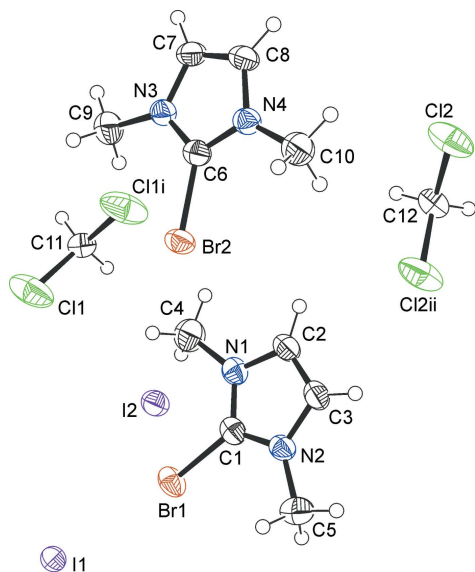
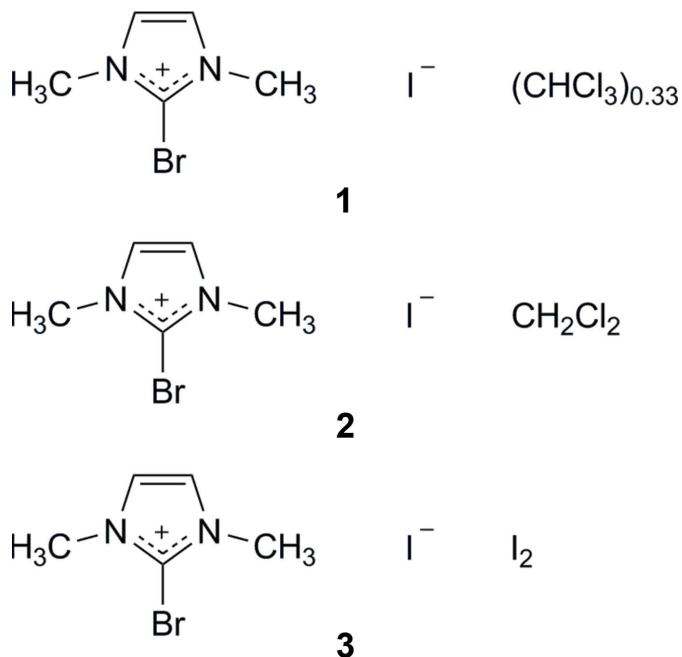


Figure 2
The molecular structure of the iodide **2**, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms. [Symmetry codes: (i) $\frac{1}{2} - x, y, \frac{3}{2} - z$, (ii) $\frac{1}{2} - x, y, \frac{1}{2} - z$.]

anions, two anions and two half-molecules of dichloromethane (both completed by crystallographic twofold symmetry) in the asymmetric unit. In **3**, the iodine molecule is generated by crystallographic inversion symmetry.



3. Supramolecular features

Halogen-halogen interactions constitute the main supramolecular features of the three compounds. The cations in **1** are arranged in a tridimensional array of chains by C—H \cdots I1 interactions. The chloroform molecule bridges these chains by C—H7 \cdots Cl1 and C—H9 \cdots I2 hydrogen bonds (Table 1). Interhalogen Br1 \cdots I2($x, y, -1 + z$) [3.544 (1) Å] and Br2 \cdots I2 [3.546 (2) Å] contacts complete the network (Fig. 4). The respective C—Br \cdots I angles are 173.4 (2) and 173.6 (3)°, indicating an interaction involving the positive end cap (σ -

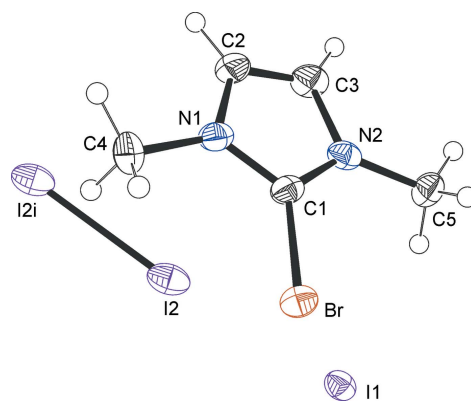


Figure 3
The molecular structure of the iodide **3**, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms. [Symmetry code: (i) $1 - x, 1 - y, -z$.]

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

<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>
C7—H7···Cl1 ⁱ	0.95	2.82	3.623 (7)	142
C8—H8C···I1 ⁱ	0.98	3.02	3.935 (6)	156
C9—H9···I2 ⁱⁱ	1.00	2.77	3.760 (8)	169
C2—H2···I1 ⁱⁱⁱ	0.95	3.01	3.932 (6)	165
C3—H3···I1 ^{iv}	0.95	3.12	3.952 (9)	147

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

hole) of the terminal Br atom (Awwadi *et al.*, 2006; Clark *et al.*, 2007).

This type of interaction is also identified in the structures of compounds **2** and **3**. In the dichloromethane solvate **2**, almost linear halogen interactions Br1···I1 [3.483 (1) Å] and

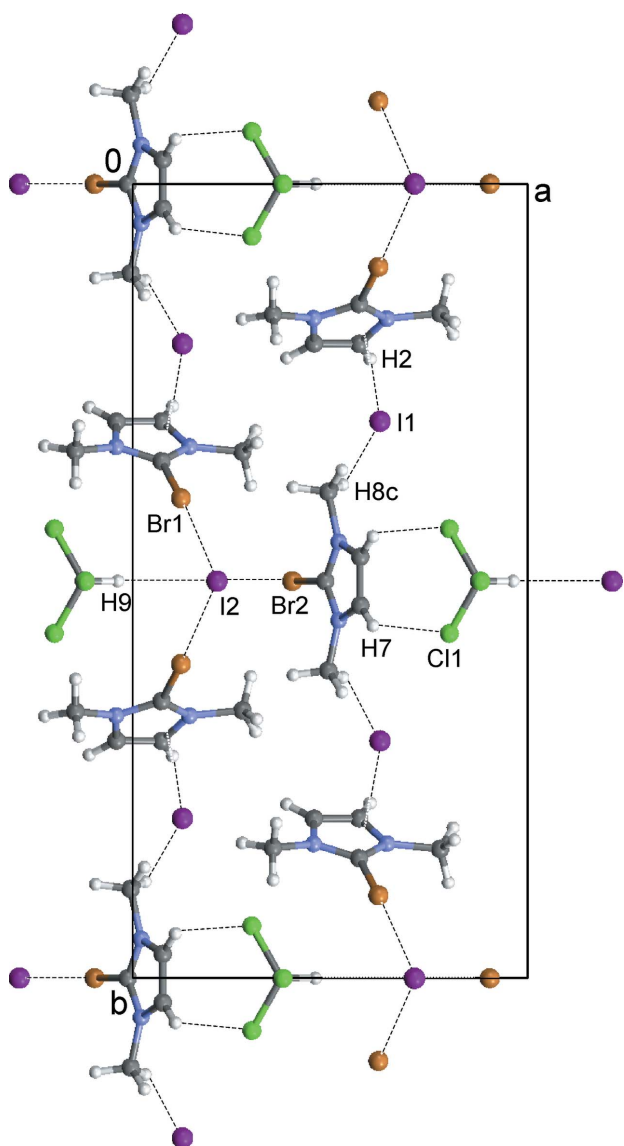


Figure 4
The crystal packing of compound **1** viewed along the *c* axis showing the C—H···Cl and C—H···I hydrogen bonds (see Table 1) and Br···I short contacts as dashed lines.

Table 2
Hydrogen-bond geometry (Å, °) for **2**.

<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>
C11—H11A···I2 ⁱ	0.99	2.86	3.834 (2)	169
C12—H12A···I1 ⁱⁱ	0.99	2.89	3.862 (2)	170

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

Table 3
Hydrogen-bond geometry (Å, °) for **3**.

<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>
C5—H5B···I1 ⁱ	0.98	3.03	3.986 (8)	166

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

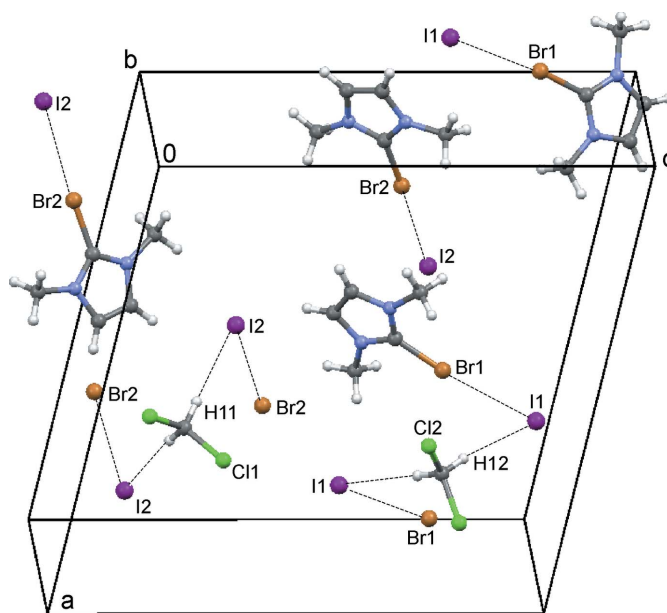


Figure 5
The crystal packing of compound **2** viewed along the *b* axis showing the C—H···I hydrogen bonds involving the solvent (see Table 2) and Br···I short contacts as dashed lines.

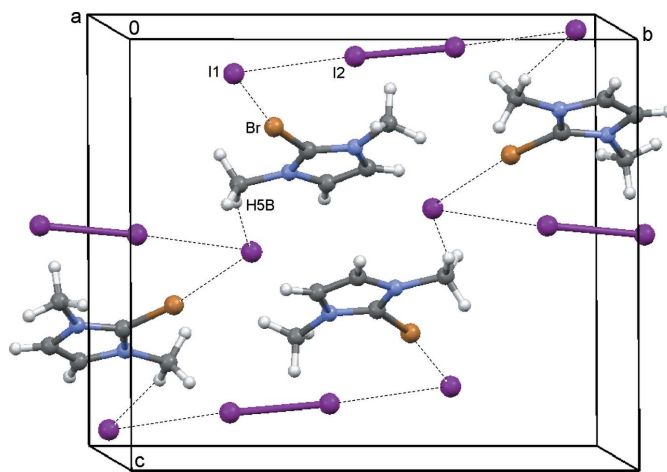


Figure 6
The crystal packing of compound **3** viewed along the *a* axis showing the C—H···I hydrogen bonds (see Table 3) and Br···I and I···I short contacts as dashed lines.

Table 4
Experimental details.

	1	2	3
Crystal data			
Chemical formula	$3C_5H_8BrN_2^+ \cdot 3I^- \cdot CHCl_3$	$2C_5H_8BrN_2^+ \cdot 2I^- \cdot CH_2Cl_2$	$C_5H_8BrN_2^+ \cdot I^- \cdot 0.5I_2$
M_r	1028.20	690.79	429.83
Crystal system, space group	Monoclinic, <i>Cm</i>	Monoclinic, <i>P2₁/n</i>	Monoclinic, <i>P2₁/n</i>
Temperature (K)	173	193	173
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.9135 (14), 21.9492 (10), 6.4529 (6)	16.0223 (8), 8.5334 (4), 16.2881 (8)	6.0861 (4), 14.4773 (11), 12.0303 (7)
β (°)	128.314 (16)	101.590 (1)	97.812 (5)
<i>V</i> (Å ³)	1546.2 (3)	2181.58 (18)	1050.16 (12)
<i>Z</i>	2	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	7.18	6.79	9.74
Crystal size (mm)	0.26 × 0.14 × 0.06	0.18 × 0.16 × 0.14	0.36 × 0.10 × 0.08
Data collection			
Diffractometer	Gemini-R Ultra	Quest Photon 100	Gemini-R Ultra
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2014)	Multi-scan (<i>SADABS</i> ; Bruker, 2014)	Analytical
T_{min} , T_{max}	0.427, 1	0.296, 0.433	0.065, 0.446
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4904, 2522, 2426	62055, 4302, 3952	6287, 1912, 1746
R_{int}	0.026	0.028	0.030
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.602	0.617	0.602
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.018, 0.035, 0.95	0.022, 0.063, 1.09	0.036, 0.080, 1.34
No. of reflections	2522	4302	1912
No. of parameters	151	196	93
No. of restraints	2	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.41, -0.44	1.07, -0.76	0.76, -0.97
Absolute structure	Flack x determined using 961 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)	–	–
Absolute structure parameter	0.038 (8)	–	–

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *CrysAlis PRO* (Oxford Diffraction, 2014), *SIR2002* (Burla *et al.*, 2003), *SHELXTL* (Sheldrick, 2008), *SHELXL2014* and *SHELXL2017* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

Br2···I2 [3.411 (1) Å] exhibit C–Br···I angles of 173.7 (1) and 176.7 (1)°, respectively (Fig. 5). The I1 and I2 anions are linked by hydrogen bonds donated by the solvent molecules (Table 2).

In **3**, a molecular addition compound with iodine (Fig. 6), interactions I1···I2 [3.426 (1) Å] and I2–I2 [related by inversion, bond length 2.826 (1) Å] are present. The I1···Br(1 + *x*, *y*, *z*) [3.499 (1) Å] interaction displays a C–Br···I angle of 168.0 (2)° (Fig. 6) and the iodide anion (I1) accepts a hydrogen bond from the methyl group (Table 3).

4. Database survey

A search of the Cambridge Structural Database (Version 5.38; Groom *et al.*, 2016) for 2-halogeno-1,3-dialkyl or diarylimidazolium salts gave 30 hits. When carbon substituents were allowed in positions 4 and 5, the tally was 34. Of these 64 compounds, there were 11 containing chlorine, 19 bromine and 33 iodine. Closely related imidazolin-2-ylidene–iodine (Kuhn *et al.*, 1993) and imidazolin-2-ylidene–bromine (Kuhn *et al.*, 2004) coordination compounds have been reported.

5. Synthesis and crystallization

Compound 1: A solution of 2-bromo-1-methylimidazole (150 µl, 1.54 mmol) in CHCl₃ (1 ml) was carefully layered over a solution of CH₃I (190 µl, 3.07 mmol) in CHCl₃ (2 ml). The mixture was kept at room temperature and protected from light. After 2 h, the formation of colourless crystals of **1** was observed. The product was collected after seven days at 278 K, yielding 252 mg (48%); m.p. 453 K (decomposition). ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.81 (s, 6H), 7.90 (s, 2H), 8.31 (s) ppm. ¹³C NMR (75 MHz, DMSO-*d*₆): δ 36.8 (2C), 79.3, 123.5, 124.5 (2C) ppm. IR (neat): ν 3066, 2931, 1521, 1240, 1098, 765, 738, 652, 635 cm⁻¹.

Compound 2: A solution of 2-bromo-1-methylimidazole (150 µl, 1.54 mmol) in CH₂Cl₂ (1 ml) was carefully layered over a solution of CH₃I (190 µl, 3.07 mmol) in CH₂Cl₂ (2 ml). The mixture was kept at room temperature and protected from light. After 2 h, the formation of colourless crystals of **2** was observed. The product was collected after 18 h, yielding 145 mg (27%); m.p. 452–453 K (decomposition). ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.81 (s, 6H), 5.75, 7.90 (s, 2H) ppm. ¹³C NMR (75 MHz, DMSO-*d*₆): δ 36.8 (2C), 55.0, 123.3, 124.7

(2C) ppm. IR (neat): ν 3066, 3011, 2944, 1523, 1240, 1101, 779, 728, 696, 635 cm^{-1} .

Compound **3**: The I₂ adduct was obtained as a byproduct of **1** and **2** in the form of brown crystals of **3**; approximate yield 10%; m.p. 451 K (decomposition). ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.81 (s, 6H), 7.89 (d, 2H) ppm IR (neat): ν 3063, 1523, 1226, 739, 634 cm^{-1} .

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. All H atoms were positioned geometrically (C–H = 0.95–1.0 Å) and treated as riding with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$.

Acknowledgements

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supporting information

Acta Cryst. (2018). E74, 497-501 [https://doi.org/10.1107/S2056989018003390]

Synthesis and crystal structures of 2-bromo-1,3-dimethylimidazolium iodides

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Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2014) for (1); *APEX2* (Bruker, 2014) for (2); *CrysAlis PRO* (Oxford Diffraction, 2014) for (3). Cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2014) for (1); *SAINTE* (Bruker, 2014) for (2); *CrysAlis PRO* (Oxford Diffraction, 2014) for (3). Data reduction: *CrysAlis PRO* (Oxford Diffraction, 2014) for (1); *SAINTE* (Bruker, 2014) for (2); *CrysAlis PRO* (Oxford Diffraction, 2014) for (3). Program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003) for (1), (3); *SHELXTL* (Sheldrick, 2008) for (2). Program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015) for (1), (3); *SHELXL2014* (Sheldrick, 2015) for (2). For all structures, molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *Mercury* (Macrae *et al.*, 2008).

2-Bromo-1,3-dimethylimidazolium iodide chloroform 0.33-solvate (1)

Crystal data

$3\text{C}_5\text{H}_8\text{BrN}_2^+ \cdot 3\text{I}^- \cdot \text{CHCl}_3$
 $M_r = 1028.20$
 Monoclinic, *Cm*
 $a = 13.9135$ (14) Å
 $b = 21.9492$ (10) Å
 $c = 6.4529$ (6) Å
 $\beta = 128.314$ (16)°
 $V = 1546.2$ (3) Å³
 $Z = 2$

$F(000) = 956$
 $D_x = 2.208$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 3263 reflections
 $\theta = 3.3\text{--}28.4^\circ$
 $\mu = 7.18$ mm⁻¹
 $T = 173$ K
 Prismatic, colourless
 $0.26 \times 0.14 \times 0.06$ mm

Data collection

Gemini-R Ultra
 diffractometer
 Radiation source: Enhance (Mo) X-ray Source
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Oxford Diffraction, 2014)
 $T_{\min} = 0.427$, $T_{\max} = 1$
 4904 measured reflections

2522 independent reflections
 2426 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\max} = 25.3^\circ$, $\theta_{\min} = 3.4^\circ$
 $h = -16 \rightarrow 13$
 $k = -22 \rightarrow 26$
 $l = -7 \rightarrow 7$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.018$
 $wR(F^2) = 0.035$
 $S = 0.95$

2522 reflections
 151 parameters
 2 restraints
 Hydrogen site location: inferred from
 neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0049P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack x determined using
961 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons *et al.*,
2013)

Absolute structure parameter: 0.038 (8)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.12194 (6)	0.39565 (3)	-0.07927 (14)	0.03269 (15)
C2	0.0731 (6)	0.2987 (3)	0.3714 (13)	0.0354 (15)
H2	0.100922	0.279634	0.532098	0.042*
C3	-0.0414 (6)	0.2992 (3)	0.1483 (14)	0.0353 (16)
H3	-0.110268	0.280970	0.121251	0.042*
C1	0.0731 (5)	0.3488 (2)	0.0790 (11)	0.0246 (14)
N1	0.1435 (4)	0.3305 (2)	0.3284 (9)	0.0284 (11)
C4	0.2778 (6)	0.3372 (3)	0.5169 (14)	0.0405 (17)
H4A	0.313345	0.325857	0.430407	0.061*
H4B	0.310751	0.310582	0.669397	0.061*
H4C	0.298588	0.379637	0.576266	0.061*
N2	-0.0420 (4)	0.3308 (2)	-0.0364 (10)	0.0283 (12)
C5	-0.1452 (5)	0.3358 (3)	-0.3220 (12)	0.0353 (15)
H5A	-0.144701	0.376365	-0.385102	0.053*
H5B	-0.222661	0.329753	-0.351574	0.053*
H5C	-0.136654	0.304732	-0.418510	0.053*
I1	0.62590 (4)	0.29885 (2)	0.94514 (6)	0.02823 (10)
I2	0.21304 (5)	0.500000	0.65285 (9)	0.02890 (13)
Br2	0.40367 (7)	0.500000	0.46275 (15)	0.0313 (2)
C8	0.4948 (7)	0.6124 (3)	0.2888 (14)	0.0462 (18)
H8A	0.405961	0.618288	0.177487	0.069*
H8B	0.529339	0.639748	0.230124	0.069*
H8C	0.531481	0.621371	0.472955	0.069*
N3	0.5211 (4)	0.5494 (2)	0.2675 (9)	0.0315 (12)
C6	0.4874 (7)	0.500000	0.3242 (15)	0.0269 (19)
C7	0.5758 (6)	0.5305 (3)	0.1602 (12)	0.0399 (16)
H7	0.607822	0.556086	0.097268	0.048*
C9	0.8764 (8)	0.500000	0.0794 (19)	0.038 (2)
H9	0.962304	0.500000	0.247965	0.045*
Cl1	0.8016 (2)	0.43407 (7)	0.0686 (5)	0.0569 (5)
Cl2	0.8806 (3)	0.500000	-0.1852 (6)	0.0636 (8)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0383 (3)	0.0293 (3)	0.0368 (3)	-0.0004 (3)	0.0265 (3)	0.0013 (3)
C2	0.052 (4)	0.032 (3)	0.033 (4)	0.001 (3)	0.031 (4)	-0.001 (3)
C3	0.046 (4)	0.031 (4)	0.050 (4)	0.002 (3)	0.040 (4)	0.003 (3)
C1	0.029 (3)	0.019 (3)	0.023 (3)	0.003 (3)	0.015 (3)	-0.003 (3)
N1	0.032 (3)	0.025 (3)	0.026 (3)	0.003 (2)	0.017 (2)	-0.003 (2)
C4	0.043 (4)	0.032 (3)	0.038 (4)	0.006 (3)	0.021 (4)	0.002 (3)
N2	0.028 (3)	0.024 (3)	0.035 (3)	0.002 (2)	0.021 (2)	-0.001 (2)
C5	0.022 (3)	0.033 (3)	0.031 (4)	0.003 (3)	0.007 (3)	0.005 (3)
I1	0.03254 (19)	0.02663 (18)	0.0289 (2)	-0.00359 (18)	0.02071 (17)	0.00012 (18)
I2	0.0286 (3)	0.0344 (3)	0.0280 (3)	0.000	0.0196 (3)	0.000
Br2	0.0373 (5)	0.0385 (5)	0.0292 (5)	0.000	0.0262 (4)	0.000
C8	0.059 (5)	0.044 (4)	0.052 (5)	-0.021 (3)	0.043 (4)	-0.015 (3)
N3	0.028 (3)	0.045 (3)	0.023 (3)	-0.011 (2)	0.016 (2)	-0.005 (2)
C6	0.021 (4)	0.044 (5)	0.018 (4)	0.000	0.013 (4)	0.000
C7	0.031 (3)	0.064 (4)	0.031 (4)	-0.008 (3)	0.022 (3)	-0.004 (3)
C9	0.034 (5)	0.031 (5)	0.053 (6)	0.000	0.030 (5)	0.000
Cl1	0.0748 (12)	0.0285 (7)	0.1059 (15)	0.0015 (10)	0.0751 (12)	0.0040 (11)
Cl2	0.102 (2)	0.0363 (14)	0.097 (2)	0.000	0.084 (2)	0.000

Geometric parameters (\AA , $^\circ$)

Br1—C1	1.850 (6)	C5—H5C	0.9800
C2—C3	1.329 (9)	Br2—C6	1.857 (8)
C2—N1	1.364 (8)	C8—N3	1.457 (8)
C2—H2	0.9500	C8—H8A	0.9800
C3—N2	1.374 (8)	C8—H8B	0.9800
C3—H3	0.9500	C8—H8C	0.9800
C1—N1	1.325 (7)	N3—C6	1.321 (6)
C1—N2	1.340 (7)	N3—C7	1.372 (7)
N1—C4	1.475 (8)	C7—C7 ⁱ	1.340 (13)
C4—H4A	0.9800	C7—H7	0.9500
C4—H4B	0.9800	C9—Cl2	1.744 (9)
C4—H4C	0.9800	C9—Cl1 ⁱ	1.759 (5)
N2—C5	1.480 (8)	C9—Cl1	1.759 (5)
C5—H5A	0.9800	C9—H9	1.0000
C5—H5B	0.9800		
C3—C2—N1	107.7 (6)	N2—C5—H5C	109.5
C3—C2—H2	126.1	H5A—C5—H5C	109.5
N1—C2—H2	126.1	H5B—C5—H5C	109.5
C2—C3—N2	107.6 (6)	N3—C8—H8A	109.5
C2—C3—H3	126.2	N3—C8—H8B	109.5
N2—C3—H3	126.2	H8A—C8—H8B	109.5
N1—C1—N2	108.3 (5)	N3—C8—H8C	109.5
N1—C1—Br1	126.2 (4)	H8A—C8—H8C	109.5

N2—C1—Br1	125.3 (4)	H8B—C8—H8C	109.5
C1—N1—C2	108.6 (5)	C6—N3—C7	107.1 (5)
C1—N1—C4	125.2 (5)	C6—N3—C8	126.8 (5)
C2—N1—C4	125.8 (5)	C7—N3—C8	125.8 (5)
N1—C4—H4A	109.5	N3 ⁱ —C6—N3	110.5 (7)
N1—C4—H4B	109.5	N3 ⁱ —C6—Br2	124.8 (4)
H4A—C4—H4B	109.5	N3—C6—Br2	124.8 (4)
N1—C4—H4C	109.5	C7 ⁱ —C7—N3	107.6 (4)
H4A—C4—H4C	109.5	C7 ⁱ —C7—H7	126.2
H4B—C4—H4C	109.5	N3—C7—H7	126.2
C1—N2—C3	107.7 (5)	Cl2—C9—Cl1 ⁱ	109.8 (4)
C1—N2—C5	125.0 (5)	Cl2—C9—Cl1	109.8 (4)
C3—N2—C5	126.6 (5)	Cl1 ⁱ —C9—Cl1	110.8 (5)
N2—C5—H5A	109.5	Cl2—C9—H9	108.8
N2—C5—H5B	109.5	Cl1 ⁱ —C9—H9	108.8
H5A—C5—H5B	109.5	Cl1—C9—H9	108.8
N1—C2—C3—N2	0.8 (7)	Br1—C1—N2—C5	11.5 (8)
N2—C1—N1—C2	1.8 (6)	C2—C3—N2—C1	0.3 (7)
Br1—C1—N1—C2	178.1 (4)	C2—C3—N2—C5	171.0 (5)
N2—C1—N1—C4	175.1 (5)	C7—N3—C6—N3 ⁱ	2.1 (8)
Br1—C1—N1—C4	-8.6 (8)	C8—N3—C6—N3 ⁱ	176.4 (4)
C3—C2—N1—C1	-1.6 (7)	C7—N3—C6—Br2	-177.6 (5)
C3—C2—N1—C4	-174.9 (6)	C8—N3—C6—Br2	-3.3 (10)
N1—C1—N2—C3	-1.3 (6)	C6—N3—C7—C7 ⁱ	-1.3 (5)
Br1—C1—N2—C3	-177.6 (4)	C8—N3—C7—C7 ⁱ	-175.7 (5)
N1—C1—N2—C5	-172.2 (5)		

Symmetry code: (i) $x, -y+1, z$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 \cdots Cl1 ⁱ	0.95	2.82	3.623 (7)	142
C8—H8C \cdots I1 ⁱ	0.98	3.02	3.935 (6)	156
C9—H9 \cdots I2 ⁱⁱ	1.00	2.77	3.760 (8)	169
C2—H2 \cdots I1 ⁱⁱⁱ	0.95	3.01	3.932 (6)	165
C3—H3 \cdots I1 ^{iv}	0.95	3.12	3.952 (9)	147

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

2-Bromo-1,3-dimethylimidazolium iodide dichloromethane hemisolvate (2)

Crystal data

$2\text{C}_3\text{H}_8\text{BrN}_2^+ \cdot 2\text{I}^- \cdot \text{CH}_2\text{Cl}_2$
 $M_r = 690.79$
 Monoclinic, $P2_1/n$
 $a = 16.0223$ (8) \AA
 $b = 8.5334$ (4) \AA
 $c = 16.2881$ (8) \AA
 $\beta = 101.590$ (1) $^\circ$

$V = 2181.58$ (18) \AA^3
 $Z = 4$
 $F(000) = 1288$
 $D_x = 2.103$ Mg m^{-3}
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA
 Cell parameters from 9539 reflections
 $\theta = 2.5\text{--}26.8^\circ$

$\mu = 6.79 \text{ mm}^{-1}$
 $T = 193 \text{ K}$

Prism, colourless
 $0.18 \times 0.16 \times 0.14 \text{ mm}$

Data collection

Quest Photon 100
 diffractometer
 Radiation source: Incoatec Microfocus
 Multi layered optics monochromator
 Detector resolution: $10.4 \text{ pixels mm}^{-1}$
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2014)
 $T_{\min} = 0.296$, $T_{\max} = 0.433$

62055 measured reflections
 4302 independent reflections
 3952 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.4^\circ$
 $h = -19 \rightarrow 19$
 $k = -10 \rightarrow 10$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.063$
 $S = 1.09$
 4302 reflections
 196 parameters
 0 restraints
 Hydrogen site location: inferred from
 neighbouring sites

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 2.6592P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.07 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.76 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL2014
 (Sheldrick, 2015),
 $F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.00234 (11)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
I1	0.67871 (2)	0.51565 (2)	0.93259 (2)	0.03524 (8)	
I2	0.43487 (2)	1.00139 (2)	0.67926 (2)	0.03507 (8)	
Br1	0.58435 (2)	0.60778 (4)	0.72654 (2)	0.04235 (10)	
Br2	0.23431 (2)	0.90371 (4)	0.57720 (2)	0.03775 (10)	
N1	0.44318 (17)	0.5987 (3)	0.58815 (16)	0.0365 (6)	
N2	0.54830 (15)	0.7333 (3)	0.55936 (15)	0.0345 (5)	
N3	0.06441 (15)	0.7732 (3)	0.54179 (15)	0.0344 (5)	
N4	0.09163 (16)	0.9098 (3)	0.43839 (16)	0.0347 (5)	
C1	0.52208 (19)	0.6502 (4)	0.61831 (18)	0.0347 (6)	
C2	0.4178 (2)	0.6538 (4)	0.50716 (19)	0.0387 (7)	
H2	0.3644	0.6356	0.4708	0.046*	
C3	0.4831 (2)	0.7381 (4)	0.48965 (19)	0.0389 (7)	
H3	0.4841	0.7913	0.4386	0.047*	
C4	0.3892 (3)	0.5077 (5)	0.6337 (3)	0.0550 (10)	
H4A	0.4211	0.4167	0.6602	0.083*	
H4B	0.3382	0.4720	0.5945	0.083*	
H4C	0.3724	0.5737	0.6769	0.083*	

C5	0.6292 (2)	0.8180 (4)	0.5680 (2)	0.0494 (8)	
H5A	0.6345	0.8927	0.6145	0.074*	
H5B	0.6305	0.8747	0.5160	0.074*	
H5C	0.6765	0.7433	0.5793	0.074*	
C6	0.12335 (18)	0.8585 (3)	0.51581 (19)	0.0334 (6)	
C7	-0.00661 (19)	0.7668 (4)	0.4784 (2)	0.0383 (7)	
H7	-0.0580	0.7122	0.4797	0.046*	
C8	0.01002 (19)	0.8519 (4)	0.4140 (2)	0.0387 (7)	
H8	-0.0274	0.8689	0.3617	0.046*	
C9	0.0746 (2)	0.6917 (5)	0.6219 (2)	0.0503 (8)	
H9A	0.1254	0.6251	0.6298	0.075*	
H9B	0.0243	0.6266	0.6224	0.075*	
H9C	0.0809	0.7686	0.6674	0.075*	
C10	0.1357 (3)	1.0097 (5)	0.3880 (3)	0.0530 (10)	
H10A	0.1510	1.1091	0.4172	0.080*	
H10B	0.0982	1.0301	0.3336	0.080*	
H10C	0.1875	0.9569	0.3792	0.080*	
C11	0.2500	0.2120 (5)	0.7500	0.0409 (10)	
H11A	0.2931	0.1437	0.7324	0.049*	0.5
H11B	0.2069	0.1436	0.7676	0.049*	0.5
C11	0.29912 (8)	0.32404 (14)	0.83510 (8)	0.0829 (4)	
C12	0.2500	0.7005 (5)	0.2500	0.0416 (10)	
H12A	0.2689	0.6322	0.2081	0.050*	0.5
H12B	0.2311	0.6322	0.2919	0.050*	0.5
Cl2	0.16400 (7)	0.81502 (12)	0.20000 (7)	0.0695 (3)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.03410 (12)	0.03872 (13)	0.03155 (12)	0.00227 (8)	0.00338 (9)	0.00373 (7)
I2	0.03022 (12)	0.04024 (13)	0.03290 (12)	0.00221 (7)	0.00191 (8)	-0.00410 (7)
Br1	0.04481 (19)	0.04589 (19)	0.03212 (17)	0.01593 (14)	-0.00236 (13)	0.00018 (13)
Br2	0.03094 (16)	0.04058 (18)	0.03833 (17)	-0.00138 (12)	-0.00111 (12)	-0.01062 (12)
N1	0.0399 (14)	0.0358 (14)	0.0331 (13)	0.0018 (11)	0.0057 (11)	-0.0024 (10)
N2	0.0315 (12)	0.0374 (13)	0.0331 (13)	0.0056 (10)	0.0025 (10)	-0.0051 (11)
N3	0.0320 (13)	0.0379 (13)	0.0326 (13)	0.0021 (10)	0.0050 (10)	-0.0042 (11)
N4	0.0326 (13)	0.0356 (13)	0.0349 (13)	0.0003 (10)	0.0046 (10)	-0.0052 (10)
C1	0.0382 (16)	0.0325 (15)	0.0317 (15)	0.0085 (12)	0.0032 (12)	-0.0038 (12)
C2	0.0384 (16)	0.0429 (17)	0.0315 (15)	0.0033 (14)	-0.0004 (12)	-0.0052 (13)
C3	0.0413 (17)	0.0454 (18)	0.0282 (15)	0.0076 (14)	0.0026 (12)	-0.0018 (13)
C4	0.058 (2)	0.057 (2)	0.051 (2)	-0.0060 (17)	0.0125 (19)	0.0077 (17)
C5	0.0364 (17)	0.054 (2)	0.055 (2)	-0.0011 (15)	0.0048 (15)	-0.0014 (17)
C6	0.0317 (14)	0.0315 (14)	0.0356 (15)	0.0037 (12)	0.0032 (12)	-0.0082 (12)
C7	0.0270 (14)	0.0438 (18)	0.0428 (17)	-0.0011 (12)	0.0039 (12)	-0.0061 (14)
C8	0.0306 (15)	0.0435 (17)	0.0383 (16)	0.0011 (13)	-0.0019 (12)	-0.0042 (14)
C9	0.054 (2)	0.058 (2)	0.0390 (18)	-0.0011 (17)	0.0089 (16)	0.0067 (16)
C10	0.051 (2)	0.061 (2)	0.046 (2)	-0.0109 (17)	0.0077 (17)	0.0060 (16)
C11	0.034 (2)	0.042 (2)	0.046 (3)	0.000	0.0059 (19)	0.000

Cl1	0.0724 (7)	0.0690 (7)	0.0889 (8)	0.0138 (6)	-0.0277 (6)	-0.0346 (6)
C12	0.044 (2)	0.040 (2)	0.037 (2)	0.000	-0.0023 (19)	0.000
Cl2	0.0684 (6)	0.0583 (6)	0.0657 (6)	0.0183 (5)	-0.0249 (5)	-0.0070 (5)

Geometric parameters (Å, °)

Br1—C1	1.878 (3)	C4—H4C	0.9800
Br2—C6	1.896 (3)	C5—H5A	0.9800
N1—C1	1.335 (4)	C5—H5B	0.9800
N1—C2	1.382 (4)	C5—H5C	0.9800
N1—C4	1.469 (5)	C7—C8	1.345 (5)
N2—C1	1.328 (4)	C7—H7	0.9500
N2—C3	1.380 (4)	C8—H8	0.9500
N2—C5	1.465 (4)	C9—H9A	0.9800
N3—C6	1.327 (4)	C9—H9B	0.9800
N3—C7	1.376 (4)	C9—H9C	0.9800
N3—C9	1.459 (4)	C10—H10A	0.9800
N4—C6	1.335 (4)	C10—H10B	0.9800
N4—C8	1.380 (4)	C10—H10C	0.9800
N4—C10	1.460 (4)	C11—Cl1	1.737 (3)
C2—C3	1.347 (5)	C11—H11A	0.9900
C2—H2	0.9500	C11—H11B	0.9900
C3—H3	0.9500	C12—Cl2	1.751 (3)
C4—H4A	0.9800	C12—H12A	0.9900
C4—H4B	0.9800	C12—H12B	0.9900
C1—N1—C2	108.3 (3)	N3—C6—N4	108.7 (3)
C1—N1—C4	126.7 (3)	N3—C6—Br2	126.5 (2)
C2—N1—C4	124.8 (3)	N4—C6—Br2	124.7 (2)
C1—N2—C3	108.3 (3)	C8—C7—N3	107.5 (3)
C1—N2—C5	126.6 (3)	C8—C7—H7	126.3
C3—N2—C5	124.9 (3)	N3—C7—H7	126.3
C6—N3—C7	108.5 (3)	C7—C8—N4	107.1 (3)
C6—N3—C9	126.0 (3)	C7—C8—H8	126.4
C7—N3—C9	125.4 (3)	N4—C8—H8	126.4
C6—N4—C8	108.2 (3)	N3—C9—H9A	109.5
C6—N4—C10	126.0 (3)	N3—C9—H9B	109.5
C8—N4—C10	125.8 (3)	H9A—C9—H9B	109.5
N2—C1—N1	108.9 (3)	N3—C9—H9C	109.5
N2—C1—Br1	126.6 (2)	H9A—C9—H9C	109.5
N1—C1—Br1	124.5 (2)	H9B—C9—H9C	109.5
C3—C2—N1	106.9 (3)	N4—C10—H10A	109.5
C3—C2—H2	126.5	N4—C10—H10B	109.5
N1—C2—H2	126.5	H10A—C10—H10B	109.5
C2—C3—N2	107.6 (3)	N4—C10—H10C	109.5
C2—C3—H3	126.2	H10A—C10—H10C	109.5
N2—C3—H3	126.2	H10B—C10—H10C	109.5
N1—C4—H4A	109.5	Cl1 ⁱ —C11—Cl1	113.2 (3)

N1—C4—H4B	109.5	C11 ⁱ —C11—H11A	108.9
H4A—C4—H4B	109.5	C11—C11—H11A	108.9
N1—C4—H4C	109.5	C11 ⁱ —C11—H11B	108.9
H4A—C4—H4C	109.5	C11—C11—H11B	108.9
H4B—C4—H4C	109.5	H11A—C11—H11B	107.7
N2—C5—H5A	109.5	C12—C12—C12 ⁱⁱ	112.1 (3)
N2—C5—H5B	109.5	C12—C12—H12A	109.2
H5A—C5—H5B	109.5	C12 ⁱⁱ —C12—H12A	109.2
N2—C5—H5C	109.5	C12—C12—H12B	109.2
H5A—C5—H5C	109.5	C12 ⁱⁱ —C12—H12B	109.2
H5B—C5—H5C	109.5	H12A—C12—H12B	107.9
C3—N2—C1—N1	1.4 (3)	C7—N3—C6—N4	1.4 (3)
C5—N2—C1—N1	176.4 (3)	C9—N3—C6—N4	177.9 (3)
C3—N2—C1—Br1	-179.2 (2)	C7—N3—C6—Br2	-179.2 (2)
C5—N2—C1—Br1	-4.3 (4)	C9—N3—C6—Br2	-2.6 (4)
C2—N1—C1—N2	-1.1 (3)	C8—N4—C6—N3	-1.2 (3)
C4—N1—C1—N2	-177.4 (3)	C10—N4—C6—N3	178.9 (3)
C2—N1—C1—Br1	179.5 (2)	C8—N4—C6—Br2	179.3 (2)
C4—N1—C1—Br1	3.2 (4)	C10—N4—C6—Br2	-0.5 (4)
C1—N1—C2—C3	0.3 (3)	C6—N3—C7—C8	-1.0 (3)
C4—N1—C2—C3	176.7 (3)	C9—N3—C7—C8	-177.5 (3)
N1—C2—C3—N2	0.5 (3)	N3—C7—C8—N4	0.2 (4)
C1—N2—C3—C2	-1.2 (3)	C6—N4—C8—C7	0.6 (3)
C5—N2—C3—C2	-176.2 (3)	C10—N4—C8—C7	-179.6 (3)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C11—H11A \cdots I2 ⁱⁱⁱ	0.99	2.86	3.834 (2)	169
C12—H12A \cdots I1 ^{iv}	0.99	2.89	3.862 (2)	170

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

2-Bromo-1,3-dimethylimidazolium iodide hemi(diiodide) (3)

Crystal data

$C_5H_8BrN_2^+ \cdot I^- \cdot 0.5I_2$
 $M_r = 429.83$
 Monoclinic, $P2_1/n$
 $a = 6.0861$ (4) \AA
 $b = 14.4773$ (11) \AA
 $c = 12.0303$ (7) \AA
 $\beta = 97.812$ (5) $^\circ$
 $V = 1050.16$ (12) \AA^3
 $Z = 4$

$F(000) = 772$
 $D_x = 2.719$ Mg m^{-3}
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA
 Cell parameters from 3142 reflections
 $\theta = 3.3\text{--}27.6^\circ$
 $\mu = 9.74$ mm^{-1}
 $T = 173$ K
 Lath shaped, red-brown
 $0.36 \times 0.10 \times 0.08$ mm

Data collection

Gemini-R Ultra
diffractometer

ω scans

Absorption correction: analytical

$T_{\min} = 0.065$, $T_{\max} = 0.446$

6287 measured reflections

1912 independent reflections

1746 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 3.3^\circ$

$h = -6 \rightarrow 7$

$k = -17 \rightarrow 13$

$l = -14 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.080$

$S = 1.34$

1912 reflections

93 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0102P)^2 + 8.6812P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.97 \text{ e } \text{\AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.59338 (9)	0.17085 (4)	0.05590 (4)	0.02835 (16)
I2	0.51595 (9)	0.40299 (5)	0.01140 (5)	0.03726 (18)
Br	0.04393 (13)	0.28681 (6)	0.20566 (7)	0.0274 (2)
N2	0.4459 (11)	0.3501 (5)	0.3339 (5)	0.0246 (15)
N1	0.2322 (11)	0.4618 (5)	0.2630 (5)	0.0269 (15)
C1	0.2538 (13)	0.3703 (6)	0.2721 (6)	0.0236 (17)
C4	0.0471 (14)	0.5121 (6)	0.1973 (7)	0.034 (2)
H4A	0.076771	0.518793	0.119610	0.051*
H4B	0.032629	0.573356	0.230131	0.051*
H4C	-0.090958	0.477458	0.198377	0.051*
C3	0.5500 (14)	0.4327 (6)	0.3649 (7)	0.031 (2)
H3	0.690601	0.439536	0.409197	0.037*
C5	0.5298 (14)	0.2578 (6)	0.3667 (7)	0.0293 (19)
H5A	0.422473	0.225817	0.406887	0.044*
H5B	0.671533	0.263396	0.415747	0.044*
H5C	0.551338	0.222595	0.299519	0.044*
C2	0.4191 (15)	0.5011 (6)	0.3217 (7)	0.032 (2)
H2	0.449093	0.565310	0.329958	0.039*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0248 (3)	0.0341 (3)	0.0254 (3)	-0.0032 (2)	0.0007 (2)	0.0003 (2)
I2	0.0282 (3)	0.0522 (4)	0.0320 (3)	-0.0097 (3)	0.0062 (2)	-0.0096 (3)
Br	0.0267 (4)	0.0248 (5)	0.0306 (4)	-0.0047 (3)	0.0028 (3)	-0.0036 (3)
N2	0.025 (3)	0.030 (4)	0.020 (3)	-0.005 (3)	0.005 (3)	-0.003 (3)
N1	0.030 (4)	0.024 (4)	0.026 (4)	0.001 (3)	0.002 (3)	-0.006 (3)
C1	0.031 (4)	0.021 (4)	0.021 (4)	-0.005 (4)	0.011 (3)	-0.003 (3)
C4	0.035 (5)	0.033 (5)	0.033 (5)	0.008 (4)	-0.003 (4)	0.007 (4)
C3	0.024 (4)	0.034 (5)	0.034 (5)	-0.013 (4)	0.002 (3)	-0.003 (4)
C5	0.029 (4)	0.023 (5)	0.036 (5)	0.002 (4)	0.003 (4)	0.001 (3)
C2	0.041 (5)	0.023 (5)	0.031 (5)	-0.008 (4)	0.002 (4)	-0.003 (4)

Geometric parameters (\AA , $^\circ$)

I2—I2 ⁱ	2.8265 (14)	C4—H4B	0.9800
Br—C1	1.858 (8)	C4—H4C	0.9800
N2—C1	1.330 (10)	C3—C2	1.331 (12)
N2—C3	1.380 (11)	C3—H3	0.9500
N2—C5	1.465 (11)	C5—H5A	0.9800
N1—C1	1.335 (11)	C5—H5B	0.9800
N1—C2	1.377 (11)	C5—H5C	0.9800
N1—C4	1.477 (10)	C2—H2	0.9500
C4—H4A	0.9800		
C1—N2—C3	107.3 (7)	H4B—C4—H4C	109.5
C1—N2—C5	126.7 (7)	C2—C3—N2	108.1 (7)
C3—N2—C5	125.9 (7)	C2—C3—H3	125.9
C1—N1—C2	107.6 (7)	N2—C3—H3	125.9
C1—N1—C4	126.3 (7)	N2—C5—H5A	109.5
C2—N1—C4	126.0 (7)	N2—C5—H5B	109.5
N2—C1—N1	109.4 (7)	H5A—C5—H5B	109.5
N2—C1—Br	126.8 (6)	N2—C5—H5C	109.5
N1—C1—Br	123.8 (6)	H5A—C5—H5C	109.5
N1—C4—H4A	109.5	H5B—C5—H5C	109.5
N1—C4—H4B	109.5	C3—C2—N1	107.6 (8)
H4A—C4—H4B	109.5	C3—C2—H2	126.2
N1—C4—H4C	109.5	N1—C2—H2	126.2
H4A—C4—H4C	109.5		
C3—N2—C1—N1	-0.1 (8)	C4—N1—C1—Br	-2.2 (11)
C5—N2—C1—N1	178.0 (7)	C1—N2—C3—C2	0.2 (9)
C3—N2—C1—Br	179.2 (6)	C5—N2—C3—C2	-177.9 (7)
C5—N2—C1—Br	-2.7 (11)	N2—C3—C2—N1	-0.2 (10)
C2—N1—C1—N2	0.0 (9)	C1—N1—C2—C3	0.2 (9)

C4—N1—C1—N2	177.1 (7)	C4—N1—C2—C3	-177.0 (8)
C2—N1—C1—Br	-179.4 (6)		

Symmetry code: (i) $-x+1, -y+1, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C5—H5B \cdots I1 ⁱⁱ	0.98	3.03	3.986 (8)	166

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