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1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

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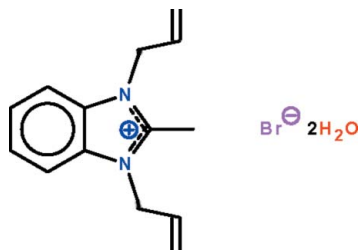
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in solvent or counterion; R factor = 0.027; wR factor = 0.085; data-to-parameter ratio = 18.0.

The bonds in the five-membered ring of the title hydrated salt, $\text{C}_{14}\text{H}_{17}\text{N}_2^+\cdot\text{Br}^-\cdot 2\text{H}_2\text{O}$, are delocalized. The cation lies on a special position of m site symmetry such that the mirror plane passes through the imidazolyl-methyl bond and is perpendicular to the plane of the cation. The anion lies on another special position of 2 site symmetry. The anion and uncoordinated water molecule are linked into a chain by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. One of the water O atoms is disordered over two sites of equal occupancy.

Related literature

For the crystal structure of 1,3-diallylbenzimidazolium bromide, see: Holtgrewe *et al.* (2009). For those of the 1-allyl-3-(cyanobenzyl)benzimidazolium bromide and its hydrate, see: Xu *et al.* (2008); Xu & Ye (2008).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{17}\text{N}_2^+\cdot\text{Br}^-\cdot 2\text{H}_2\text{O}$
 $M_r = 329.24$
 Monoclinic, $C2/m$
 $a = 13.2888$ (2) Å
 $b = 16.8763$ (2) Å
 $c = 7.3897$ (1) Å
 $\beta = 109.773$ (1)°

$V = 1559.54$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.64$ mm⁻¹
 $T = 295$ K
 $0.4 \times 0.3 \times 0.2$ mm

Data collection

Bruker APEXII diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.403$, $T_{\max} = 0.590$

11870 measured reflections
 1851 independent reflections
 1497 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.085$
 $S = 0.95$
 1851 reflections
 103 parameters
 6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H11}\cdots\text{Br1}$	0.86 (1)	2.49 (1)	3.351 (2)	178 (2)
$\text{O1}-\text{H13}\cdots\text{O1}^{\text{i}}$	0.86 (1)	1.98 (1)	2.822 (5)	166 (4)
$\text{O1}-\text{H12}\cdots\text{O1}^{\text{ii}}$	0.85 (1)	1.97 (2)	2.748 (4)	152 (3)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2636).

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1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

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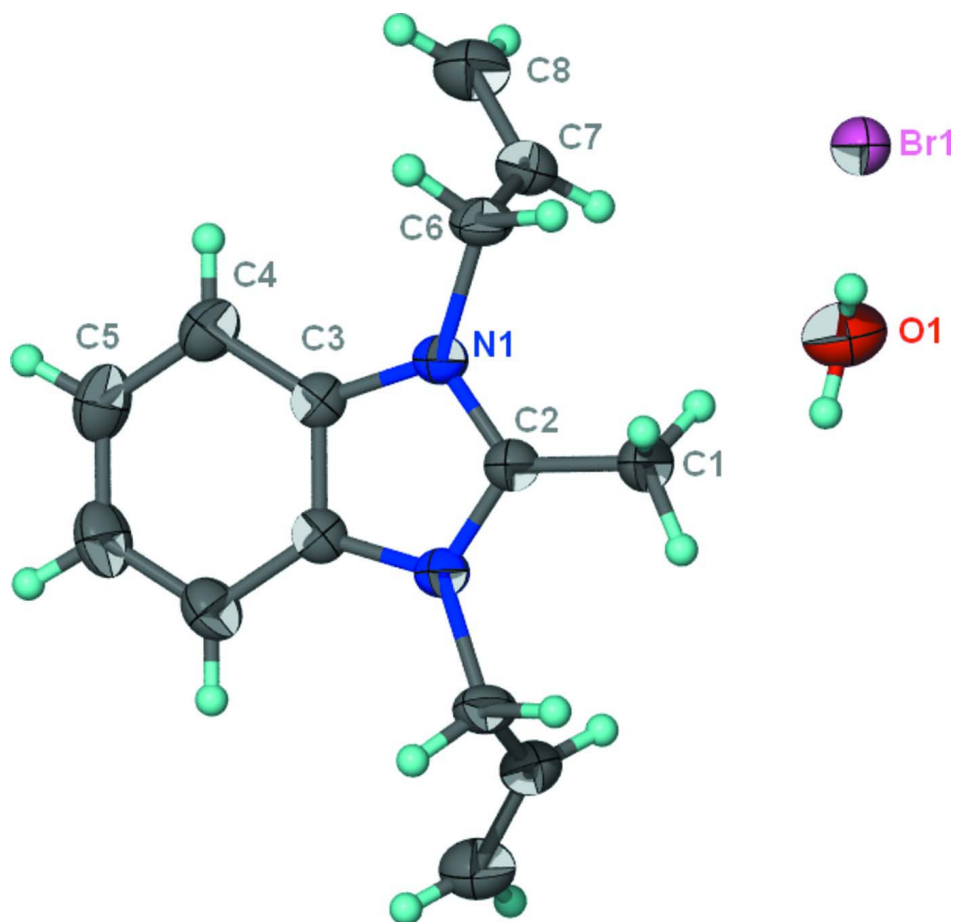
S1. Experimental

2-Methylbenzimidazole (1 g, 7.5 mmol), potassium carbonate (1.55 g, 11.25 mmol) and tetra-*n*-butylammonium bromide (0.18 g, 0.75 mmol) were stirred in *N,N*-dimethylformamide (50 ml) for an hour. To this suspension was added allyl bromide (1.96 ml, 22.5 mmol); the mixture was stirred for two days. The mixture was filtered and the solvent removed under vacuum. The residue was crystallized from ethanol to give yellow crystals in 60% yield; m.p. 516–518 K. The formulation was established by ¹H- and ¹³C-NMR spectroscopic analysis.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U(\text{C})$. The methyl H-atoms lie on general positions, and are disordered; their occupancies are all 0.5.

The water H-atoms were located in a difference Fourier map and were refined with distance restraints (O—H 0.85 ± 0.01 Å; H···H 1.39 ± 0.01 Å). That hydrogen-bonded to Br1 is ordered whereas the other is disordered over two positions of 0.5 site occupancy. The isotropic temperature factors of the three H-atoms were refined.

**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of $[C_{14}H_{19}N_2O][Br] \cdot H_2O$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in the water molecule is not shown. The unlabeled atoms are related to the labeled ones by the $x, 1 - y, z$ symmetry operation.

1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

Crystal data

$C_{14}H_{17}N_2^+ \cdot Br^- \cdot 2H_2O$

$M_r = 329.24$

Monoclinic, $C2/m$

Hall symbol: $-C 2y$

$a = 13.2888 (2) \text{ \AA}$

$b = 16.8763 (2) \text{ \AA}$

$c = 7.3897 (1) \text{ \AA}$

$\beta = 109.773 (1)^\circ$

$V = 1559.54 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 680$

$D_x = 1.402 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 7035 reflections

$\theta = 3.6\text{--}24.3^\circ$

$\mu = 2.64 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Prism, yellow

$0.4 \times 0.3 \times 0.2 \text{ mm}$

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.403$, $T_{\max} = 0.590$

11870 measured reflections
 1851 independent reflections
 1497 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.4^\circ$
 $h = -14 \rightarrow 17$
 $k = -21 \rightarrow 19$
 $l = -8 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.085$
 $S = 0.95$
 1851 reflections
 103 parameters
 6 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.349P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$

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)
Br1	0.0000	0.691870 (15)	0.5000	0.05502 (14)	
O1	0.07379 (17)	0.58143 (11)	0.9005 (3)	0.0787 (5)	
H11	0.055 (2)	0.6089 (13)	0.796 (2)	0.084 (9)*	
H12	0.086 (3)	0.5342 (9)	0.874 (5)	0.075 (18)*	0.50
H13	0.021 (2)	0.582 (2)	0.942 (5)	0.10 (2)*	0.50
N1	0.34476 (11)	0.56471 (8)	0.6215 (2)	0.0381 (3)	
C1	0.1655 (2)	0.5000	0.4821 (5)	0.0557 (7)	
H1A	0.1386	0.4473	0.4841	0.083*	0.50
H1B	0.1472	0.5176	0.3514	0.083*	0.50
H1C	0.1343	0.5351	0.5507	0.083*	0.50
C2	0.2831 (2)	0.5000	0.5747 (4)	0.0393 (5)	
C3	0.45113 (13)	0.54109 (10)	0.7010 (2)	0.0375 (4)	
C4	0.54525 (15)	0.58475 (12)	0.7654 (3)	0.0487 (5)	
H4	0.5454	0.6399	0.7646	0.058*	
C5	0.63843 (16)	0.54125 (14)	0.8307 (3)	0.0562 (5)	
H5	0.7034	0.5679	0.8762	0.067*	
C6	0.30794 (16)	0.64801 (10)	0.5930 (3)	0.0457 (4)	
H6A	0.2385	0.6504	0.4925	0.055*	
H6B	0.3576	0.6791	0.5517	0.055*	
C7	0.30000 (19)	0.68243 (11)	0.7729 (3)	0.0509 (5)	
H7	0.2527	0.6592	0.8254	0.061*	
C8	0.3551 (2)	0.74283 (14)	0.8607 (3)	0.0640 (6)	
H8A	0.4031	0.7673	0.8115	0.077*	
H8B	0.3468	0.7618	0.9729	0.077*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0512 (2)	0.04358 (19)	0.0763 (2)	0.000	0.02951 (16)	0.000

O1	0.0922 (14)	0.0556 (11)	0.0888 (13)	-0.0055 (9)	0.0312 (12)	0.0044 (9)
N1	0.0389 (8)	0.0313 (7)	0.0430 (8)	0.0003 (6)	0.0122 (6)	0.0006 (6)
C1	0.0375 (14)	0.0427 (15)	0.082 (2)	0.000	0.0133 (14)	0.000
C2	0.0392 (13)	0.0351 (13)	0.0433 (13)	0.000	0.0135 (11)	0.000
C3	0.0385 (9)	0.0402 (9)	0.0329 (8)	-0.0015 (7)	0.0109 (7)	-0.0002 (7)
C4	0.0459 (10)	0.0509 (11)	0.0466 (10)	-0.0100 (8)	0.0124 (8)	-0.0048 (8)
C5	0.0398 (10)	0.0764 (13)	0.0470 (10)	-0.0110 (10)	0.0076 (8)	-0.0070 (9)
C6	0.0497 (10)	0.0306 (9)	0.0526 (10)	0.0022 (8)	0.0120 (8)	0.0076 (7)
C7	0.0527 (12)	0.0383 (10)	0.0654 (13)	0.0045 (8)	0.0248 (10)	0.0033 (8)
C8	0.0748 (15)	0.0484 (12)	0.0691 (14)	-0.0005 (11)	0.0248 (12)	-0.0055 (10)

Geometric parameters (Å, °)

O1—H11	0.86 (1)	C3—C4	1.390 (3)
O1—H12	0.85 (1)	C4—C5	1.379 (3)
O1—H13	0.86 (1)	C4—H4	0.9300
N1—C2	1.339 (2)	C5—C5 ⁱ	1.392 (5)
N1—C3	1.393 (2)	C5—H5	0.9300
N1—C6	1.480 (2)	C6—C7	1.487 (3)
C1—C2	1.479 (4)	C6—H6A	0.9700
C1—H1A	0.9600	C6—H6B	0.9700
C1—H1B	0.9600	C7—C8	1.294 (3)
C1—H1C	0.9600	C7—H7	0.9300
C2—N1 ⁱ	1.339 (2)	C8—H8A	0.9300
C3—C3 ⁱ	1.387 (4)	C8—H8B	0.9300
H11—O1—H12	108.7 (16)	C5—C4—C3	115.81 (19)
H11—O1—H13	106.6 (16)	C5—C4—H4	122.1
H12—O1—H13	109.4 (17)	C3—C4—H4	122.1
C2—N1—C3	108.70 (15)	C4—C5—C5 ⁱ	122.17 (12)
C2—N1—C6	126.45 (16)	C4—C5—H5	118.9
C3—N1—C6	124.84 (15)	C5 ⁱ —C5—H5	118.9
C2—C1—H1A	109.5	N1—C6—C7	111.34 (15)
C2—C1—H1B	109.5	N1—C6—H6A	109.4
H1A—C1—H1B	109.5	C7—C6—H6A	109.4
C2—C1—H1C	109.5	N1—C6—H6B	109.4
H1A—C1—H1C	109.5	C7—C6—H6B	109.4
H1B—C1—H1C	109.5	H6A—C6—H6B	108.0
N1—C2—N1 ⁱ	109.3 (2)	C8—C7—C6	123.8 (2)
N1—C2—C1	125.33 (11)	C8—C7—H7	118.1
N1 ⁱ —C2—C1	125.33 (11)	C6—C7—H7	118.1
C3 ⁱ —C3—C4	122.02 (12)	C7—C8—H8A	120.0
C3 ⁱ —C3—N1	106.63 (9)	C7—C8—H8B	120.0
C4—C3—N1	131.31 (17)	H8A—C8—H8B	120.0
C3—N1—C2—N1 ⁱ	0.3 (2)	C6—N1—C3—C4	-2.1 (3)
C6—N1—C2—N1 ⁱ	179.79 (12)	C3 ⁱ —C3—C4—C5	-0.5 (2)
C3—N1—C2—C1	-178.4 (2)	N1—C3—C4—C5	-177.78 (17)

C6—N1—C2—C1	1.0 (4)	C3—C4—C5—C5 ⁱ	0.5 (2)
C2—N1—C3—C3 ⁱ	-0.20 (15)	C2—N1—C6—C7	98.5 (2)
C6—N1—C3—C3 ⁱ	-179.67 (13)	C3—N1—C6—C7	-82.1 (2)
C2—N1—C3—C4	177.40 (19)	N1—C6—C7—C8	118.6 (2)

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O1—H11...Br1	0.86 (1)	2.49 (1)	3.351 (2)	178 (2)
O1—H13...O1 ⁱⁱ	0.86 (1)	1.98 (1)	2.822 (5)	166 (4)
O1—H12...O1 ⁱ	0.85 (1)	1.97 (2)	2.748 (4)	152 (3)

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