2-Hydroxymethyl-1,3-dimethyl-1H-benzimidazol-3-ium iodide

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In the cation of the title compound, $C_{10}H_{13}N_2O^+\cdot I^-$, all non-H atoms, with the exception of the O atom, are essentially coplanar, with a maximum deviation of 0.04 (1) Å. In the crystal, the cations and anions are arranged in layers parallel to (100). The cations are connected to the anions via an O—H···I hydrogen bond and there are significant π–π stacking interactions between cation layers, with centroid–centroid distances in the range 3.606 (5)–3.630 (5) Å. A weak intramolecular C—H···O hydrogen bond is also observed. The crystal studied was an inversion twin with refined components of 0.52 (5) and 0.48 (5).

Related literature

For applications of this class of compounds, see: Tonelli *et al.* (2010); Preston (1974); Hazelton *et al.* (1995); Kucukguzel *et al.* (2001); Islam *et al.* (1991); Li *et al.* (2003); Abboud *et al.* (2006). For our previous work on imidazole derivatives, see: Bahnous *et al.* (2012); Zama *et al.* (2013); Chelghoum *et al.* (2011).
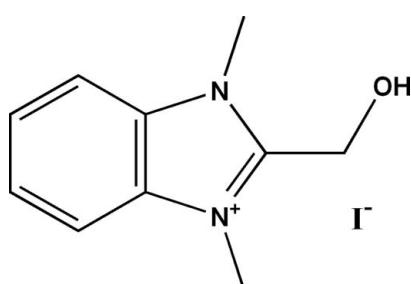
Experimental

Crystal data

$C_{10}H_{13}N_2O^+\cdot I^-$	V = 1127.4 (2) Å^3
M_r = 304.12	Z = 4
Orthorhombic, $P2_1nb$	Mo $K\alpha$ radiation
a = 6.5690 (7) Å	μ = 2.81 mm^{-1}
b = 10.1342 (10) Å	T = 150 K
c = 16.9573 (19) Å	0.14 × 0.13 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer	10018 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002) T_{min} = 0.647, T_{max} = 0.747	4002 independent reflections
	3243 reflections with $I > 2\sigma(I)$
	R_{int} = 0.02

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027	$\Delta\rho_{max}$ = 1.34 e Å^{-3}
$wR(F^2)$ = 0.068	$\Delta\rho_{min}$ = −0.72 e Å^{-3}
S = 1.05	Absolute structure: Flack (1983), 1518 Friedel pairs
4002 reflections	Absolute structure parameter: 0.48 (5)
131 parameters	
1 restraint	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···I1^i	0.82	2.66	3.473 (3)	171
C5—H5A···O1	0.96	2.52	3.170 (5)	125

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CRYSCAL* (T. Roisnel, local program).

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supporting information

2-Hydroxymethyl-1,3-dimethyl-1*H*-benzimidazol-3-ium iodide

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S1. Comment

Benzimidazole, is isosteric with indole and purine nuclei, which are present in a number of fundamental cellular components and bioactive compounds. This structural similarity means the benzimidazole molecule is endowed with a variety of interesting biological properties (Kucukguzel, *et al.*, 2001; Islam *et al.*, 1991; Tonelli *et al.*, 2010). Some of these compounds are marketed as antifungal, (Preston, 1974), antihelmintic, (Hazelton *et al.*, 1995). Furthermore, benzimidazole derivatives can be also used as epoxy resin curing agents, catalysts, and metallic surface treatment agents (Li *et al.*, 2003; Abboud *et al.*, 2006). In previous work, we have reported the synthesis and structure determination of some new heterocyclic compounds bearing an imidazole unit (Bahnous *et al.*, 2012; Zama *et al.*, 2013; Chelghoum *et al.*, 2011). Herein, we describe the synthesis and the structure determination of 1,3-dimethyl-2-hydroxymethyl-benzimidazolium iodide (I) resulting from the quaternization reaction of 1-methyl-2-hydroxymethylbenzimidazole with methyl iodide.

The molecular structure of (I) is shown in Fig. 1. The asymmetric unit contains a 1,3-dimethyl-2-hydroxymethyl-benzimidazolium cation and an iodide anion. All non-H atoms in the cation, with the exception of the O atom, are essentially co-planar with a maximum deviation of 0.04 (1)Å for N1. In the crystal, the cations and anions are arranged in layers parallel to (100) (Fig. 2). The cations are hydrogen bonded to the anions via an O—H⋯I hydrogen bond and there are significant π—π stacking interactions between cation layers with centroid-centroid distances in the range 3.606 (5) - 3.630 (5)Å.

S2. Experimental

To a solution of 1-methyl-2-hydroxymethylbenzimidazole derivatives (10 mmol) in 20 ml of acetonitrile, was added 30 mmol of methyl iodide. The reaction mixture was refluxed. When the reaction was over (TLC), the solvent volume was reduced and the crude product was then filtered off and washed with cold acetonitrile. Suitable crystals for X-ray analysis were obtained by slow evaporation of a water solution of (I).

S3. Refinement

Approximate positions for all H atoms were first obtained from the difference electron density map. However, the H atoms were ultimately placed in idealized positions and treated as riding. The applied constraints were as follows: C_{aryl}—H_{aryl} = 0.93 Å; $C_{methylene}$—$H_{methylene}$ = 0.97 Å; C_{methyl}—H_{methyl} = 0.96 Å and $C_{hydroxy}$—$H_{hydroxy}$ = 0.82 Å. The idealized methyl group was allowed to rotate about the C—C bond during the refinement by application of the command AFIX 137 in *SHELXL97* (Sheldrick, 2008). $U_{iso}(H_{methyl}$ or $H_{hydroxy}) = 1.5U_{eq}(C_{methyl}$ or $O_{hydroxy})$ or $U_{iso}(H_{aryl}$ or $H_{methylene}) = 1.2 U_{eq}(C_{aryl}$ or $C_{methylene})$. The crystal used is an inversion twin with refined components 0.52 (5) and 0.48 (5).
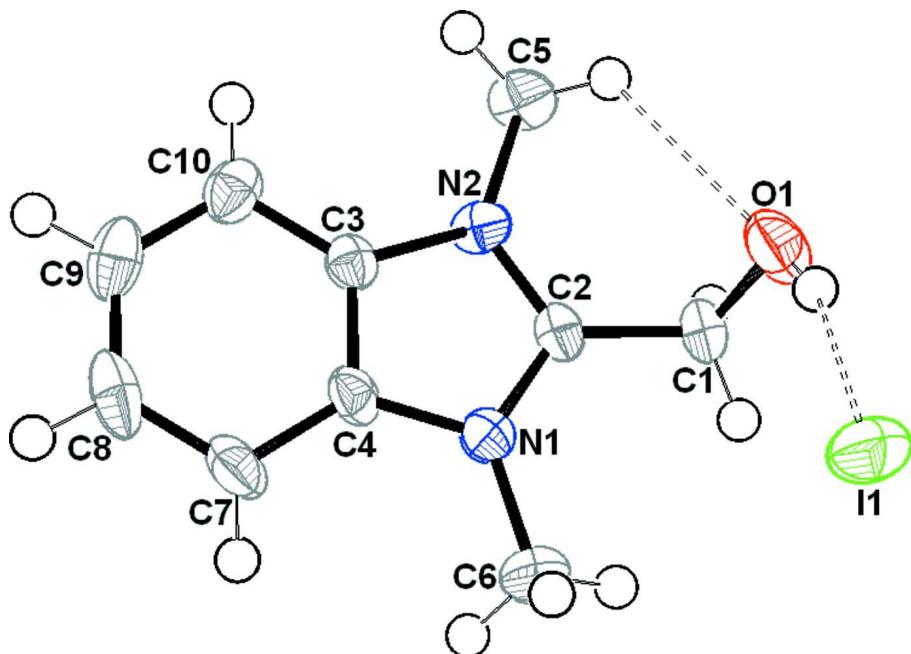
Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius. Hydrogen bonds are shown as dashed lines.
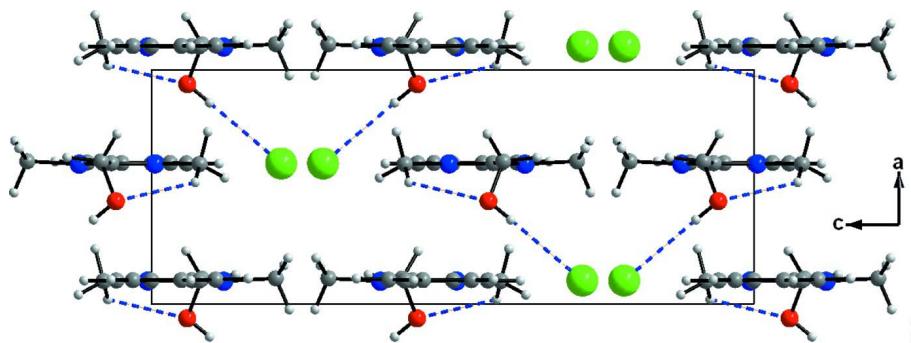
Figure 2

Alternating layers of (I) viewed along the *b* axis showing hydrogen bonds as dashed lines.

2-Hydroxymethyl-1,3-dimethyl-1*H*-benzimidazol-3-ium iodide

Crystal data

$C_{10}H_{13}N_2O^+ \cdot I^-$	$F(000) = 592$
$M_r = 304.12$	$D_x = 1.792$ Mg m^{-3}
Orthorhombic, $P2_1nb$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P -2bc 2a	Cell parameters from 4274 reflections
$a = 6.5690$ (7) Å	$\theta = 2.4–34.0°$
$b = 10.1342$ (10) Å	$\mu = 2.81$ mm^{-1}
$c = 16.9357$ (19) Å	$T = 150$ K
$V = 1127.4$ (2) Å^3	Cube, colorless
$Z = 4$	$0.14 \times 0.13 \times 0.12$ mm

Data collection

Bruker APEXII CCD diffractometer	4002 independent reflections
Graphite monochromator	3243 reflections with $I > 2\sigma(I)$
CCD rotation images, thin slices scans	$R_{int} = 0.02$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)	$\theta_{max} = 34.2°$, $\theta_{min} = 3.1°$
$T_{min} = 0.647$, $T_{max} = 0.747$	$h = -9 \rightarrow 10$
10018 measured reflections	$k = -16 \rightarrow 15$
	$l = -26 \rightarrow 19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2 + 1.1204P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.068$	
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.003$
4002 reflections	$\Delta\rho_{max} = 1.34$ e Å^{-3}
131 parameters	$\Delta\rho_{min} = -0.72$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 1518 Friedel pairs
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: 0.48 (5)
Secondary atom site location: difference Fourier map	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	1.0638 (4)	0.0386 (3)	0.05918 (17)	0.0303 (8)
N1	0.9055 (15)	0.3459 (2)	0.11990 (13)	0.0203 (5)
N2	0.9033 (12)	0.2982 (2)	−0.00606 (12)	0.0168 (5)
C1	0.893 (2)	0.1018 (3)	0.0837 (2)	0.0284 (9)
C2	0.9025 (18)	0.2473 (2)	0.06700 (14)	0.0192 (5)
C3	0.9014 (18)	0.4343 (2)	−0.00046 (14)	0.0165 (5)
C4	0.9022 (15)	0.4649 (2)	0.07953 (15)	0.0179 (6)
C5	0.9046 (16)	0.2214 (3)	−0.07961 (15)	0.0226 (7)
C6	0.893 (2)	0.3340 (3)	0.20631 (18)	0.0326 (11)
C7	0.901 (2)	0.5947 (2)	0.10657 (18)	0.0239 (6)
C8	0.9001 (18)	0.6923 (2)	0.0495 (2)	0.0277 (7)
C9	0.9004 (16)	0.6620 (3)	−0.03096 (19)	0.0255 (7)
C10	0.9028 (14)	0.5327 (2)	−0.05820 (16)	0.0218 (6)
I1	0.3999 (3)	0.08238 (2)	0.21526 (1)	0.0330 (1)
H1	1.15370	0.04750	0.09236	0.0454*
H1A	0.77590	0.06427	0.05712	0.0341*

H1B	0.87606	0.08812	0.13999	0.0341*
H5A	0.98363	0.14266	−0.07223	0.0339*
H5B	0.76764	0.19799	−0.09352	0.0339*
H5C	0.96352	0.27334	−0.12114	0.0339*
H6A	0.83795	0.24932	0.21993	0.0487*
H6B	1.02677	0.34281	0.22861	0.0487*
H6C	0.80648	0.40223	0.22679	0.0487*
H7	0.90136	0.61465	0.16017	0.0287*
H8	0.89907	0.78024	0.06512	0.0333*
H9	0.89902	0.73060	−0.06745	0.0306*
H10	0.90517	0.51273	−0.11180	0.0262*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0335 (14)	0.0250 (11)	0.0324 (14)	0.0107 (11)	−0.0066 (12)	−0.0073 (10)
N1	0.0282 (11)	0.0161 (8)	0.0166 (9)	−0.002 (3)	0.000 (4)	−0.0013 (7)
N2	0.0179 (9)	0.0168 (8)	0.0158 (9)	−0.011 (2)	0.001 (3)	−0.0022 (7)
C1	0.045 (2)	0.0144 (10)	0.0258 (13)	0.009 (3)	0.004 (4)	0.0015 (9)
C2	0.0215 (10)	0.0150 (8)	0.0210 (10)	0.003 (3)	0.003 (5)	−0.0004 (8)
C3	0.0142 (8)	0.0155 (9)	0.0197 (10)	−0.001 (3)	0.001 (5)	−0.0004 (7)
C4	0.0179 (10)	0.0152 (9)	0.0206 (11)	0.006 (3)	−0.003 (4)	−0.0020 (8)
C5	0.0248 (13)	0.0236 (11)	0.0194 (11)	−0.010 (3)	−0.003 (4)	−0.0061 (9)
C6	0.053 (3)	0.0289 (12)	0.0158 (12)	0.003 (4)	−0.001 (4)	−0.0010 (9)
C7	0.0250 (11)	0.0174 (9)	0.0292 (13)	0.005 (4)	0.001 (6)	−0.0065 (9)
C8	0.0222 (11)	0.0130 (9)	0.0480 (17)	0.000 (4)	−0.001 (6)	−0.0015 (10)
C9	0.0183 (11)	0.0205 (10)	0.0377 (15)	−0.008 (3)	−0.003 (5)	0.0089 (10)
C10	0.0188 (10)	0.0226 (10)	0.0240 (12)	0.002 (4)	−0.001 (5)	0.0059 (9)
I1	0.0493 (1)	0.0319 (1)	0.0177 (1)	−0.0001 (3)	−0.0001 (4)	−0.0009 (1)

Geometric parameters (Å, º)

O1—C1	1.357 (11)	C9—C10	1.389 (4)
O1—H1	0.8200	C1—H1A	0.9700
N1—C4	1.387 (3)	C1—H1B	0.9700
N1—C6	1.471 (4)	C5—H5A	0.9600
N1—C2	1.342 (3)	C5—H5B	0.9600
N2—C3	1.383 (3)	C5—H5C	0.9600
N2—C5	1.469 (3)	C6—H6A	0.9600
N2—C2	1.341 (3)	C6—H6B	0.9600
C1—C2	1.503 (4)	C6—H6C	0.9600
C3—C10	1.397 (3)	C7—H7	0.9300
C3—C4	1.390 (3)	C8—H8	0.9300
C4—C7	1.393 (3)	C9—H9	0.9300
C7—C8	1.383 (4)	C10—H10	0.9300
C8—C9	1.397 (5)		
C1—O1—H1	109.00	C2—C1—H1A	109.00

C2—N1—C4	108.6 (2)	C2—C1—H1B	109.00
C2—N1—C6	127.0 (2)	H1A—C1—H1B	108.00
C4—N1—C6	124.1 (2)	N2—C5—H5A	109.00
C2—N2—C3	108.69 (19)	N2—C5—H5B	109.00
C2—N2—C5	125.4 (2)	N2—C5—H5C	109.00
C3—N2—C5	125.9 (2)	H5A—C5—H5B	109.00
O1—C1—C2	111.8 (8)	H5A—C5—H5C	109.00
N1—C2—C1	127.3 (2)	H5B—C5—H5C	109.00
N2—C2—C1	123.5 (2)	N1—C6—H6A	109.00
N1—C2—N2	109.24 (19)	N1—C6—H6B	109.00
N2—C3—C10	131.6 (2)	N1—C6—H6C	109.00
C4—C3—C10	121.5 (2)	H6A—C6—H6B	109.00
N2—C3—C4	106.82 (19)	H6A—C6—H6C	110.00
N1—C4—C7	131.3 (2)	H6B—C6—H6C	109.00
C3—C4—C7	122.1 (2)	C4—C7—H7	122.00
N1—C4—C3	106.66 (19)	C8—C7—H7	122.00
C4—C7—C8	116.5 (3)	C7—C8—H8	119.00
C7—C8—C9	121.6 (2)	C9—C8—H8	119.00
C8—C9—C10	122.1 (3)	C8—C9—H9	119.00
C3—C10—C9	116.2 (2)	C10—C9—H9	119.00
O1—C1—H1A	109.00	C3—C10—H10	122.00
O1—C1—H1B	109.00	C9—C10—H10	122.00
C2—N1—C4—C7	179.2 (12)	C3—N2—C2—C1	176.7 (11)
C6—N1—C4—C7	4.8 (19)	O1—C1—C2—N2	66.5 (13)
C4—N1—C2—N2	1.8 (13)	O1—C1—C2—N1	−115.6 (11)
C6—N1—C2—N2	175.9 (10)	N2—C3—C10—C9	−179.9 (11)
C4—N1—C2—C1	−176.4 (11)	C4—C3—C10—C9	−1.4 (15)
C6—N1—C2—C1	−2 (2)	C10—C3—C4—C7	1.1 (17)
C6—N1—C4—C3	−175.6 (10)	N2—C3—C4—N1	0.3 (12)
C2—N1—C4—C3	−1.2 (12)	N2—C3—C4—C7	179.9 (10)
C5—N2—C2—N1	178.8 (9)	C10—C3—C4—N1	−178.6 (10)
C5—N2—C2—C1	−3.0 (17)	N1—C4—C7—C8	179.2 (11)
C3—N2—C2—N1	−1.6 (13)	C3—C4—C7—C8	−0.4 (17)
C5—N2—C3—C4	−179.6 (9)	C4—C7—C8—C9	0.0 (18)
C2—N2—C3—C10	179.5 (12)	C7—C8—C9—C10	−0.4 (18)
C2—N2—C3—C4	0.8 (12)	C8—C9—C10—C3	1.1 (15)
C5—N2—C3—C10	−0.9 (18)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···I1[i]	0.8200	2.6600	3.473 (3)	171.00
C5—H5A···O1	0.9600	2.5200	3.170 (5)	125.00

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