1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

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The bonds in the five-membered ring of the title hydrated salt, $C_{14}H_{17}N_2{}^+ \cdot Br^- \cdot 2H_2O$, 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 O—H···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).
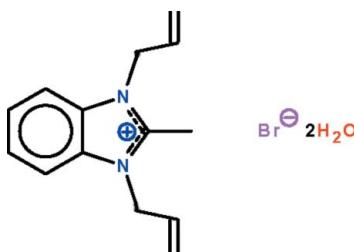
Experimental

Crystal data

$C_{14}H_{17}N_2{}^+ \cdot Br^- \cdot 2H_2O$
$M_r = 329.24$
Monoclinic, $C2/m$
$a = 13.2888\ (2)$ Å
$b = 16.8763\ (2)$ Å
$c = 7.3897\ (1)$ Å
$\beta = 109.773\ (1)°$

$V = 1559.54\ (4)$ Å^3
$Z = 4$
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 $I > 2\sigma(I)$
$R_{int} = 0.024$

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
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Br1	0.86 (1)	2.49 (1)	3.351 (2)	178 (2)
O1—H13···O1^i	0.86 (1)	1.98 (1)	2.822 (5)	166 (4)
O1—H12···O1^{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).

References

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supplementary materials

1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

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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 ally 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 ^{1}H– and ^{13}C-NMR spectroscopic analysis.

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(H) set to 1.2U(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.

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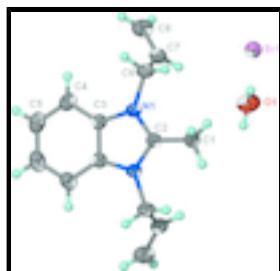
Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of [$C_{14}H_{19}N_2O$][Br]·H_2O at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder in the water molecule is not shown. The unlabeled atoms are related to the labeled ones by the $x, 1 - y, z$ symmetry operation.

1,3-Diallyl-2-methylbenzimidazolium bromide dihydrate

Crystal data

$C_{14}H_{17}N_2^+ \cdot Br^- \cdot 2H_2O$	$F_{000} = 680$
$M_r = 329.24$	$D_x = 1.402$ Mg m^{-3}
Monoclinic, $C2/m$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2y	Cell parameters from 7035 reflections
$a = 13.2888$ (2) Å	$\theta = 3.6–24.3°$
$b = 16.8763$ (2) Å	$\mu = 2.64$ mm^{-1}

$c = 7.3897$ (1) Å

$\beta = 109.773$ (1)°

$V = 1559.54$ (4) Å^3

$Z = 4$

$T = 295$ K

Prism, yellow

$0.4 \times 0.3 \times 0.2$ mm

Data collection

Bruker APEXII diffractometer	1851 independent reflections
Radiation source: fine-focus sealed tube	1497 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 295$ K	$\theta_{max} = 27.5°$
φ and ω scans	$\theta_{min} = 2.4°$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -14 \rightarrow 17$
$T_{min} = 0.403$, $T_{max} = 0.590$	$k = -21 \rightarrow 19$
11870 measured reflections	$l = -8 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.349P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} = 0.001$
1851 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
103 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	y	z	U_{iso}*/U_{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 ($Å^2$)

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

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
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: (ii) $-x, y, -z+2$; (i) $x, -y+1, z$.

Fig. 1