organic compounds

2-Hydroxymethyl-1,3-dimethyl-1H-imidazol-3-ium triiodide

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The crystal packing of the title salt, $C_6H_{11}N_2O^+ \cdot I_3^-$, can be described as consisting of alternating layers of cations and anions parallel to the (100) plane along the a-axis direction. The components are linked by O—H···I, C—H···I and C—H···O interactions, generating a three-dimensional network. The O atom deviates from the imidazol ring by 0.896 (2) Å.

Related literature

For the importance of heterocyclic compounds and their applications, see: Pandey *et al.* (2009); Nasser (2000). For the biological activity of imidazole and imidazolium derivatives, see: Ucucu *et al.* (2001); Dominianni *et al.* (1989); Ozkay *et al.* (2010). For our previous work on imidazole derivatives, see: Bahnous *et al.* (2012); Zama *et al.* (2013); Chelghoum *et al.* (2011).
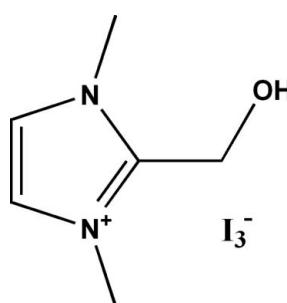
Experimental

Crystal data

$C_6H_{11}N_2O^+ \cdot I_3^-$
M_r = 507.87
Monoclinic, $P2_1/c$
a = 7.1647 (8) Å
b = 15.5586 (19) Å
c = 11.3201 (13) Å
β = 96.026 (7)°
V = 1254.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.44 mm^{-1}
T = 150 K
$0.24 \times 0.03 \times 0.02$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.781, T_{max} = 1.000
7061 measured reflections
2222 independent reflections
2104 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.015
$wR(F^2)$ = 0.034
S = 1.15
2222 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = -0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···I1^i	0.82	3.03	3.741 (2)	146
C1—H1B···I3^{ii}	0.97	3.05	3.924 (3)	151
C4—H4···O1^{iii}	0.93	2.60	3.421 (4)	148

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SMART* (Bruker, 2006); data reduction: *SMART*; 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).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5333).

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supporting information

2-Hydroxymethyl-1,3-dimethyl-1*H*-imidazol-3-ium triiodide

Mohamed El Hadi Said, Abdelmalek Bouraiou, Sofiane Bouacida, Hocine Merazig, Ali Belfaitah and Aissa Chibani

S1. Comment

Heterocyclic compounds have so far been synthesized mainly due to the wide range of biological activities. At present, the role of heterocyclic compounds has become increasingly important in designing new class of structural entities of medicinal importance (Pandey *et al.*, 2009; Nasser, 2000). Imidazole is a nitrogen containing heterocyclic ring which possesses biological and pharmaceutical importance (Ozkay, *et al.*, 2010). It forms the main structure of some well known components of human organisms, *i.e.* the amino acid histidine, Vit-B12, a component of DNA base structure and purines, histamine and biotin (Ucucu, *et al.*, 2001). In other hand, imidazolium salts are known for the wide range of their biological activity. A large variety of these salts have been used as anti-inflammatory, antibacterial, antifungal and thromboxane synthetase inhibitior (Dominianni *et al.*, 1989). In continuation of our studies on imidazole derivatives (Bahnous *et al.*, 2012; Zama *et al.*, 2013 and Chelghoum *et al.*, 2011). We report herein the synthesis and crystal structure of a new imidazolium salt, I, bearing two methyl groups at C-1 and C-3 positions, a hydroxymethyl at C-2 and a triiodide anion that balance the charge.

The molecular geometry and the atom-numbering scheme of (I) are shown in Fig. 1. The asymmetric unit of title molecule, $C_6H_{11}N_2O$, I_3, contains a 1,3-dimethyl-2-hydroxymethylidazolium cation and triiodide anion. The crystal packing can be described as alternating layers parallel to the (100) plane along the *a* axis, where triiodide anion is located in these layers (Fig. 2) and they are linked together by O—H⋯I, C—H⋯I and C—H⋯O intermolecular hydrogen bonds As shown in the Figure 2, the imidazol rings of the symmetry related layers are intercalated, however the centroid to centroid distance between the imidazol rings are too long (5.3400 (19) and 5.6641 (19) Å) for considering π-π interactions. These interaction bonds link the molecules within the layers and also link the layers together, reinforcing the cohesion of the ionic structure. Hydrogen-bonding parameters are listed in table 1.

S2. Experimental

The treatment of 1,3-dimethyl-2-hydroxymethylimidazolium iodide (Chelghoum *et al.*, 2011) with diluted sulfuric acid solution, during ten days in opened flask for slow evaporation, gave the title compound as a brown crystals. The crystals are filtered off and washed with water. Suitable crystal of compound (I) was selected and X-ray crystallographic analysis confirmed the structural assignment (Fig. 1).

S3. Refinement

Approximate positions for all the H atoms were first obtained from the difference electron density map. However, the H atoms were situated into idealized positions and the H-atoms have been refined within the riding atom approximation. The applied constraints were as follow: 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). $U_{iso}(H_{methyl \; or \; hydroxy}) = 1.5 U_{eq}(C_{methyl \; or \; hydroxy})$ or $U_{iso}(H_{aryl}$ or $H_{methylene}) = 1.2 \; U_{eq}(C_{aryl}$ or $C_{methylene})$.
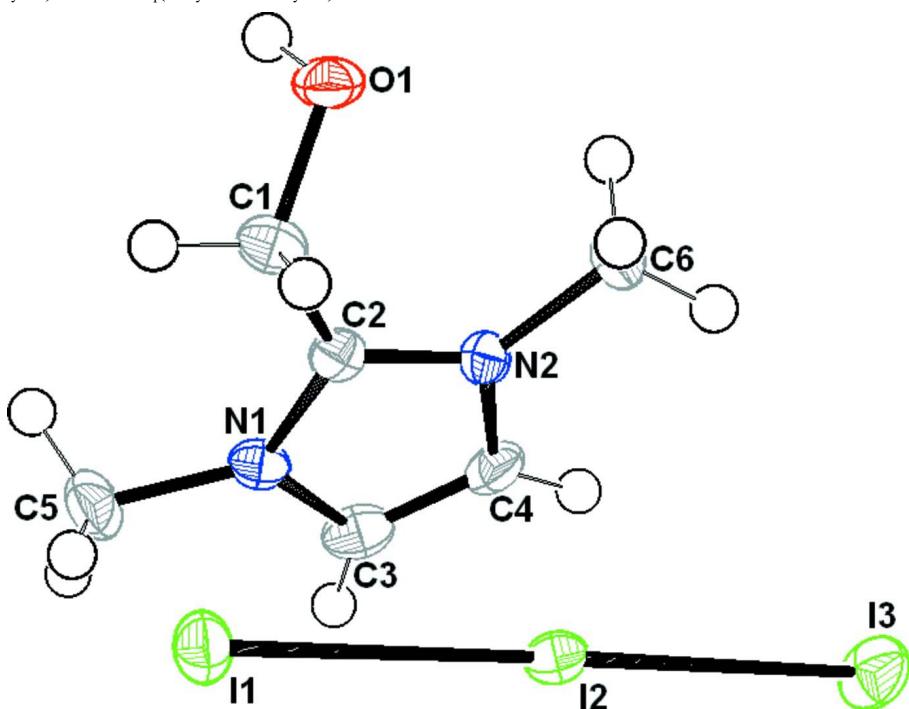
Figure 1

The molecular geometry of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.
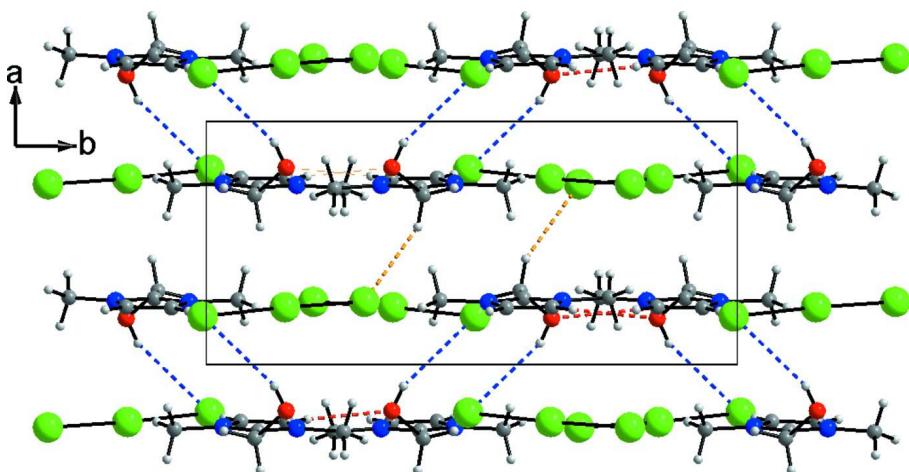
Figure 2

Alternating layers of (I) viewed down the *c* axis showing hydrgen bond as dashed line.

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$C_6H_{11}N_2O^+ \cdot I_3^-$	Monoclinic, $P2_1/c$
$M_r = 507.87$	Hall symbol: -P 2ybc

a = 7.1647 (8) Å
b = 15.5586 (19) Å
c = 11.3201 (13) Å
β = 96.026 (7)°
V = 1254.9 (3) Å^3
Z = 4
$F(000)$ = 912
D_x = 2.688 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5058 reflections
θ = 2.2–25.1°
μ = 7.44 mm^{-1}
T = 150 K
Stick, brown
0.24 × 0.03 × 0.02 mm

Data collection

Bruker APEXII
    diffractometer
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan
    (*SADABS*; Sheldrick, 2002)
T_{min} = 0.781, T_{max} = 1.000
7061 measured reflections

2222 independent reflections
2104 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.1°, θ_{min} = 2.6°
$h = -8 \rightarrow 8$
$k = -18 \rightarrow 18$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.015
$wR(F^2)$ = 0.034
S = 1.15
2222 reflections
112 parameters
0 restraints
Primary atom site location: structure-invariant
    direct methods

Secondary atom site location: difference Fourier
    map
Hydrogen site location: inferred from
    neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 1.5935P]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
I2	0.23583 (2)	0.148350 (12)	0.326494 (15)	0.01269 (6)
I1	0.19816 (3)	−0.006510 (12)	0.171029 (17)	0.01684 (6)
I3	0.25836 (3)	0.297933 (14)	0.474053 (18)	0.02126 (6)
N2	0.7383 (3)	0.17050 (16)	0.3419 (2)	0.0144 (5)
O1	0.8099 (3)	0.15052 (14)	0.09130 (18)	0.0177 (5)
H1	0.9119	0.1286	0.0833	0.027*
N1	0.7438 (3)	0.03209 (16)	0.3568 (2)	0.0141 (5)
C2	0.7275 (4)	0.09763 (19)	0.2795 (3)	0.0132 (6)
C5	0.7344 (4)	−0.0598 (2)	0.3266 (3)	0.0209 (7)

H5A	0.61	−0.0738	0.292	0.031*
H5B	0.7643	−0.0933	0.3972	0.031*
H5C	0.8227	−0.0723	0.2707	0.031*
C6	0.7239 (4)	0.2584 (2)	0.2943 (3)	0.0196 (7)
H6A	0.8421	0.2751	0.2686	0.029*
H6B	0.6918	0.2971	0.3551	0.029*
H6C	0.6285	0.2605	0.2282	0.029*
C3	0.7663 (4)	0.0651 (2)	0.4703 (3)	0.0195 (7)
H3	0.7811	0.0336	0.5405	0.023*
C4	0.7630 (4)	0.1512 (2)	0.4610 (3)	0.0171 (7)
H4	0.7751	0.1904	0.5234	0.021*
C1	0.6972 (4)	0.0911 (2)	0.1468 (3)	0.0160 (6)
H1A	0.7269	0.0332	0.1228	0.019*
H1B	0.566	0.1018	0.1204	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I2	0.01199 (10)	0.01601 (11)	0.01018 (10)	−0.00047 (7)	0.00176 (7)	0.00073 (7)
I1	0.02125 (11)	0.01258 (11)	0.01685 (11)	−0.00009 (8)	0.00283 (8)	−0.00207 (8)
I3	0.02175 (11)	0.02351 (12)	0.01884 (12)	−0.00312 (8)	0.00360 (8)	−0.00943 (8)
N2	0.0138 (12)	0.0121 (13)	0.0176 (14)	0.0021 (10)	0.0039 (10)	0.0011 (11)
O1	0.0196 (10)	0.0200 (12)	0.0144 (11)	−0.0001 (9)	0.0058 (8)	0.0043 (9)
N1	0.0139 (12)	0.0159 (13)	0.0122 (12)	−0.0006 (10)	0.0008 (9)	0.0034 (11)
C2	0.0075 (13)	0.0149 (15)	0.0172 (15)	0.0021 (11)	0.0017 (11)	0.0017 (12)
C5	0.0276 (17)	0.0114 (16)	0.0234 (17)	0.0011 (13)	0.0022 (13)	0.0032 (13)
C6	0.0235 (16)	0.0124 (16)	0.0239 (17)	0.0016 (13)	0.0075 (13)	0.0029 (13)
C3	0.0193 (15)	0.0275 (19)	0.0117 (15)	0.0002 (13)	0.0021 (12)	0.0025 (13)
C4	0.0182 (15)	0.0213 (17)	0.0127 (15)	−0.0006 (13)	0.0054 (12)	−0.0039 (13)
C1	0.0158 (14)	0.0176 (16)	0.0141 (15)	−0.0007 (12)	−0.0004 (11)	0.0042 (13)

Geometric parameters (Å, º)

I2—I3	2.8594 (4)	C5—H5A	0.96
I2—I1	2.9792 (4)	C5—H5B	0.96
N2—C2	1.334 (4)	C5—H5C	0.96
N2—C4	1.374 (4)	C6—H6A	0.96
N2—C6	1.470 (4)	C6—H6B	0.96
O1—C1	1.417 (3)	C6—H6C	0.96
O1—H1	0.82	C3—C4	1.344 (5)
N1—C2	1.341 (4)	C3—H3	0.93
N1—C3	1.377 (4)	C4—H4	0.93
N1—C5	1.470 (4)	C1—H1A	0.97
C2—C1	1.499 (4)	C1—H1B	0.97
I3—I2—I1	178.026 (8)	N2—C6—H6B	109.5
C2—N2—C4	109.2 (3)	H6A—C6—H6B	109.5
C2—N2—C6	126.8 (3)	N2—C6—H6C	109.5

C4—N2—C6	124.0 (3)	H6A—C6—H6C	109.5
C1—O1—H1	109.5	H6B—C6—H6C	109.5
C2—N1—C3	108.6 (3)	C4—C3—N1	107.4 (3)
C2—N1—C5	126.1 (3)	C4—C3—H3	126.3
C3—N1—C5	125.3 (3)	N1—C3—H3	126.3
N2—C2—N1	107.7 (3)	C3—C4—N2	107.1 (3)
N2—C2—C1	125.7 (3)	C3—C4—H4	126.4
N1—C2—C1	126.6 (3)	N2—C4—H4	126.4
N1—C5—H5A	109.5	O1—C1—C2	111.7 (2)
N1—C5—H5B	109.5	O1—C1—H1A	109.3
H5A—C5—H5B	109.5	C2—C1—H1A	109.3
N1—C5—H5C	109.5	O1—C1—H1B	109.3
H5A—C5—H5C	109.5	C2—C1—H1B	109.3
H5B—C5—H5C	109.5	H1A—C1—H1B	107.9
N2—C6—H6A	109.5		
C4—N2—C2—N1	0.4 (3)	C2—N1—C3—C4	0.2 (3)
C6—N2—C2—N1	−178.8 (2)	C5—N1—C3—C4	−178.4 (3)
C4—N2—C2—C1	178.8 (3)	N1—C3—C4—N2	0.1 (3)
C6—N2—C2—C1	−0.4 (4)	C2—N2—C4—C3	−0.3 (3)
C3—N1—C2—N2	−0.4 (3)	C6—N2—C4—C3	179.0 (3)
C5—N1—C2—N2	178.2 (2)	N2—C2—C1—O1	44.3 (4)
C3—N1—C2—C1	−178.7 (3)	N1—C2—C1—O1	−137.6 (3)
C5—N1—C2—C1	−0.2 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···I1[i]	0.82	3.03	3.741 (2)	146
C1—H1*B*···I3[ii]	0.97	3.05	3.924 (3)	151
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Symmetry codes: (i) $x+1, y, z$; (ii) $x, -y+1/2, z-1/2$; (iii) $x, -y+1/2, z+1/2$.