N,N'-Dibenzyl-N,N,N',N'-tetramethyl-ethylenediammonium dibromide dihydrate

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In the title compound, $C_{20}H_{30}N_2{}^{2+}\cdot 2Br^-\cdot 2H_2O$, the asymmetric unit consists of half of the N,N'-dibenzyl-N,N,N',N'-tetramethylethylenediammonium cation lying across an inversion center, a bromide ion and a water molecule of solvation. There is an eight-membered dibromide dihydrate ring, which is formed *via* hydrogen bonds of the type O—H···Br.

Related literature

For related literature, see: Chen *et al.* (2006); Jayaraman *et al.* (2002); Kabak *et al.* (2000); Li *et al.* (2006); Mathew *et al.* (2002); Misra *et al.* (2007); Nastase *et al.* (2007); Pan *et al.* (2007); Srinivasan *et al.* (2003, 2005, 2007).
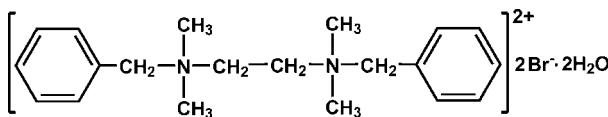
Experimental

Crystal data

$C_{20}H_{30}N_2{}^{2+}\cdot 2Br^-\cdot 2H_2O$
$M_r = 494.31$
Monoclinic, $P2_1/n$
$a = 6.7897 (14)$ Å
$b = 22.774 (5)$ Å
$c = 7.7069 (15)$ Å
$\beta = 110.15 (3)°$

$V = 1118.8 (5)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 3.64$ mm^{-1}
$T = 298 (2)$ K
$0.30 \times 0.20 \times 0.15$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*RAPID-AUTO*; Rigaku

Corporation, 1998)
$T_{min} = 0.421$, $T_{max} = 0.582$
10562 measured reflections

2547 independent reflections
1792 reflections with $I > 2.0 \sigma(I)$
$R_{int} = 0.074$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.126$
$S = 1.06$
2547 reflections
184 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
OW1—HWA···Br1^{i}	0.86 (3)	2.434 (11)	3.295 (4)	177 (4)
OW1—HWB···Br1^{ii}	0.86 (3)	2.456 (14)	3.304 (4)	170 (5)

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

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL97*.

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supporting information

N,*N*′-Dibenzyl-*N*,*N*,*N*′,*N*′-tetramethylethylenediammonium dibromide dihydrate

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S1. Comment

It is well known that organic amines, such as ethylenediamine (en), 1,3-propanediamine (1,3-pnen), *N*,*N*,*N*′,*N*′-tetra-methylethylenediamine (tmen), piperazine (pip), *etc*, have been widely used as structure-directing agents for the construction of novel supramolecular assemblies (Misra *et al.*, 2007, Nastase *et al.*, 2007, Pan *et al.*, 2007, Srinivasan *et al.*, 2007, Li *et al.*, 2006, Chen *et al.*, 2006, Mathew *et al.*, 2002, Jayaraman *et al.*, 2002, Kabak *et al.*, 2000). In a search for more organic surpramolecur amines, we have synthesized the title compound, (I), the structure of which is presented in this paper.

In the structure of (I), the asymmetric unit consists of a half molecule of *N*,*N*′-dibenzyl-*N*,*N*,*N*′,*N*′-tetramethylethylenedi-ammonium cation lying about an inversion center, a bromide ion and a water of solvation (Fig. 1). The C—C and C—N bond lengths are in good agreement with those found in other compounds containing the tmen moiety (Srinivasan *et al.*, 2003; 2005; 2007). The water molecules in (I) are hydrogen-bonded with two bromide ions forming a novel eight membered cyclic dibromide. The two phenyl rings in each cation lie parallel to each other. The phenyl rings are arranged in layers. The dihedral angle between the two pheny rings is 16.3 (5)°. The shortest distance between adjacent phenyl rings is about 3.6441 Å, which indicates the existence of stacking interactions (Fig. 2).

S2. Experimental

Tetramethylethylenediamine (tmen) (2.6 ml, 17.23 mmol) was dissolved in 15 ml of CH$_3$CN, and benzyl bromide (4.8 ml, 40.36 mmol) was added dropwise with continuous stirring over 20–30 min. White crystaline solid was filtrated from the mixture after cooling to room temperature which was dissolved in glacial acetic acid. Colourless crystal of the title compound grew from the cetic acid solution on standing for several days at room temperature.

S3. Refinement

All hydrogen atoms were found from difference Fourier maps and were refined with isotropic displacement parameters. H atoms of the water molecule were restrained at O—H bond lengths (O—H = 0.84 (1) Å) during the refinements.
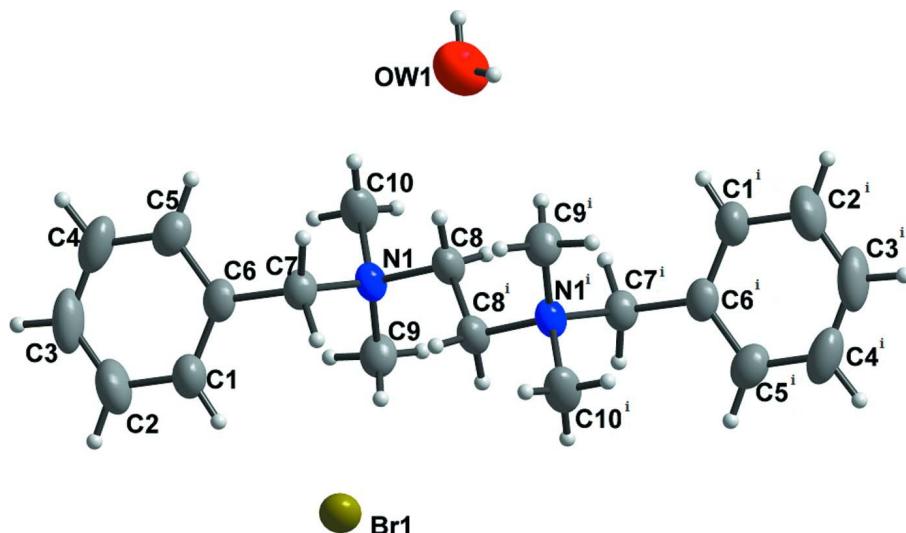
Figure 1

A view of (I) with atom labels and 50% probability displacement ellipsoids. [symmetry codes: (i) -x + 1, -y, -z + 1]
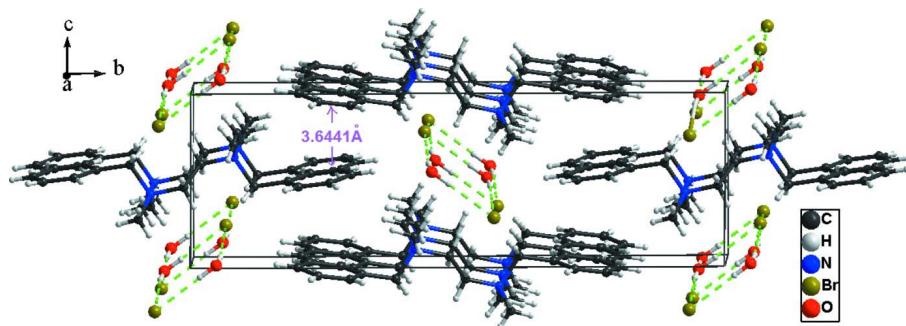
Figure 2

Packing diagram of (I) viewed down the *a* axis; H-bonding interactions are shown as dashed lines.

N,N′-Dibenzyl-N,N,N′,N′-tetramethylethylenediammonium dibromide dihydrate

Crystal data
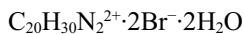
$C_{20}H_{30}N_2^{2+}·2Br^-·2H_2O$
$M_r = 494.31$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.7897$ (14) Å
$b = 22.774$ (5) Å
$c = 7.7069$ (15) Å
$β = 110.15$ (3)°
$V = 1118.8$ (5) Å^3
$Z = 2$

$F(000) = 508$
$D_x = 1.467$ Mg m^{-3}
Mo $Kα$ radiation, $λ = 0.71073$ Å
Cell parameters from 25 reflections
$θ = 12–18°$
$μ = 3.64$ mm^{-1}
$T = 298$ K
Parallelepiped, colourless
$0.30 × 0.20 × 0.15$ mm

Data collection

Rigaku R-AXIS RAPID
    diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Oscillation scans

Absorption correction: multi-scan
    (*RAPID-AUTO*; Rigaku Corporation, 1998)
$T_{min} = 0.421$, $T_{max} = 0.582$
10562 measured reflections
2547 independent reflections

1792 reflections with $I > 2.0 \sigma(I)$	$h = -8 \rightarrow 8$
$R_{int} = 0.074$	$k = -29 \rightarrow 29$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$	$l = -9 \rightarrow 10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.126$	H atoms treated by a mixture of independent
$S = 1.06$	and constrained refinement
2547 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.2784P]$
184 parameters	where $P = (F_o^2 + 2F_c^2)/3$
3 restraints	$(\Delta/\sigma)_{max} = 0.002$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.51$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -0.72$ 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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	0.89391 (7)	0.065641 (16)	0.24054 (6)	0.05367 (19)
OW1	0.2419 (7)	0.04996 (18)	1.0319 (6)	0.0917 (12)
N1	0.5346 (5)	0.07807 (11)	0.6087 (4)	0.0371 (7)
C1	0.7021 (7)	0.19997 (16)	0.4469 (6)	0.0531 (10)
C2	0.7549 (9)	0.25943 (18)	0.4717 (7)	0.0681 (13)
C3	0.6231 (10)	0.29803 (17)	0.5114 (6)	0.0705 (15)
C4	0.4387 (10)	0.27891 (18)	0.5281 (6)	0.0654 (14)
C5	0.3847 (8)	0.21992 (18)	0.5030 (6)	0.0547 (10)
C6	0.5200 (6)	0.17978 (14)	0.4658 (5)	0.0408 (8)
C7	0.4612 (6)	0.11582 (14)	0.4341 (5)	0.0404 (8)
C8	0.4532 (7)	0.01555 (14)	0.5633 (6)	0.0411 (9)
C9	0.7673 (7)	0.07937 (18)	0.6952 (7)	0.0472 (9)
C10	0.4404 (9)	0.10009 (19)	0.7456 (7)	0.0547 (12)
H1	0.801 (6)	0.1761 (16)	0.433 (5)	0.049 (11)*
H2	0.885 (9)	0.272 (2)	0.457 (7)	0.082 (17)*
H3	0.652 (7)	0.338 (2)	0.530 (6)	0.068 (13)*
H4	0.336 (7)	0.3058 (19)	0.532 (6)	0.057 (12)*
H5	0.254 (8)	0.207 (2)	0.515 (7)	0.069 (14)*
H6	0.540 (6)	0.0962 (18)	0.361 (6)	0.056 (11)*
H7	0.300 (7)	0.1093 (16)	0.378 (5)	0.051 (11)*
H8	0.306 (6)	0.0181 (16)	0.511 (5)	0.044 (11)*
H9	0.488 (6)	−0.0026 (15)	0.678 (5)	0.036 (9)*

H10	0.806 (7)	0.0550 (17)	0.791 (6)	0.051 (12)*
H11	0.823 (8)	0.0694 (18)	0.609 (7)	0.067 (15)*
H12	0.804 (7)	0.1197 (18)	0.736 (6)	0.054 (11)*
H13	0.485 (7)	0.0805 (19)	0.840 (7)	0.049 (12)*
H14	0.284 (8)	0.0988 (19)	0.684 (6)	0.058 (13)*
H15	0.487 (7)	0.137 (2)	0.777 (6)	0.057 (12)*
HWA	0.211 (6)	0.0193 (13)	0.962 (5)	0.068*
HWB	0.143 (5)	0.0568 (17)	1.074 (6)	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0612 (3)	0.0525 (3)	0.0569 (3)	0.00285 (18)	0.0325 (2)	0.00037 (16)
OW1	0.109 (3)	0.088 (3)	0.092 (3)	−0.014 (2)	0.053 (3)	−0.014 (2)
N1	0.0483 (18)	0.0294 (13)	0.0354 (16)	−0.0048 (12)	0.0166 (14)	−0.0002 (10)
C1	0.066 (3)	0.0360 (18)	0.062 (3)	0.0025 (19)	0.028 (2)	0.0058 (17)
C2	0.083 (4)	0.042 (2)	0.074 (3)	−0.013 (2)	0.021 (3)	0.008 (2)
C3	0.108 (4)	0.0284 (19)	0.061 (3)	−0.006 (2)	0.010 (3)	−0.0005 (17)
C4	0.095 (4)	0.039 (2)	0.057 (3)	0.021 (3)	0.020 (3)	0.0005 (17)
C5	0.061 (3)	0.046 (2)	0.055 (3)	0.012 (2)	0.019 (2)	0.0028 (17)
C6	0.052 (2)	0.0329 (16)	0.0333 (19)	0.0021 (15)	0.0099 (17)	0.0025 (13)
C7	0.048 (2)	0.0320 (16)	0.041 (2)	−0.0019 (15)	0.0143 (19)	−0.0020 (14)
C8	0.054 (3)	0.0313 (17)	0.045 (2)	−0.0043 (16)	0.025 (2)	−0.0008 (14)
C9	0.049 (3)	0.042 (2)	0.045 (2)	−0.0009 (17)	0.009 (2)	0.0017 (17)
C10	0.086 (4)	0.039 (2)	0.051 (3)	−0.001 (2)	0.040 (3)	−0.0040 (19)

Geometric parameters (Å, º)

OW1—HWA	0.86 (3)	C5—C6	1.394 (6)
OW1—HWB	0.86 (3)	C5—H5	0.97 (5)
N1—C9	1.488 (5)	C6—C7	1.508 (5)
N1—C10	1.496 (5)	C7—H6	1.00 (4)
N1—C8	1.524 (4)	C7—H7	1.04 (4)
N1—C7	1.528 (4)	C8—C8^i	1.511 (7)
C1—C6	1.374 (6)	C8—H8	0.94 (4)
C1—C2	1.397 (6)	C8—H9	0.93 (4)
C1—H1	0.90 (4)	C9—H10	0.89 (4)
C2—C3	1.362 (8)	C9—H11	0.90 (5)
C2—H2	0.97 (6)	C9—H12	0.97 (4)
C3—C4	1.373 (8)	C10—H13	0.82 (5)
C3—H3	0.94 (5)	C10—H14	1.00 (5)
C4—C5	1.388 (6)	C10—H15	0.90 (4)
C4—H4	0.94 (5)		
HWA—OW1—HWB	109.2 (17)	C6—C7—N1	114.4 (3)
C9—N1—C10	108.9 (3)	C6—C7—H6	111 (2)
C9—N1—C8	111.5 (3)	N1—C7—H6	100 (2)
C10—N1—C8	105.4 (3)	C6—C7—H7	113 (2)

C9—N1—C7	110.9 (3)	N1—C7—H7	105 (2)
C10—N1—C7	110.1 (3)	H6—C7—H7	113 (3)
C8—N1—C7	109.9 (3)	C8ⁱ—C8—N1	112.7 (4)
C6—C1—C2	120.5 (4)	C8ⁱ—C8—H8	112 (2)
C6—C1—H1	123 (2)	N1—C8—H8	107 (2)
C2—C1—H1	116 (2)	C8ⁱ—C8—H9	112 (2)
C3—C2—C1	119.9 (5)	N1—C8—H9	104 (2)
C3—C2—H2	122 (3)	H8—C8—H9	109 (3)
C1—C2—H2	118 (3)	N1—C9—H10	109 (3)
C2—C3—C4	120.5 (4)	N1—C9—H11	109 (3)
C2—C3—H3	123 (3)	H10—C9—H11	112 (4)
C4—C3—H3	117 (3)	N1—C9—H12	106 (3)
C3—C4—C5	120.0 (4)	H10—C9—H12	111 (4)
C3—C4—H4	121 (3)	H11—C9—H12	110 (4)
C5—C4—H4	118 (3)	N1—C10—H13	109 (3)
C4—C5—C6	120.2 (5)	N1—C10—H14	107 (3)
C4—C5—H5	119 (3)	H13—C10—H14	115 (4)
C6—C5—H5	120 (3)	N1—C10—H15	108 (3)
C1—C6—C5	118.9 (3)	H13—C10—H15	106 (4)
C1—C6—C7	120.3 (3)	H14—C10—H15	112 (4)
C5—C6—C7	120.7 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O*W*1—H*WA*⋯Br1ⁱ	0.86 (3)	2.43 (1)	3.295 (4)	177 (4)
O*W*1—H*WB*⋯Br1ⁱⁱ	0.86 (3)	2.46 (1)	3.304 (4)	170 (5)

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