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Bis(2-hydroxyethyl)(2-nitrobenzyl)ammonium Bromide

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Abstract

The title compound $C_{11}H_{17}N_2O_4^+\cdot Br^-$ exhibits both intramolecular (N—H·O) and intermolecular (O—H·Br) hydrogen bonding. The latter results in the formation of infinite one-dimensional chains of alternating cations and anions.

Experimental

The title compound was prepared by a literature method (Barbiere, 1944). Crystals were grown from a cooled ethanolic solution.

Refinement

Hydrogen atoms were placed in calculated positions and allowed to ride on their parent C or N atoms, with the exception of H11 and H17 which were located using difference circular Fourier synthesis and then allowed to ride on their parent O atoms. The maximum peak of 1.41 e $Å^{-3}$ in the final difference Fourier synthesis was located 0.98 Å from Br.

Computing details

Data collection: STADI-4 (Stoe & Cie, 1996a); cell refinement: STADI-4; data reduction: *X-RED* (Stoe & Cie, 1996b); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* version 5.03 (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

N-(2-nitrobenzyl)diethanolammonium bromide

Crystal data

$C_{11}H_{17}N_2O_4^+\cdot Br^-$	$V = 1336\ (3)\ Å^3$
$M_r = 321.18$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$
$a = 6.644\ (13)$ Å	$\mu = 3.09\ mm^{-1}$
$b = 23.030\ (4)$ Å	$T = 150\ (2)$ K
$c = 8.856\ (3)$ Å	0.48 × 0.12 × 0.08 mm
$\beta = 99.64\ (2)^\circ$	

CIF access

Data collection

Stoe Stadi-4 four-circle with Oxford Cryosystems open-flow cryostat (Cosier & Glazer, 1986) diffractometer	1786 reflections with $I > 2\sigma(I)$
Absorption correction: ψ-scans North, Phillips & Mathews, 1968	$R_{int} = 0.044$
$T_{min} = 0.655$, $T_{max} = 0.781$	3 standard reflections
2520 measured reflections	every 60 min
2357 independent reflections	intensity decay: random variation +−6.9%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$	8 restraints
$wR(F^2) = 0.117$	See text
$S = 1.03$	$\Delta\rho_{max} = 1.41$ e Å^{-3}
2357 reflections	$\Delta\rho_{min} = -0.58$ e Å^{-3}
165 parameters	

Table 1
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14A···O1	0.93	2.08	2.786 (7)	131
N14—H14A···O11	0.93	2.27	2.742 (5)	111
N14—H14A···O17	0.93	2.45	2.876 (6)	108
O11—H11···Br	0.84	2.43	3.255 (4)	168
O17—H17···Br^{i}	0.84	2.48	3.319 (6)	174

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

Acknowledgements

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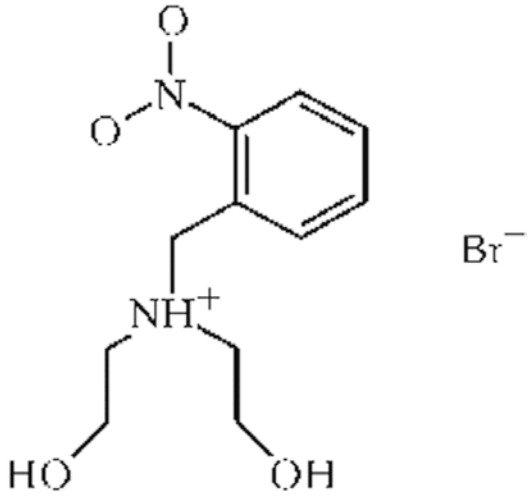
Scheme 1



supplementary materials

N-(2-nitrobenzyl)diethanolammonium bromide

Crystal data

$C_{11}H_{17}N_2O_4^+ \cdot Br^-$	$F_{000} = 656$
$M_r = 321.18$	$D_x = 1.597$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.644$ (13) Å	Cell parameters from 39 reflections
$b = 23.030$ (4) Å	$\theta = 12.5$–15.5°
$c = 8.856$ (3) Å	$\mu = 3.09$ mm^{-1}
$\beta = 99.64$ (2)°	$T = 150$ (2) K
$V = 1336$ (3) Å^3	Needle, yellow
$Z = 4$	0.48 × 0.12 × 0.08 mm

Data collection

Stoe Stadi-4 four-circle with Oxford Cryosystems open-flow cryostat (Cosier & Glazer, 1986) diffractometer	$R_{int} = 0.044$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0^\circ$
Monochromator: graphite	$\theta_{min} = 2.9^\circ$
$T = 150(2)$ K	$h = -7 \rightarrow 7$
ω/θ scans	$k = 0 \rightarrow 27$
Absorption correction: ψ-scans North, Phillips & Mathews, 1968	$l = -1 \rightarrow 10$
$T_{min} = 0.655$, $T_{max} = 0.781$	3 standard reflections
2520 measured reflections	every 60 min
2357 independent reflections	intensity decay: random variation +−6.9%
1786 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: see text
$R[F^2 > 2\sigma(F^2)] = 0.048$	See text
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 5.9611P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2357 reflections	$\Delta\rho_{max} = 1.41$ e Å^{-3}
165 parameters	$\Delta\rho_{min} = -0.58$ e Å^{-3}
8 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br	0.50702 (8)	0.33941 (2)	0.47290 (6)	0.02496 (18)
O1	−0.1945 (6)	0.39233 (18)	−0.1047 (5)	0.0330 (10)
O2	−0.3594 (7)	0.4732 (2)	−0.1207 (6)	0.0515 (13)
N1	−0.2134 (7)	0.4434 (2)	−0.1436 (5)	0.0288 (11)
C1	−0.0581 (8)	0.4706 (2)	−0.2187 (5)	0.0266 (12)
C2	0.0698 (7)	0.4369 (2)	−0.2944 (5)	0.0247 (12)
C3	0.2152 (8)	0.4670 (2)	−0.3590 (6)	0.0297 (13)
H3A	0.3049	0.4458	−0.4115	0.036*
C4	0.2339 (9)	0.5267 (2)	−0.3497 (6)	0.0369 (15)
H4A	0.3351	0.5461	−0.3950	0.044*
C5	0.1032 (9)	0.5582 (3)	−0.2734 (6)	0.0375 (15)
H5A	0.1153	0.5993	−0.2662	0.045*
C6	−0.0428 (9)	0.5303 (2)	−0.2088 (6)	0.0345 (14)
H6A	−0.1330	0.5518	−0.1574	0.041*
C21	0.0523 (8)	0.3719 (2)	−0.3221 (6)	0.0211 (11)
H21A	−0.0938	0.3618	−0.3523	0.025*
H21B	0.1222	0.3620	−0.4090	0.025*
O11	0.2702 (6)	0.35141 (17)	0.1215 (4)	0.0296 (9)
H11	0.3131	0.3477	0.2157	0.044*
C12	0.4190 (8)	0.3301 (2)	0.0380 (6)	0.0247 (12)
H12A	0.4228	0.2872	0.0415	0.030*
H12B	0.5558	0.3451	0.0826	0.030*
C13	0.3601 (8)	0.3507 (2)	−0.1240 (6)	0.0256 (12)
H13A	0.4510	0.3326	−0.1886	0.031*
H13B	0.3777	0.3933	−0.1280	0.031*
N14	0.1411 (6)	0.33521 (18)	−0.1862 (5)	0.0194 (9)
H14A	0.0643	0.3416	−0.1091	0.023*
C15	0.1209 (9)	0.2719 (2)	−0.2307 (6)	0.0252 (12)
H15A	0.1538	0.2670	−0.3349	0.030*
H15B	0.2205	0.2489	−0.1591	0.030*
C16	−0.0910 (9)	0.2493 (2)	−0.2277 (6)	0.0275 (13)
H16A	−0.1930	0.2729	−0.2955	0.033*
H16B	−0.1027	0.2085	−0.2635	0.033*

O17	−0.1251 (7)	0.2528 (2)	−0.0754 (4)	0.0418 (11)
H17	−0.2247	0.2317	−0.0644	0.063*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0255 (3)	0.0247 (3)	0.0247 (3)	−0.0002 (3)	0.0043 (2)	−0.0004 (2)
O1	0.027 (2)	0.032 (2)	0.042 (2)	0.0000 (18)	0.0118 (18)	0.0034 (19)
O2	0.037 (3)	0.049 (3)	0.074 (3)	0.011 (2)	0.022 (2)	−0.002 (3)
N1	0.022 (3)	0.034 (3)	0.032 (3)	0.001 (2)	0.008 (2)	−0.008 (2)
C1	0.025 (3)	0.025 (3)	0.028 (3)	0.000 (2)	−0.001 (2)	0.001 (2)
C2	0.025 (3)	0.022 (3)	0.024 (3)	0.002 (2)	−0.003 (2)	0.000 (2)
C3	0.030 (3)	0.024 (3)	0.034 (3)	−0.002 (2)	0.000 (2)	0.007 (2)
C4	0.036 (4)	0.031 (3)	0.041 (4)	−0.011 (3)	−0.002 (3)	0.010 (3)
C5	0.048 (4)	0.020 (3)	0.041 (4)	−0.008 (3)	−0.001 (3)	0.002 (3)
C6	0.036 (4)	0.022 (3)	0.041 (4)	0.009 (3)	−0.005 (3)	−0.007 (3)
C21	0.023 (3)	0.023 (3)	0.018 (3)	0.004 (2)	0.003 (2)	−0.002 (2)
O11	0.032 (2)	0.036 (2)	0.0205 (19)	0.0101 (18)	0.0044 (16)	−0.0005 (17)
C12	0.021 (3)	0.026 (3)	0.026 (3)	0.002 (2)	−0.002 (2)	0.003 (2)
C13	0.018 (3)	0.028 (3)	0.028 (3)	−0.001 (2)	−0.003 (2)	0.002 (2)
N14	0.020 (2)	0.020 (2)	0.020 (2)	−0.0015 (19)	0.0060 (17)	−0.0022 (18)
C15	0.032 (3)	0.018 (3)	0.026 (3)	0.001 (2)	0.003 (2)	0.000 (2)
C16	0.031 (3)	0.024 (3)	0.028 (3)	−0.002 (2)	0.004 (2)	0.004 (2)
O17	0.048 (3)	0.052 (3)	0.026 (2)	−0.024 (2)	0.010 (2)	−0.001 (2)

Geometric parameters (Å, °)

O1—N1	1.226 (6)	C5—C6	1.367 (8)
O2—N1	1.233 (6)	C21—N14	1.507 (6)
N1—C1	1.459 (7)	O11—C12	1.417 (7)
C1—C6	1.382 (7)	C12—C13	1.500 (7)
C1—C2	1.401 (7)	C13—N14	1.510 (7)
C2—C3	1.388 (8)	N14—C15	1.511 (7)
C2—C21	1.518 (7)	C15—C16	1.506 (8)
C3—C4	1.383 (7)	C16—O17	1.407 (7)
C4—C5	1.390 (8)		
O1—N1—O2	122.3 (5)	C6—C5—C4	120.2 (5)
O1—N1—C1	119.5 (4)	C5—C6—C1	119.5 (6)
O2—N1—C1	118.2 (5)	N14—C21—C2	114.5 (4)
C6—C1—C2	122.5 (6)	O11—C12—C13	107.3 (4)
C6—C1—N1	116.7 (5)	C12—C13—N14	110.8 (4)
C2—C1—N1	120.8 (5)	C21—N14—C13	112.5 (4)
C3—C2—C1	116.1 (5)	C21—N14—C15	109.1 (4)
C3—C2—C21	117.7 (5)	C13—N14—C15	111.2 (4)
C1—C2—C21	125.9 (5)	C16—C15—N14	111.8 (4)
C4—C3—C2	122.3 (6)	O17—C16—C15	107.3 (4)
C3—C4—C5	119.4 (6)		
O1—N1—C1—C6	−156.5 (4)	C2—C1—C6—C5	−0.6 (5)

O2—N1—C1—C6	23.0 (6)	N1—C1—C6—C5	178.4 (4)
O1—N1—C1—C2	22.5 (6)	C3—C2—C21—N14	105.1 (4)
O2—N1—C1—C2	−158.1 (4)	C1—C2—C21—N14	−80.1 (5)
C6—C1—C2—C3	0.3 (2)	O11—C12—C13—N14	−52.2 (6)
N1—C1—C2—C3	−178.6 (4)	C2—C21—N14—C13	−54.0 (6)
C6—C1—C2—C21	−174.6 (4)	C2—C21—N14—C15	−177.9 (4)
N1—C1—C2—C21	6.5 (5)	C12—C13—N14—C21	159.6 (4)
C1—C2—C3—C4	0.1 (2)	C12—C13—N14—C15	−77.7 (5)
C21—C2—C3—C4	175.4 (4)	C21—N14—C15—C16	−79.8 (5)
C2—C3—C4—C5	−0.1 (5)	C13—N14—C15—C16	155.5 (4)
C3—C4—C5—C6	−0.2 (6)	N14—C15—C16—O17	−63.5 (6)
C4—C5—C6—C1	0.5 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14A···O1	0.93	2.08	2.786 (7)	131
N14—H14A···O11	0.93	2.27	2.742 (5)	111
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O11—H11···Br	0.84	2.43	3.255 (4)	168
O17—H17···Br^{i}	0.84	2.48	3.319 (6)	174

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