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2,5-Dimethylbufotenine and 2,5-dimethylbufotenidine: novel derivatives of natural tryptamines found in *Bufo alvarius* toads

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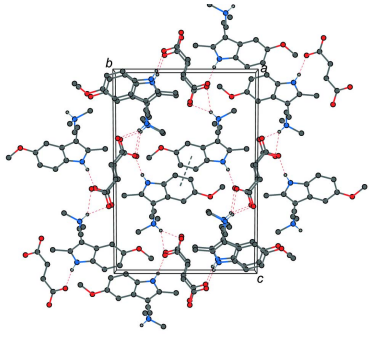
The solid-state structure of the bufotenine derivative bis(5-methoxy-2,*N*,*N*-trimethyltryptammonium) (5-MeO-2-Me-DMT) fumarate (systematic name: bis{[2-(5-methoxy-2-methyl-1*H*-indol-3-yl)ethyl]dimethylazanium} (2*E*)-but-2-enedioate), $2C_{14}H_{21}N_2O^+\cdot C_4H_2O_4^{2-}$, the bufotenidine derivative 5-methoxy-2,*N*,*N*,*N*-tetramethyltryptammonium (5-MeO-2-Me-TMT) iodide {systematic name: [2-(5-methoxy-2-methyl-1*H*-indol-3-yl)ethyl]trimethylazanium iodide}, $C_{15}H_{23}N_2O^+\cdot I^-$, and the hydrate of the same {systematic name: [2-(5-methoxy-2-methyl-1*H*-indol-3-yl)ethyl]trimethylazanium iodide monohydrate}, $C_{15}H_{23}N_2O^+\cdot I^-\cdot H_2O$, are reported. The structure of 5-MeO-2-Me-DMT fumarate possesses one tryptammonium cation and a half of a fumarate dianion in the asymmetric unit, linked together by N—H···O hydrogen bonds in infinite two-dimensional networks parallel to the (101) plane. The structure of 5-MeO-2-Me-TMT iodide possesses one tryptammonium cation and one iodide anion in the asymmetric unit. The ions are linked *via* N—H···I hydrogen bonds, and indoles are coupled in dimers through π–π interactions. The hydrate of 5-MeO-2-Me-TMT iodide possesses one tryptammonium cation, one iodide anion and one water molecule in the asymmetric unit. It shows N—H···I and O—H···I hydrogen bonds that couple the tryptammonium cations into dimers.

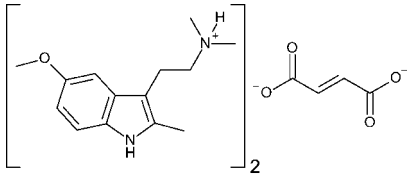
1. Chemical context

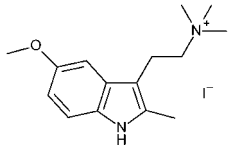
Bufotenine, the *N*,*N*-dimethyl analogue of serotonin, and bufotenidine, the *N*,*N*,*N*-trimethyl analogue of serotonin, were both identified in toad secretions in 1934 (Wieland *et al.*, 1934). These and other indoalkylamines found in the paratoid glands of *Bufo alvarius* toads can lead to psychotropic activity in humans and other animals. Bufotenine is believed to have psychedelic properties due to its activity as a serotonin 2A agonist (Egan *et al.*, 2000). Bufotenidine (5-HTQ) is a site-selective serotonin 5-HT_3 binder (Glennon *et al.*, 1991), and has demonstrated paralytic activity in rats (Bhattacharya & Sanyal, 1972). The best known psychedelic compound in these secretions is the *O*-methylated version of bufotenine [5-methoxy-*N*,*N*-dimethyltryptamine (5-MeO-DMT)] (Spencer Jr *et al.*, 1987). Known as the 'God Molecule', 5-MeO-DMT has been used by humans in religious ceremonies where it is traditionally administered by smoking, or vaporizing the secretions of *Bufo alvarius* toads. 5-MeO-DMT has also been administered intravenously, though it is inactive through oral consumption (Weil & Davis, 1994).

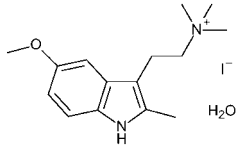
5-Methoxy-2,*N*,*N*-trimethyltryptamine (5-MeO-2-Me-DMT, 2,5-dimethylbufotenine) was first reported in 1955, and crystallized as its picrate salt in two different forms (Shaw,



1955). A detailed synthesis of the freebase of the compound was reported by Alexander Shulgin, who also described its clinical effects on humans, with psychotropic activity occurring within an hour of oral consumption accompanied by physical stimulation (Shulgin & Shulgin, 2016). By contrast, 5-MeO-DMT is not orally active, unless consumed in combination with a monoamine oxidase inhibitor (MAOI). The methylation of the 2-position provides oral activity in 5-MeO-2-Me-DMT, likely by limiting its decomposition by monoamine-oxidases, and also appears to reduce activity at the 5-HT_{2A} receptor, making it significantly less active than inhaled 5-MeO-DMT. Bioassays of this compound have shown it to be an agonist for the serotonin 5-HT_6 receptor (K_i = 89 n*M*) (Glennon, *et al.* 2000) and the serotonin 5-HT_7 receptor (K_i = 1,120 n*M*) (Vermeulen, *et al.* 2003).







Herein we report the structure of 5-methoxy-2,*N*,*N*-trimethyltryptammonium fumarate. We also report the synthesis of 5-methoxy-2,*N*,*N*,*N*-tetramethyltryptammonium iodide (a bufotenidine analogue), along with its structure. Lastly, we report the structure of the first solvate of 5-methoxy-2,*N*,*N*,*N*-tetramethyltryptammonium iodide as its hydrate.

2. Structural commentary

The asymmetric unit of bis(5-methoxy-2,*N*,*N*-trimethyltryptammonium) fumarate contains one tryptammonium cation and one half of a fumarate dianion (Fig. 1, left). The cation possesses a near planar unit containing the indole, the methyl and the methoxy groups, with mean deviation from planarity of 0.047 Å. The ethylamino group is turned away from this plane, with a C2—C9—C10—C11 torsion angle of −95.4 (2)°. The hydrogens of the 2-methyl group carbon (C1) exhibit a rotational disorder over two positions with 50% occupancy. Half of the fumarate is present in the asymmetric unit, with the other half generated by inversion. The dianion is slightly distorted from planarity with an r.m.s. deviation of 0.076 Å. The carboxylate unit is delocalized with C—O distances of 1.222 (3) and 1.225 (2) Å.

The asymmetric unit of 5-methoxy-2,*N*,*N*,*N*-tetramethyltryptammonium iodide contains one tryptammonium cation and one iodide anion (Fig. 1, center). The indole ring, methyl and methoxy groups of the cation are near planar, with a mean deviation from planarity of 0.050 Å. The ethylammonium arm is turned away from the plane with a C7—C8—C9—C10 torsion angle of 100.9 (4)°. The asymmetric unit of its hydrate contains one tryptammonium cation, one iodide anion, and one water molecule (Fig. 1, right). The tryptammonium cation is very similar to the non-hydrate, with a mean deviation from planarity of 0.043 Å for the indole ring, methyl and methoxy groups of the cation, and a C1—C8—C9—C10 torsion angle of 98.0 (2)°. The metrical parameters of the three structures are very similar, with the major difference observed being the elongated N—C(methyl) bonds in the quaternary salts.

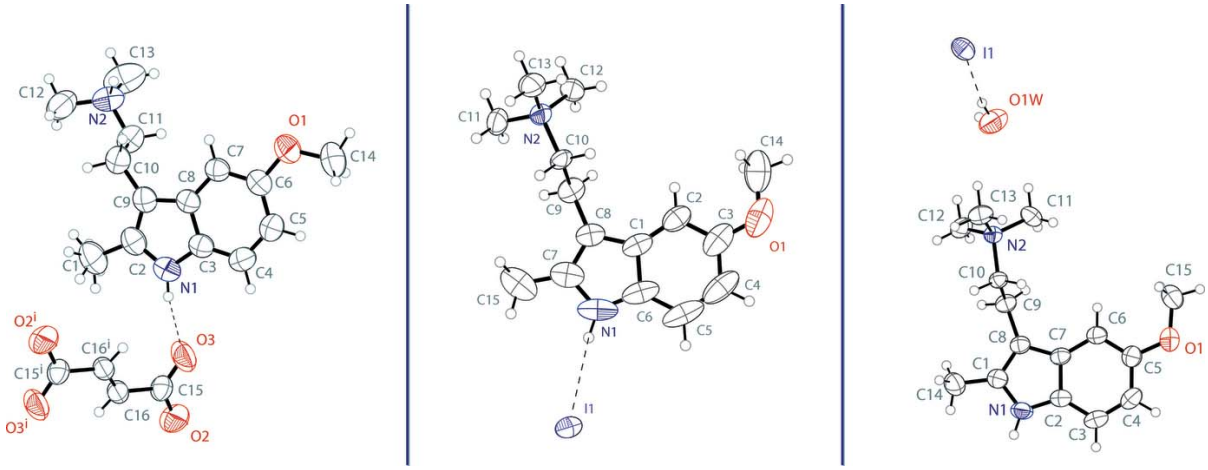


Figure 1
The molecular structure of bis(5-MeO-2-Me-DMT) fumarate (left), 5-MeO-2-Me-TMT iodide (center), and 5-MeO-2-Me-TMT iodide hydrate (right), showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Symmetry code: (i) $-x, -y, 1-z$.

Table 1
Hydrogen-bond geometry (Å, °) for bis(5-MeO-2-Me-DMT) fumarate.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.87 (1)	1.95 (1)	2.810 (3)	168 (2)
N2—H2···O3^{i}	0.88 (1)	2.18 (2)	2.892 (2)	138 (2)
N2—H2···O2^{i}	0.88 (1)	2.00 (1)	2.837 (2)	160 (2)

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

Table 2
Hydrogen-bond geometry (Å, °) for 5-MeO-2-Me-TMTiodide .

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1	0.86 (1)	2.83 (2)	3.662 (3)	161 (4)

Table 3
Hydrogen-bond geometry (Å, °) for 5-MeO-2-Me-TMT iodide hydrate.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···I1^{i}	0.89 (1)	2.74 (1)	3.617 (2)	168 (4)
O1*W*—H1*WB*···I1	0.89 (1)	2.76 (2)	3.618 (2)	164 (4)
N1—H1···I1^{ii}	0.86 (1)	2.96 (1)	3.7416 (17)	153 (2)

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

3. Supramolecular features

In the structure of 5-MeO-2-Me-DMT fumarate, the ammonium nitrogen exhibits a bifurcated N—H···(O,O) hydrogen bond with the two oxygens of a carboxylate unit, and the indole nitrogen is involved in an N—H···O hydrogen bond with one of the carboxylate oxygens (Table 1). This series of N—H···O hydrogen bonds connects the ions together in an infinite two-dimensional network parallel to the (101) plane. The six-membered rings of inversion-related indoles stack with parallel slipped π–π interactions [intercentroid distance = 3.9105 (15) Å, interplanar distance = 3.7688 (19) Å, and slippage = 1.043 (3) Å]. The packing of 5-MeO-2-Me-DMT fumarate is shown at the top of Fig. 2.

In the structure of 5-MeO-2-Me-TMT iodide, the tryptammonium cation and the iodide anion are held together in the asymmetric unit *via* N-–H···I hydrogen bonds, between the indole nitrogen and the iodide (Table 2). The six-membered rings of inversion-related indoles stack with parallel slipped π–π interactions [intercentroid distance = 3.716 (3) Å, interplanar distance = 3.488 (4) Å, and slippage = 1.282 (7) Å] that pair the tryptammonium cations together as dimers in the solid state. The packing of 5-MeO-2-Me-TMT iodide is shown in the center of Fig. 2.

In the structure of the hydrate of 5-MeO-2-Me-TMT iodide, the tryptammonium cation shows an N—H···I hydrogen bond between the indole nitrogen and a symmetry-generated iodide. The water molecule forms O—H···I hydrogen bonds with the iodide anion and another symmetry-generated iodide (Table 3). The interactions of two water molecules and two iodide anions form diamond-shaped rings with graph-set notation $R_4^2(8)$ (Etter *et al.*, 1990). The N—H···I hydrogen bonds combine with the rings to couple the tryptammonium cations together as dimers. The packing of the hydrate of 5-MeO-2-Me-TMT iodide is shown as the bottom of Fig. 2. In moving from 5-MeO-2-Me-TMT to its hydrate, the N—H···I

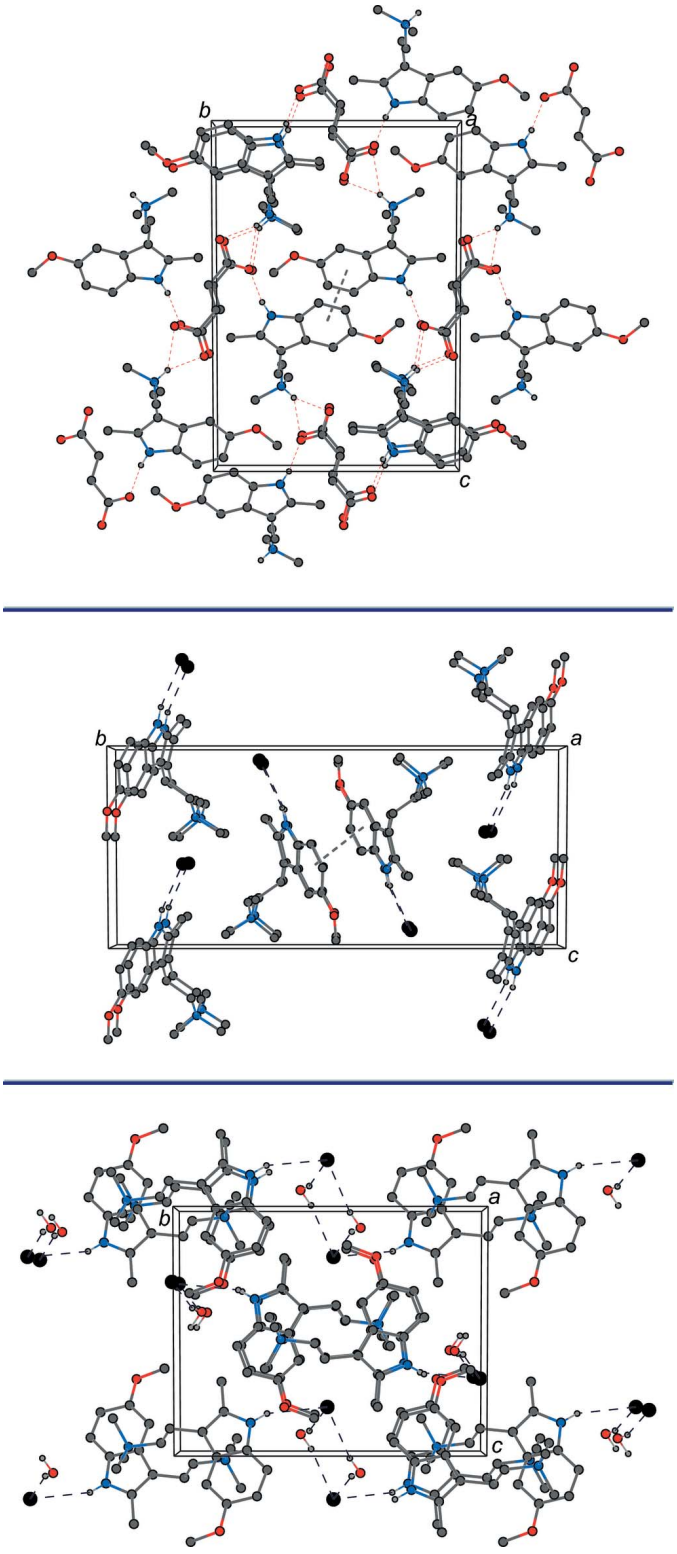


Figure 2
The crystal packing of bis(5-MeO-2-Me-DMT) fumarate (top), 5-MeO-2-Me-TMT iodide (center), and 5-MeO-2-Me-TMT iodide hydrate (bottom), all shown along the *a* axis (*OLEX2*; Dolomanov *et al.*, 2009). Hydrogen bonds and π–π interactions are shown as dashed lines. H atoms not involved in hydrogen bonding are omitted for clarity.

Table 4
Experimental details.

	bis(5-MeO-2-Me-DMT) fumarate	5-MeO-2-Me-TMT iodide	5-MeO-2-Me-TMT iodide hydrate
Crystal data			
Chemical formula	$C_{14}H_{21}N_2O^+\cdot 0.5C_4H_2O_4^{2-}$	$C_{15}H_{23}N_2O^+\cdot I^-$	$C_{15}H_{23}N_2O^+\cdot I^-\cdot H_2O$
M_r	290.35	374.25	392.27
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	297	297	297
a, b, c (Å)	7.7368 (3), 12.1233 (5), 17.5528 (8)	7.5067 (8), 22.657 (3), 10.0894 (11)	10.9091 (10), 14.0910 (11), 11.4029 (10)
β (°)	102.154 (1)	97.225 (4)	100.338 (3)
V (Å^3)	1609.47 (12)	1702.4 (3)	1724.4 (3)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	1.88	1.86
Crystal size (mm)	0.37 × 0.24 × 0.21	0.43 × 0.20 × 0.03	0.38 × 0.22 × 0.20
Data collection			
Diffractometer	Bruker D8 Venture CMOS	Bruker D8 Venture CMOS	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2018)	Multi-scan (*SADABS*; Bruker, 2018)	Multi-scan (*SADABS*; Bruker, 2018)
T_{min}, T_{max}	0.698, 0.745	0.621, 0.745	0.486, 0.562
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	36409, 3005, 2456	40959, 3207, 2875	40738, 3326, 3051
R_{int}	0.040	0.029	0.025
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.611	0.611	0.618
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.157, 1.03	0.028, 0.061, 1.17	0.021, 0.054, 1.10
No. of reflections	3005	3207	3326
No. of parameters	200	180	195
No. of restraints	2	1	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.28	0.46, −0.80	0.40, −0.36

Computer programs: *APEX3* and *SAINT* (Bruker, 2018), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), and *publCIF* (Westrip, 2010).

interaction is elongated as the O—H···I interactions weaken the amine–halide interaction.

4. Database survey

The structure of bufotenine (BUFTEN: Falkenberg, 1972) and its borane adduct (OYOCIQ: Moreira *et al.*, 2015) have been reported. The unit cell of 5-MeO-DMT (QQQAGY: Bergin *et al.*, 1968) and the single crystal structure of its hydrochloride (MOTYPT: Falkenberg & Carlström, 1971) are the other two structures reported for naturally occurring tryptamines of toads. The other simple 5-methoxy tryptamine whose structure is reported is the synthetic compound, 5-methoxy-*N*,*N*-diallyltryptamine (5-MeO-DALT) (CCDC 1995802: Chadeayne *et al.*, 2020*b*). The only two structures of 2-methyltryptamines reported are of the antipsychotic drug oxypertine (CAGXIR: Léger *et al.*, 1983) and its bromide salt (OXYPEB10: Fillers & Hawkinson, 1978), which are used to treat schizophrenia. While the structure of bufotenidine has never been reported, the structure of four quaternary tryptammoniums have, and those are the iodide salts of 4-hydroxy-*N*,*N*,*N*-trimethyltryptamine (4-HO-TMT) and 4-acetoxy-*N*,*N*,*N*-trimethyltryptamine (4-AcO-TMT) (XUXFAA and XUXDUS: Chadeayne, Pham, Reid *et al.*, 2020), and *N*,*N*-dimethyl-*N*-*n*-propyltryptammonium (DMPT) and *N*,*N*-dimethyl-*N*-allyltryptammonium (DMALT) as their iodide salts (CCDC 2017817 and CCDC 2017818: Chadeayne *et al.*, 2020*a*).

5. Synthesis and crystallization

Crystals of 5-MeO-2-Me-DMT fumarate suitable for diffraction studies were obtained from the evaporation of a methanol solution of a commercial sample (The Indole Shop). 5-MeO-2-Me-TMT iodide was synthesized when 128 mg of 5-MeO-2-Me-DMT fumarate was dissolved in 6 mL of methanol, and 6 mL of methyliodide was added. The mixture was refluxed under an atmosphere of nitrogen for 12 h. The solvent was removed *in vacuo* to yield a bright-yellow powder. The powder was washed with diethyl ether to yield 127 mg of a light-yellow powder. The product was recrystallized from methanol and water to yield two different crystalline forms. The product was analyzed by ^{1}H and ^{13}C NMR. ^{1}H NMR (400 MHz, D_2O): δ 7.36 (*d*, *J* = 8.8 Hz, 1 H, Ar*H*), 7.04 (*d*, *J* = 2.3 Hz, 1 H, Ar*H*), 6.88 (*dd*, *J* = 8.8, 2.4 Hz, 1 H, Ar*H*), 3.88 (*s*, 3 H, O*CH*$_3$), 3.44–3.40 (*m*, 2 H, *CH*$_2$), 3.21 (*s*, 9 H, *CH*$_3$), 3.16–3.12 (*m* 2 H, *CH*$_2$), 2.38 (*s*, 3 H, *CH*$_3$). ^{13}C NMR (100 MHz, D_2O): δ 152.6 (Ar*C*), 135.1 (Ar*C*), 130.3 (Ar*C*), 127.3 (Ar*C*), 111.6 (Ar*C*), 109.9 (Ar*C*), 103.0 (Ar*C*), 99.9 (Ar*C*), 65.2 (Ak*C*), 55.9 (Ak*C*), 52.4 (Ak*C*), 17.2 (Ak*C*), 10.5 (Ak*C*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The hydrogen atoms on the indole nitrogen of each structure (H1) and H2 in the fumarate structure were found from a difference-Fourier map and were refined isotropically, using DFIX restraints with N—H distances of 0.87 (1) Å. Isotropic displacement parameters were set to $1.2U_{eq}$ of the parent nitrogen atom. The hydrogen atoms on the water of the hydrate structure (H1*WA*, H1*WB*) were found from a difference-Fourier map and were refined isotropically, using a DFIX restraint with an O—H distance of 0.88 (1) Å. Isotropic displacement parameters were set to $1.5U_{eq}$ of the parent oxygen atom. All other hydrogen atoms were placed in calculated positions (C—H = 0.93-0.97 Å). Isotropic displacement parameters were set to $1.2U_{eq}$(C) or $1.5U_{eq}$(C-methyl). A certain number of reflections is missing from the data of all three structures. This is likely a beamstop related technical issue which could not be resolved as of yet.

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2,5-Dimethylbufotenine and 2,5-dimethylbufotenidine: novel derivatives of natural tryptamines found in *Bufo alvarius* toads

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Computing details

For all structures, data collection: *APEX3* (Bruker, 2018); cell refinement: *SAINT* (Bruker, 2018); data reduction: *SAINT* (Bruker, 2018); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Bis{[2-(5-methoxy-2-methyl-1*H*-indol-3-yl)ethyl]dimethylazanium} 2*E*)-but-2-enedioate (umd1954c_a)

Crystal data

$C_{14}H_{21}N_2O^+ \cdot 0.5C_4H_2O_4^{2-}$
M_r = 290.35
Monoclinic, $P2_1/n$
a = 7.7368 (3) Å
b = 12.1233 (5) Å
c = 17.5528 (8) Å
β = 102.154 (1)°
V = 1609.47 (12) Å^3
Z = 4
$F(000)$ = 624
D_x = 1.198 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9878 reflections
θ = 2.7–25.6°
μ = 0.08 mm^{-1}
T = 297 K
BLOCK, colourless
0.37 × 0.24 × 0.21 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2018)
T_{min} = 0.698, T_{max} = 0.745
36409 measured reflections
3005 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 25.7°, θ_{min} = 2.9°
h = −9→9
k = −14→14
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.157
S = 1.03
3005 reflections
200 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.6102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.12 (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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.3263 (2)	0.67305 (13)	0.37842 (10)	0.0836 (5)	
O3	0.1859 (3)	0.14716 (14)	0.58082 (10)	0.1018 (7)	
O2	0.1411 (3)	0.02997 (16)	0.66613 (8)	0.0945 (6)	
N1	0.2833 (2)	0.23477 (15)	0.44770 (11)	0.0669 (5)	
H1	0.257 (3)	0.1984 (18)	0.4864 (10)	0.080*	
N2	0.7825 (2)	0.27124 (16)	0.23889 (10)	0.0658 (5)	
H2	0.738 (3)	0.3232 (15)	0.2059 (11)	0.079*	
C1	0.3791 (4)	0.0682 (2)	0.38388 (19)	0.0938 (8)	
H1A	0.334479	0.032317	0.424551	0.141*	0.5
H1B	0.313416	0.043848	0.334105	0.141*	0.5
H1C	0.501665	0.050005	0.388708	0.141*	0.5
H1D	0.431894	0.051796	0.340358	0.141*	0.5
H1E	0.452957	0.040265	0.430804	0.141*	0.5
H1F	0.264708	0.034108	0.376201	0.141*	0.5
C2	0.3598 (3)	0.19070 (17)	0.39056 (13)	0.0656 (6)	
C3	0.2816 (2)	0.34787 (16)	0.44112 (11)	0.0560 (5)	
C4	0.2189 (3)	0.42778 (19)	0.48417 (12)	0.0659 (6)	
H4	0.167979	0.408617	0.525816	0.079*	
C5	0.2334 (3)	0.53672 (18)	0.46408 (13)	0.0667 (6)	
H5	0.193328	0.591857	0.492890	0.080*	
C6	0.3078 (3)	0.56512 (16)	0.40087 (12)	0.0605 (5)	
C7	0.3669 (2)	0.48565 (16)	0.35662 (11)	0.0557 (5)	
H7	0.413399	0.505344	0.313799	0.067*	
C8	0.3562 (2)	0.37523 (15)	0.37691 (10)	0.0515 (5)	
C9	0.4050 (2)	0.27364 (16)	0.34566 (12)	0.0574 (5)	
C10	0.4906 (3)	0.26084 (19)	0.27770 (13)	0.0665 (6)	
H10A	0.444341	0.315705	0.238481	0.080*	
H10B	0.464230	0.188392	0.254737	0.080*	
C11	0.6900 (3)	0.27476 (19)	0.30381 (12)	0.0651 (6)	
H11A	0.714481	0.344780	0.330661	0.078*	
H11B	0.735842	0.216709	0.340560	0.078*	
C12	0.7638 (4)	0.1629 (2)	0.19843 (16)	0.0978 (9)	
H12A	0.641302	0.149529	0.176090	0.147*	
H12B	0.807970	0.105647	0.235151	0.147*	
H12C	0.829966	0.163573	0.157907	0.147*	
C13	0.9701 (3)	0.2993 (3)	0.26402 (18)	0.1124 (12)	
H13A	0.981166	0.371141	0.287591	0.169*	
H13B	1.024691	0.299440	0.219710	0.169*	
H13C	1.027309	0.245802	0.301211	0.169*	

C14	0.2885 (4)	0.7581 (2)	0.42723 (17)	0.0922 (8)
H14A	0.311697	0.828292	0.406078	0.138*
H14B	0.361662	0.749779	0.478335	0.138*
H14C	0.166321	0.754253	0.430504	0.138*
C15	0.1294 (3)	0.05865 (16)	0.59833 (10)	0.0567 (5)
C16	0.0335 (3)	−0.01511 (14)	0.53555 (10)	0.0530 (5)
H16	0.020537	−0.088678	0.548278	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1053 (13)	0.0551 (9)	0.0918 (11)	0.0036 (8)	0.0242 (9)	0.0046 (8)
O3	0.1565 (19)	0.0704 (10)	0.0670 (10)	−0.0486 (11)	−0.0027 (10)	−0.0076 (8)
O2	0.1179 (14)	0.1187 (15)	0.0407 (8)	−0.0287 (11)	0.0028 (8)	−0.0025 (8)
N1	0.0666 (11)	0.0622 (11)	0.0717 (11)	−0.0061 (8)	0.0140 (9)	0.0090 (8)
N2	0.0500 (9)	0.0853 (13)	0.0599 (10)	0.0160 (8)	0.0066 (7)	0.0249 (9)
C1	0.0986 (19)	0.0601 (14)	0.121 (2)	0.0031 (13)	0.0187 (16)	0.0006 (14)
C2	0.0545 (11)	0.0587 (11)	0.0790 (14)	−0.0016 (9)	0.0032 (10)	−0.0014 (10)
C3	0.0470 (9)	0.0604 (11)	0.0580 (11)	−0.0049 (8)	0.0051 (8)	0.0038 (8)
C4	0.0643 (12)	0.0782 (14)	0.0570 (11)	−0.0023 (10)	0.0166 (9)	0.0029 (10)
C5	0.0673 (13)	0.0677 (13)	0.0656 (12)	0.0062 (10)	0.0149 (10)	−0.0073 (10)
C6	0.0561 (11)	0.0571 (11)	0.0647 (12)	−0.0013 (8)	0.0042 (9)	0.0034 (9)
C7	0.0474 (9)	0.0609 (11)	0.0575 (11)	−0.0052 (8)	0.0085 (8)	0.0019 (8)
C8	0.0378 (8)	0.0581 (10)	0.0555 (10)	−0.0050 (7)	0.0027 (7)	−0.0004 (8)
C9	0.0421 (9)	0.0606 (11)	0.0671 (11)	−0.0022 (8)	0.0060 (8)	−0.0052 (9)
C10	0.0516 (11)	0.0749 (13)	0.0711 (13)	−0.0013 (9)	0.0088 (9)	−0.0118 (10)
C11	0.0517 (11)	0.0813 (14)	0.0602 (11)	0.0098 (9)	0.0067 (9)	−0.0021 (10)
C12	0.130 (2)	0.0959 (19)	0.0772 (16)	0.0241 (17)	0.0451 (16)	0.0052 (14)
C13	0.0471 (12)	0.196 (4)	0.0931 (19)	0.0094 (16)	0.0133 (12)	0.017 (2)
C14	0.106 (2)	0.0614 (13)	0.1019 (19)	0.0060 (13)	0.0050 (16)	−0.0115 (13)
C15	0.0618 (11)	0.0609 (11)	0.0447 (10)	−0.0017 (9)	0.0053 (8)	−0.0072 (8)
C16	0.0666 (11)	0.0443 (9)	0.0460 (9)	−0.0062 (8)	0.0074 (8)	0.0000 (7)

Geometric parameters (Å, º)

O1—C6	1.382 (2)	C5—C6	1.396 (3)
O1—C14	1.410 (3)	C6—C7	1.375 (3)
O3—C15	1.222 (2)	C7—H7	0.9300
O2—C15	1.225 (2)	C7—C8	1.392 (3)
N1—H1	0.870 (10)	C8—C9	1.431 (3)
N1—C2	1.375 (3)	C9—C10	1.490 (3)
N1—C3	1.376 (3)	C10—H10A	0.9700
N2—H2	0.876 (10)	C10—H10B	0.9700
N2—C11	1.469 (3)	C10—C11	1.523 (3)
N2—C12	1.485 (3)	C11—H11A	0.9700
N2—C13	1.465 (3)	C11—H11B	0.9700
C1—H1A	0.9600	C12—H12A	0.9600
C1—H1B	0.9600	C12—H12B	0.9600

C1—H1C	0.9600	C12—H12C	0.9600
C1—H1D	0.9600	C13—H13A	0.9600
C1—H1E	0.9600	C13—H13B	0.9600
C1—H1F	0.9600	C13—H13C	0.9600
C1—C2	1.500 (3)	C14—H14A	0.9600
C2—C9	1.368 (3)	C14—H14B	0.9600
C3—C4	1.378 (3)	C14—H14C	0.9600
C3—C8	1.410 (3)	C15—C16	1.490 (2)
C4—H4	0.9300	C16—C16^{i}	1.299 (3)
C4—C5	1.378 (3)	C16—H16	0.9300
C5—H5	0.9300		

C6—O1—C14	118.16 (19)	C7—C8—C3	119.16 (18)
C2—N1—H1	125.5 (17)	C7—C8—C9	134.02 (18)
C2—N1—C3	108.90 (17)	C2—C9—C8	106.98 (18)
C3—N1—H1	124.8 (17)	C2—C9—C10	126.55 (19)
C11—N2—H2	107.6 (16)	C8—C9—C10	126.46 (18)
C11—N2—C12	112.47 (19)	C9—C10—H10A	109.7
C12—N2—H2	109.6 (16)	C9—C10—H10B	109.7
C13—N2—H2	105.0 (16)	C9—C10—C11	109.91 (17)
C13—N2—C11	111.93 (19)	H10A—C10—H10B	108.2
C13—N2—C12	109.9 (2)	C11—C10—H10A	109.7
H1A—C1—H1B	109.5	C11—C10—H10B	109.7
H1A—C1—H1C	109.5	N2—C11—C10	113.05 (17)
H1B—C1—H1C	109.5	N2—C11—H11A	109.0
H1D—C1—H1E	109.5	N2—C11—H11B	109.0
H1D—C1—H1F	109.5	C10—C11—H11A	109.0
H1E—C1—H1F	109.5	C10—C11—H11B	109.0
C2—C1—H1A	109.5	H11A—C11—H11B	107.8
C2—C1—H1B	109.5	N2—C12—H12A	109.5
C2—C1—H1C	109.5	N2—C12—H12B	109.5
C2—C1—H1D	109.5	N2—C12—H12C	109.5
C2—C1—H1E	109.5	H12A—C12—H12B	109.5
C2—C1—H1F	109.5	H12A—C12—H12C	109.5
N1—C2—C1	120.5 (2)	H12B—C12—H12C	109.5
C9—C2—N1	109.64 (18)	N2—C13—H13A	109.5
C9—C2—C1	129.9 (2)	N2—C13—H13B	109.5
N1—C3—C4	130.75 (19)	N2—C13—H13C	109.5
N1—C3—C8	107.66 (18)	H13A—C13—H13B	109.5
C4—C3—C8	121.57 (18)	H13A—C13—H13C	109.5
C3—C4—H4	120.8	H13B—C13—H13C	109.5
C5—C4—C3	118.46 (19)	O1—C14—H14A	109.5
C5—C4—H4	120.8	O1—C14—H14B	109.5
C4—C5—H5	119.7	O1—C14—H14C	109.5
C4—C5—C6	120.6 (2)	H14A—C14—H14B	109.5
C6—C5—H5	119.7	H14A—C14—H14C	109.5
O1—C6—C5	122.99 (19)	H14B—C14—H14C	109.5
C7—C6—O1	115.82 (18)	O3—C15—O2	122.43 (19)

C7—C6—C5	121.19 (19)	O3—C15—C16	119.32 (17)
C6—C7—H7	120.5	O2—C15—C16	118.16 (18)
C6—C7—C8	118.98 (18)	C15—C16—H16	117.4
C8—C7—H7	120.5	C16^{i}—C16—C15	125.2 (2)
C3—C8—C9	106.82 (17)	C16^{i}—C16—H16	117.4
O1—C6—C7—C8	178.56 (17)	C3—C8—C9—C10	−179.36 (17)
O3—C15—C16—C16^{i}	−17.2 (4)	C4—C3—C8—C7	0.1 (3)
O2—C15—C16—C16^{i}	159.4 (3)	C4—C3—C8—C9	−179.39 (17)
N1—C2—C9—C8	0.7 (2)	C4—C5—C6—O1	−179.70 (19)
N1—C2—C9—C10	−179.92 (18)	C4—C5—C6—C7	0.6 (3)
N1—C3—C4—C5	−179.6 (2)	C5—C6—C7—C8	−1.7 (3)
N1—C3—C8—C7	178.78 (16)	C6—C7—C8—C3	1.4 (3)
N1—C3—C8—C9	−0.70 (19)	C6—C7—C8—C9	−179.32 (18)
C1—C2—C9—C8	179.4 (2)	C7—C8—C9—C2	−179.38 (19)
C1—C2—C9—C10	−1.3 (3)	C7—C8—C9—C10	1.3 (3)
C2—N1—C3—C4	179.7 (2)	C8—C3—C4—C5	−1.2 (3)
C2—N1—C3—C8	1.2 (2)	C8—C9—C10—C11	83.8 (2)
C2—C9—C10—C11	−95.4 (2)	C9—C10—C11—N2	−175.81 (18)
C3—N1—C2—C1	−180.0 (2)	C12—N2—C11—C10	−63.3 (2)
C3—N1—C2—C9	−1.2 (2)	C13—N2—C11—C10	172.4 (2)
C3—C4—C5—C6	0.9 (3)	C14—O1—C6—C5	8.0 (3)
C3—C8—C9—C2	0.0 (2)	C14—O1—C6—C7	−172.3 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.87 (1)	1.95 (1)	2.810 (3)	168 (2)
N2—H2···O3^{ii}	0.88 (1)	2.18 (2)	2.892 (2)	138 (2)
N2—H2···O2^{ii}	0.88 (1)	2.00 (1)	2.837 (2)	160 (2)

Symmetry code: (ii) *x*+1/2, −*y*+1/2, *z*−1/2.

[2-(5-Methoxy-2-methyl-1*H*-indol-3-yl)ethyl]trimethylazanium iodide (umd2018f_a)

Crystal data

$C_{15}H_{23}N_2O^+\cdot I^-$
M_r = 374.25
Monoclinic, $P2_1/n$
a = 7.5067 (8) Å
b = 22.657 (3) Å
c = 10.0894 (11) Å
β = 97.225 (4)°
V = 1702.4 (3) Å^3
Z = 4
$F(000)$ = 752
D_x = 1.460 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9773 reflections
θ = 2.9–25.7°
μ = 1.88 mm^{-1}
T = 297 K
PLATE, colourless
0.43 × 0.20 × 0.03 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2018)
T_{min} = 0.621, T_{max} = 0.745
40959 measured reflections
3207 independent reflections
2875 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.7°, θ_{min} = 2.7°
h = −9→8
k = −27→27
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 1.17
3207 reflections
180 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 2.306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.43011 (3)	0.33522 (2)	0.91956 (2)	0.05592 (9)
O1	0.1841 (4)	0.49375 (15)	0.1758 (4)	0.1013 (11)
N1	0.5763 (6)	0.39020 (16)	0.6094 (3)	0.0793 (11)
H1	0.563 (6)	0.3830 (19)	0.6915 (17)	0.095*
N2	0.9563 (3)	0.31308 (11)	0.1516 (3)	0.0481 (6)
C1	0.5377 (4)	0.41400 (14)	0.3926 (4)	0.0538 (8)
C2	0.4496 (5)	0.43986 (15)	0.2768 (4)	0.0596 (9)
H2	0.499719	0.438575	0.197133	0.072*
C3	0.2864 (5)	0.46743 (17)	0.2829 (5)	0.0763 (12)
C4	0.2127 (6)	0.4695 (2)	0.4038 (6)	0.0954 (18)
H4	0.103476	0.488605	0.406264	0.114*
C5	0.2955 (6)	0.4446 (2)	0.5172 (6)	0.0921 (17)
H5	0.244398	0.446392	0.596395	0.110*
C6	0.4595 (6)	0.41628 (17)	0.5120 (4)	0.0687 (11)
C7	0.7278 (6)	0.37181 (16)	0.5563 (4)	0.0676 (10)
C8	0.7079 (5)	0.38575 (14)	0.4236 (3)	0.0529 (8)
C9	0.8472 (4)	0.37817 (15)	0.3310 (3)	0.0551 (8)
H9A	0.964663	0.375543	0.383112	0.066*
H9B	0.846742	0.412634	0.273907	0.066*
C10	0.8157 (4)	0.32358 (13)	0.2446 (3)	0.0422 (6)
H10A	0.699126	0.326878	0.191522	0.051*
H10B	0.812240	0.289466	0.302324	0.051*

C11	1.1387 (4)	0.30522 (18)	0.2286 (4)	0.0657 (10)
H11A	1.172879	0.340747	0.277101	0.099*
H11B	1.224149	0.296830	0.167952	0.099*
H11C	1.135847	0.273038	0.290226	0.099*
C12	0.9611 (5)	0.36360 (16)	0.0563 (3)	0.0615 (9)
H12A	1.000613	0.398594	0.104965	0.092*
H12B	0.843013	0.369957	0.009750	0.092*
H12C	1.042595	0.354613	−0.006982	0.092*
C13	0.9054 (6)	0.25820 (16)	0.0735 (4)	0.0669 (10)
H13A	0.991867	0.250697	0.013065	0.100*
H13B	0.788748	0.263219	0.023630	0.100*
H13C	0.902829	0.225467	0.133626	0.100*
C14	0.2614 (7)	0.4976 (2)	0.0551 (6)	0.1045 (17)
H14A	0.175989	0.514516	−0.013254	0.157*
H14B	0.293959	0.458831	0.028158	0.157*
H14C	0.366534	0.522046	0.068418	0.157*
C15	0.8829 (8)	0.3443 (2)	0.6421 (5)	0.1011 (16)
H15A	0.928146	0.311742	0.595332	0.152*
H15B	0.843973	0.330378	0.723583	0.152*
H15C	0.976182	0.373088	0.662330	0.152*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05627 (14)	0.06367 (15)	0.05083 (13)	−0.00004 (11)	0.01850 (9)	−0.00788 (10)
O1	0.0650 (18)	0.091 (2)	0.143 (3)	0.0200 (17)	−0.007 (2)	−0.037 (2)
N1	0.113 (3)	0.077 (2)	0.056 (2)	−0.041 (2)	0.040 (2)	−0.0190 (18)
N2	0.0462 (14)	0.0540 (15)	0.0465 (14)	0.0096 (12)	0.0151 (11)	0.0100 (12)
C1	0.0515 (19)	0.0489 (18)	0.065 (2)	−0.0174 (15)	0.0235 (16)	−0.0221 (16)
C2	0.0524 (19)	0.055 (2)	0.074 (2)	−0.0069 (16)	0.0175 (17)	−0.0222 (18)
C3	0.051 (2)	0.062 (2)	0.116 (4)	−0.0094 (18)	0.014 (2)	−0.040 (2)
C4	0.055 (2)	0.083 (3)	0.155 (5)	−0.022 (2)	0.041 (3)	−0.071 (3)
C5	0.078 (3)	0.090 (3)	0.122 (4)	−0.044 (3)	0.062 (3)	−0.066 (3)
C6	0.074 (3)	0.064 (2)	0.075 (3)	−0.035 (2)	0.039 (2)	−0.035 (2)
C7	0.089 (3)	0.056 (2)	0.059 (2)	−0.027 (2)	0.016 (2)	−0.0099 (17)
C8	0.059 (2)	0.0481 (18)	0.0541 (19)	−0.0156 (15)	0.0167 (15)	−0.0122 (15)
C9	0.0495 (18)	0.0537 (19)	0.064 (2)	−0.0099 (15)	0.0136 (15)	−0.0049 (16)
C10	0.0345 (14)	0.0490 (17)	0.0465 (16)	0.0011 (12)	0.0180 (12)	0.0028 (13)
C11	0.0437 (18)	0.082 (3)	0.073 (2)	0.0165 (17)	0.0138 (16)	0.023 (2)
C12	0.060 (2)	0.072 (2)	0.057 (2)	0.0108 (17)	0.0217 (16)	0.0255 (17)
C13	0.086 (3)	0.060 (2)	0.057 (2)	0.0162 (19)	0.0179 (19)	−0.0047 (17)
C14	0.091 (4)	0.066 (3)	0.150 (5)	0.013 (3)	−0.011 (4)	0.014 (3)
C15	0.132 (4)	0.096 (4)	0.071 (3)	−0.022 (3)	−0.006 (3)	0.013 (3)

Geometric parameters (Å, º)

O1—C3	1.380 (5)	C8—C9	1.497 (4)
O1—C14	1.416 (6)	C9—H9A	0.9700

N1—H1	0.863 (10)	C9—H9B	0.9700
N1—C6	1.367 (6)	C9—C10	1.514 (4)
N1—C7	1.381 (5)	C10—H10A	0.9700
N2—C10	1.517 (3)	C10—H10B	0.9700
N2—C11	1.497 (4)	C11—H11A	0.9600
N2—C12	1.498 (4)	C11—H11B	0.9600
N2—C13	1.496 (4)	C11—H11C	0.9600
C1—C2	1.397 (5)	C12—H12A	0.9600
C1—C6	1.406 (5)	C12—H12B	0.9600
C1—C8	1.428 (5)	C12—H12C	0.9600
C2—H2	0.9300	C13—H13A	0.9600
C2—C3	1.383 (5)	C13—H13B	0.9600
C3—C4	1.403 (7)	C13—H13C	0.9600
C4—H4	0.9300	C14—H14A	0.9600
C4—C5	1.354 (7)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C5—C6	1.396 (6)	C15—H15A	0.9600
C7—C8	1.365 (5)	C15—H15B	0.9600
C7—C15	1.497 (6)	C15—H15C	0.9600

C3—O1—C14	116.9 (3)	C10—C9—H9A	109.1
C6—N1—H1	129 (3)	C10—C9—H9B	109.1
C6—N1—C7	109.6 (3)	N2—C10—H10A	108.6
C7—N1—H1	121 (3)	N2—C10—H10B	108.6
C11—N2—C10	111.0 (2)	C9—C10—N2	114.5 (2)
C11—N2—C12	109.3 (3)	C9—C10—H10A	108.6
C12—N2—C10	110.6 (2)	C9—C10—H10B	108.6
C13—N2—C10	107.8 (2)	H10A—C10—H10B	107.6
C13—N2—C11	109.3 (3)	N2—C11—H11A	109.5
C13—N2—C12	108.8 (3)	N2—C11—H11B	109.5
C2—C1—C6	119.8 (4)	N2—C11—H11C	109.5
C2—C1—C8	133.4 (3)	H11A—C11—H11B	109.5
C6—C1—C8	106.7 (4)	H11A—C11—H11C	109.5
C1—C2—H2	120.6	H11B—C11—H11C	109.5
C3—C2—C1	118.8 (4)	N2—C12—H12A	109.5
C3—C2—H2	120.6	N2—C12—H12B	109.5
O1—C3—C2	124.7 (4)	N2—C12—H12C	109.5
O1—C3—C4	115.2 (4)	H12A—C12—H12B	109.5
C2—C3—C4	120.1 (5)	H12A—C12—H12C	109.5
C3—C4—H4	119.0	H12B—C12—H12C	109.5
C5—C4—C3	122.1 (4)	N2—C13—H13A	109.5
C5—C4—H4	119.0	N2—C13—H13B	109.5
C4—C5—H5	120.9	N2—C13—H13C	109.5
C4—C5—C6	118.3 (4)	H13A—C13—H13B	109.5
C6—C5—H5	120.9	H13A—C13—H13C	109.5
N1—C6—C1	107.6 (4)	H13B—C13—H13C	109.5
N1—C6—C5	131.4 (4)	O1—C14—H14A	109.5
C5—C6—C1	120.9 (5)	O1—C14—H14B	109.5

N1—C7—C15	121.3 (4)	O1—C14—H14C	109.5
C8—C7—N1	108.5 (4)	H14A—C14—H14B	109.5
C8—C7—C15	130.1 (4)	H14A—C14—H14C	109.5
C1—C8—C9	126.2 (3)	H14B—C14—H14C	109.5
C7—C8—C1	107.6 (3)	C7—C15—H15A	109.5
C7—C8—C9	125.9 (4)	C7—C15—H15B	109.5
C8—C9—H9A	109.1	C7—C15—H15C	109.5
C8—C9—H9B	109.1	H15A—C15—H15B	109.5
C8—C9—C10	112.5 (2)	H15A—C15—H15C	109.5
H9A—C9—H9B	107.8	H15B—C15—H15C	109.5

O1—C3—C4—C5	178.7 (4)	C6—C1—C8—C7	0.3 (4)
N1—C7—C8—C1	0.1 (4)	C6—C1—C8—C9	−174.2 (3)
N1—C7—C8—C9	174.5 (3)	C7—N1—C6—C1	0.6 (4)
C1—C2—C3—O1	−178.8 (3)	C7—N1—C6—C5	−175.7 (4)
C1—C2—C3—C4	0.5 (5)	C7—C8—C9—C10	100.9 (4)
C1—C8—C9—C10	−85.7 (4)	C8—C1—C2—C3	−175.7 (3)
C2—C1—C6—N1	−177.4 (3)	C8—C1—C6—N1	−0.5 (4)
C2—C1—C6—C5	−0.7 (5)	C8—C1—C6—C5	176.2 (3)
C2—C1—C8—C7	176.6 (3)	C8—C9—C10—N2	−178.5 (3)
C2—C1—C8—C9	2.1 (6)	C11—N2—C10—C9	60.6 (3)
C2—C3—C4—C5	−0.7 (6)	C12—N2—C10—C9	−60.8 (4)
C3—C4—C5—C6	0.2 (6)	C13—N2—C10—C9	−179.7 (3)
C4—C5—C6—N1	176.4 (4)	C14—O1—C3—C2	−6.5 (6)
C4—C5—C6—C1	0.5 (6)	C14—O1—C3—C4	174.1 (4)
C6—N1—C7—C8	−0.4 (4)	C15—C7—C8—C1	−177.0 (4)
C6—N1—C7—C15	177.0 (3)	C15—C7—C8—C9	−2.6 (6)
C6—C1—C2—C3	0.2 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1	0.86 (1)	2.83 (2)	3.662 (3)	161 (4)

[2-(5-Methoxy-2-methyl-1*H*-indol-3-yl)ethyl]trimethylazanium iodide monohydrate (umd2009b_a)

Crystal data

$C_{15}H_{23}N_2O^+·I^-·H_2O$
M_r = 392.27
Monoclinic, $P2_1/c$
a = 10.9091 (10) Å
b = 14.0910 (11) Å
c = 11.4029 (10) Å
β = 100.338 (3)°
V = 1724.4 (3) Å^3
Z = 4
$F(000)$ = 792
D_x = 1.511 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9808 reflections
θ = 2.8–26.0°
μ = 1.86 mm^{-1}
T = 297 K
BLOCK, colourless
0.38 × 0.22 × 0.20 mm

Data collection

Bruker D8 Venture CMOS diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2018)
T_{min} = 0.486, T_{max} = 0.562
40738 measured reflections
3326 independent reflections
3051 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.1°, θ_{min} = 3.8°
h = −13→13
k = −17→17
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.054
S = 1.10
3326 reflections
195 parameters
3 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 0.9135P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.66404 (2)	0.98962 (2)	0.30669 (2)	0.05629 (7)
O1	−0.06415 (16)	0.35370 (12)	0.19831 (14)	0.0645 (4)
O1W	0.3969 (2)	0.90251 (15)	0.4217 (2)	0.0846 (6)
H1WA	0.384 (4)	0.937 (3)	0.483 (2)	0.127*
H1WB	0.465 (2)	0.931 (3)	0.407 (4)	0.127*
N1	0.21469 (17)	0.25708 (12)	0.63123 (17)	0.0499 (4)
H1	0.231 (2)	0.2044 (11)	0.6685 (19)	0.060*
N2	0.35006 (14)	0.65530 (10)	0.53057 (13)	0.0379 (3)
C1	0.25645 (18)	0.34390 (14)	0.67593 (18)	0.0445 (4)
C2	0.14222 (18)	0.26828 (13)	0.52036 (18)	0.0432 (4)
C3	0.0771 (2)	0.20291 (14)	0.4425 (2)	0.0544 (5)
H3	0.078699	0.138686	0.461520	0.065*
C4	0.0105 (2)	0.23508 (15)	0.3370 (2)	0.0551 (5)
H4	−0.034166	0.192097	0.283837	0.066*
C5	0.00816 (18)	0.33197 (15)	0.30727 (18)	0.0468 (4)
C6	0.07209 (17)	0.39779 (13)	0.38389 (17)	0.0414 (4)
H6	0.070468	0.461808	0.363818	0.050*
C7	0.14001 (16)	0.36583 (12)	0.49342 (17)	0.0378 (4)
C8	0.21289 (16)	0.41246 (13)	0.59414 (17)	0.0391 (4)
C9	0.23415 (19)	0.51747 (13)	0.60723 (19)	0.0421 (4)
H9A	0.253828	0.533648	0.691189	0.051*
H9B	0.158531	0.550853	0.572574	0.051*

C10	0.33991 (16)	0.54890 (12)	0.54616 (16)	0.0354 (4)
H10A	0.417725	0.525925	0.592161	0.042*
H10B	0.329195	0.519225	0.468226	0.042*
C11	0.2417 (2)	0.69182 (16)	0.4424 (2)	0.0589 (6)
H11A	0.254727	0.757586	0.426376	0.088*
H11B	0.166686	0.685097	0.474590	0.088*
H11C	0.234330	0.656169	0.369714	0.088*
C12	0.3573 (2)	0.70577 (15)	0.6465 (2)	0.0548 (5)
H12A	0.370823	0.772233	0.635447	0.082*
H12B	0.425002	0.680458	0.703390	0.082*
H12C	0.280615	0.697125	0.675441	0.082*
C13	0.4675 (2)	0.67358 (16)	0.4820 (2)	0.0553 (5)
H13A	0.475864	0.740446	0.469415	0.083*
H13B	0.463369	0.640606	0.407765	0.083*
H13C	0.538030	0.651344	0.538002	0.083*
C14	0.3351 (2)	0.35188 (18)	0.7972 (2)	0.0608 (6)
H14A	0.313836	0.408931	0.835009	0.091*
H14B	0.421438	0.353824	0.790177	0.091*
H14C	0.320503	0.297993	0.844379	0.091*
C15	−0.0569 (2)	0.44733 (19)	0.1541 (2)	0.0610 (6)
H15A	−0.107259	0.451967	0.076067	0.092*
H15B	0.028092	0.461878	0.149538	0.092*
H15C	−0.086653	0.491467	0.206657	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05807 (11)	0.05975 (11)	0.05273 (10)	0.01395 (6)	0.01446 (7)	0.00736 (6)
O1	0.0638 (10)	0.0650 (10)	0.0573 (9)	−0.0043 (8)	−0.0091 (8)	−0.0007 (8)
O1W	0.1088 (17)	0.0667 (12)	0.0840 (14)	−0.0251 (11)	0.0323 (12)	−0.0142 (10)
N1	0.0541 (10)	0.0356 (8)	0.0590 (11)	0.0022 (7)	0.0080 (8)	0.0119 (7)
N2	0.0402 (8)	0.0314 (7)	0.0412 (8)	0.0007 (6)	0.0052 (6)	0.0012 (6)
C1	0.0367 (9)	0.0451 (10)	0.0524 (11)	0.0013 (8)	0.0102 (8)	0.0061 (8)
C2	0.0436 (10)	0.0346 (9)	0.0530 (11)	0.0008 (8)	0.0134 (8)	0.0047 (8)
C3	0.0621 (13)	0.0328 (9)	0.0699 (14)	−0.0055 (9)	0.0155 (11)	−0.0010 (9)
C4	0.0550 (12)	0.0449 (11)	0.0643 (14)	−0.0095 (9)	0.0076 (10)	−0.0119 (10)
C5	0.0408 (10)	0.0510 (11)	0.0488 (11)	−0.0003 (8)	0.0086 (8)	−0.0020 (9)
C6	0.0381 (9)	0.0365 (9)	0.0507 (11)	0.0002 (7)	0.0110 (8)	0.0038 (8)
C7	0.0340 (9)	0.0328 (9)	0.0488 (10)	−0.0005 (7)	0.0135 (8)	0.0014 (7)
C8	0.0358 (9)	0.0367 (9)	0.0468 (10)	−0.0016 (7)	0.0128 (8)	0.0015 (7)
C9	0.0436 (10)	0.0358 (9)	0.0493 (11)	−0.0011 (7)	0.0146 (8)	−0.0033 (8)
C10	0.0370 (9)	0.0282 (8)	0.0411 (9)	0.0017 (7)	0.0074 (7)	−0.0007 (7)
C11	0.0586 (13)	0.0497 (12)	0.0625 (13)	0.0088 (10)	−0.0054 (11)	0.0158 (10)
C12	0.0716 (14)	0.0403 (10)	0.0530 (12)	−0.0058 (10)	0.0124 (10)	−0.0114 (9)
C13	0.0546 (12)	0.0456 (11)	0.0698 (14)	−0.0080 (9)	0.0220 (11)	0.0031 (10)
C14	0.0528 (13)	0.0664 (14)	0.0596 (13)	−0.0004 (11)	0.0003 (10)	0.0108 (11)
C15	0.0559 (13)	0.0749 (16)	0.0513 (12)	0.0025 (11)	0.0071 (10)	0.0113 (11)

Geometric parameters (Å, º)

O1—C5	1.381 (3)	C7—C8	1.434 (3)
O1—C15	1.420 (3)	C8—C9	1.501 (2)
O1W—H1WA	0.888 (10)	C9—H9A	0.9700
O1W—H1WB	0.886 (10)	C9—H9B	0.9700
N1—H1	0.858 (10)	C9—C10	1.517 (3)
N1—C1	1.371 (3)	C10—H10A	0.9700
N1—C2	1.375 (3)	C10—H10B	0.9700
N2—C10	1.516 (2)	C11—H11A	0.9600
N2—C11	1.499 (2)	C11—H11B	0.9600
N2—C12	1.491 (2)	C11—H11C	0.9600
N2—C13	1.506 (2)	C12—H12A	0.9600
C1—C8	1.367 (3)	C12—H12B	0.9600
C1—C14	1.495 (3)	C12—H12C	0.9600
C2—C3	1.384 (3)	C13—H13A	0.9600
C2—C7	1.408 (2)	C13—H13B	0.9600
C3—H3	0.9300	C13—H13C	0.9600
C3—C4	1.367 (3)	C14—H14A	0.9600
C4—H4	0.9300	C14—H14B	0.9600
C4—C5	1.406 (3)	C14—H14C	0.9600
C5—C6	1.375 (3)	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
C6—C7	1.407 (3)	C15—H15C	0.9600
C5—O1—C15	117.80 (17)	C10—C9—H9A	109.4
H1WA—O1W—H1WB	99 (4)	C10—C9—H9B	109.4
C1—N1—H1	124.2 (17)	N2—C10—C9	114.85 (14)
C1—N1—C2	109.71 (16)	N2—C10—H10A	108.6
C2—N1—H1	126.1 (17)	N2—C10—H10B	108.6
C11—N2—C10	110.66 (15)	C9—C10—H10A	108.6
C11—N2—C13	108.37 (17)	C9—C10—H10B	108.6
C12—N2—C10	111.16 (14)	H10A—C10—H10B	107.5
C12—N2—C11	109.93 (17)	N2—C11—H11A	109.5
C12—N2—C13	109.35 (16)	N2—C11—H11B	109.5
C13—N2—C10	107.28 (14)	N2—C11—H11C	109.5
N1—C1—C14	120.53 (18)	H11A—C11—H11B	109.5
C8—C1—N1	108.97 (17)	H11A—C11—H11C	109.5
C8—C1—C14	130.49 (19)	H11B—C11—H11C	109.5
N1—C2—C3	131.14 (18)	N2—C12—H12A	109.5
N1—C2—C7	107.29 (17)	N2—C12—H12B	109.5
C3—C2—C7	121.55 (19)	N2—C12—H12C	109.5
C2—C3—H3	120.8	H12A—C12—H12B	109.5
C4—C3—C2	118.35 (19)	H12A—C12—H12C	109.5
C4—C3—H3	120.8	H12B—C12—H12C	109.5
C3—C4—H4	119.4	N2—C13—H13A	109.5
C3—C4—C5	121.18 (19)	N2—C13—H13B	109.5
C5—C4—H4	119.4	N2—C13—H13C	109.5

O1—C5—C4	114.49 (19)	H13A—C13—H13B	109.5
C6—C5—O1	124.38 (19)	H13A—C13—H13C	109.5
C6—C5—C4	121.12 (19)	H13B—C13—H13C	109.5
C5—C6—H6	120.8	C1—C14—H14A	109.5
C5—C6—C7	118.31 (17)	C1—C14—H14B	109.5
C7—C6—H6	120.8	C1—C14—H14C	109.5
C2—C7—C8	106.70 (17)	H14A—C14—H14B	109.5
C6—C7—C2	119.47 (17)	H14A—C14—H14C	109.5
C6—C7—C8	133.83 (17)	H14B—C14—H14C	109.5
C1—C8—C7	107.31 (16)	O1—C15—H15A	109.5
C1—C8—C9	126.88 (18)	O1—C15—H15B	109.5
C7—C8—C9	125.80 (17)	O1—C15—H15C	109.5
C8—C9—H9A	109.4	H15A—C15—H15B	109.5
C8—C9—H9B	109.4	H15A—C15—H15C	109.5
C8—C9—C10	111.03 (15)	H15B—C15—H15C	109.5
H9A—C9—H9B	108.0		

O1—C5—C6—C7	178.59 (18)	C3—C4—C5—C6	−0.6 (3)
N1—C1—C8—C7	0.1 (2)	C4—C5—C6—C7	−0.1 (3)
N1—C1—C8—C9	178.83 (17)	C5—C6—C7—C2	1.0 (3)
N1—C2—C3—C4	179.1 (2)	C5—C6—C7—C8	−177.98 (19)
N1—C2—C7—C6	179.87 (17)	C6—C7—C8—C1	179.6 (2)
N1—C2—C7—C8	−0.9 (2)	C6—C7—C8—C9	0.8 (3)
C1—N1—C2—C3	−177.8 (2)	C7—C2—C3—C4	0.5 (3)
C1—N1—C2—C7	1.0 (2)	C7—C8—C9—C10	−83.5 (2)
C1—C8—C9—C10	98.0 (2)	C8—C9—C10—N2	167.09 (15)
C2—N1—C1—C8	−0.7 (2)	C11—N2—C10—C9	−68.4 (2)
C2—N1—C1—C14	178.50 (19)	C12—N2—C10—C9	54.1 (2)
C2—C3—C4—C5	0.3 (3)	C13—N2—C10—C9	173.61 (16)
C2—C7—C8—C1	0.5 (2)	C14—C1—C8—C7	−179.0 (2)
C2—C7—C8—C9	−178.25 (17)	C14—C1—C8—C9	−0.3 (3)
C3—C2—C7—C6	−1.2 (3)	C15—O1—C5—C4	−171.0 (2)
C3—C2—C7—C8	178.00 (18)	C15—O1—C5—C6	10.2 (3)
C3—C4—C5—O1	−179.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WA*···I1^{i}	0.89 (1)	2.74 (1)	3.617 (2)	168 (4)
O1*W*—H1*WB*···I1	0.89 (1)	2.76 (2)	3.618 (2)	164 (4)
N1—H1···I1^{ii}	0.86 (1)	2.96 (1)	3.7416 (17)	153 (2)

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