O3—C6—C7	115.3 (4)	O9*b*—C15—C16	124.0 (6)
C5—C6—C7	120.0 (4)	O6—C17—O10	124.3 (4)
O2—C7—C6	114.7 (4)	O6—C17—C18	110.8 (5)
O2—C7—C8	125.7 (4)	O10—C17—C18	124.9 (5)
C6—C7—C8	119.6 (4)	O7—C19—O11*a*	121.7 (9)
C7—C8—C9	120.7 (4)	O7—C19—O11*b*	123.5 (8)
C1—C9—C8	121.6 (4)	O7—C19—C20	112.6 (5)
C1—C9—C10	118.0 (4)	O11*a*—C19—C20	124.8 (9)
C8—C9—C10	120.4 (4)	O11*b*—C19—C20	119.5 (8)
N—C1—C1′—O4	52.2 (4)	O1—C1—C1′—C2′	−132.8 (4)
O1—C1—C1′—O4	−17.4 (5)	N—C1—C1′—C2′	−63.1 (5)
C9—C1—C1′—O4	−159.2 (3)	C9—C1—C1′—H1′	−35.3 (6)
O1—C1—C1′—H1′	106.5 (5)	C9—C1—C1′—C2′	85.4 (5)
N—C1—C1′—H1′	176.1 (4)		

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71301 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0308]

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Structure of Tetraethylammonium Bromide–Succinimide (1/2)

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Abstract

In tetraethylammonium bromide–2,5-pyrrolidinedione (1/2) the bromide ion forms hydrogen bonds to the imide H atoms in the two succinimide molecules: Br···N = 3.338 (11) and 3.380 (10) Å with N···Br···N = 102.8 (3)° and N—H···Br = 158 and 178°. The succinimide molecules are inclined at 63.72 (3)° to each other.

Comment

The title compound has been investigated as part of a study of N—Br—N bonding properties (Elding, Albertsson, Svensson & Eberson, 1990; Elding, Larsson, Svensson, Albertsson & Eberson, 1992). The solid complex was prepared by mixing *N*-bromosuccinimide (0.05 mole in 70 ml acetonitrile) with tetraethylammonium thiocyanate (0.05 mole in 15 ml acetonitrile) and then adding dry diethyl ether to the solution. The mixture was kept at 273 K for several days before the crystals were filtered off. This yielded a mixture of very small orange crystals, which could not be studied by single-crystal diffraction, and larger needle-shaped colourless crystals, of which one was chosen for this investigation.

The bromide ion is close to the NH groups in the two succinimide molecules (Fig. 1). The Br···N distances are 3.338 (11) and 3.380 (10) Å for N1 and N2, respectively. The H-atom positions were calculated with N—H = 0.95 Å. The resulting Br···H distances are 2.44 and 2.43 Å, more than 0.2 Å shorter than the sum of the van der Waals radii for Br and H (3.15 Å) (*Chemistry Data Book*, 1982). Thus, the bromide ion is hydrogen bonded to the succinimide molecules (Hamilton & Ibers, 1968). This is also indicated by the sum of the Br^- ionic radius (1.95 Å), the covalent radius of N (0.74 Å) and the covalent diameter of H (0.74 Å), which is 3.43 Å (*Chemistry Data Book*, 1982), *i.e.* close to the Br···N distances found. The shorter of the Br···N distances results in an angled hydrogen bond, N—H···Br, and the longer in an almost linear one (Table 2). The angle N1···Br···N2 = 102.8 (3)° is in good agreement

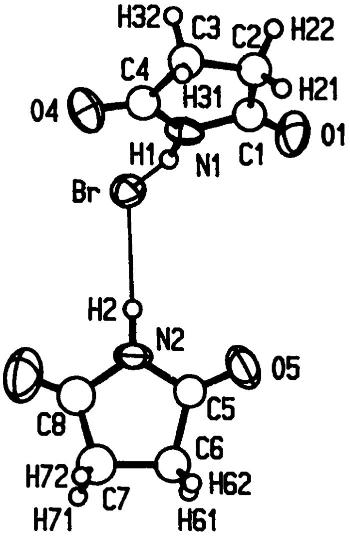

Fig. 1. Drawing of the bromide–succinimide (1/2) moiety in the structure. The H atoms are drawn with a small arbitrary radius. The displacement ellipsoids are scaled to include 50% probability.

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with tetrahedral coordination around Br with the two lone-electron (sp^3) pairs taking rather more space than the bonding ones. Closest to the bromide ion, apart from the two succinimide molecules, is C37 ($\frac{1}{2} - x$, $1 - y$, $\frac{1}{2} + z$) at 3.76 (1) Å.

The distances and angles around the Br atom are in agreement with other compounds containing a bromide ion hydrogen bonded to an N atom (Dahan & Lefébvre-Soubeyran, 1976; Oskarsson, 1973; Stålhandske, 1972).

The distance ranges are 1.49 (2)–1.55 (2) Å for C—C, 1.18 (2)–1.21 (2) Å for C—O and 1.37 (2)–1.40 (2) Å for C—N in the succinimide molecules, and 1.50 (2)–1.52 (2) Å for C—N in the tetraethylammonium ion. The angles are as expected. The least-squares planes through the five atoms in the succinimide rings are inclined 63.72 (3)° with respect to each other. The O atoms are located within these planes with the exception of O5, which is 0.147 (11) Å out of the plane. The Br atom is 0.9371 (15) and 0.1583 (15) Å out of the planes containing N1 and N2, respectively.

The bromide–succinimide (1/2) units and the tetraethylammonium ions are arranged in alternating layers parallel to the *ac* plane (Fig. 2). The crystal is stabilized by van der Waals interactions. The shortest distance is C32···O1($\frac{1}{2} - x$, $1 - y$, $-\frac{1}{2} + z$) = 3.29 (2) Å.

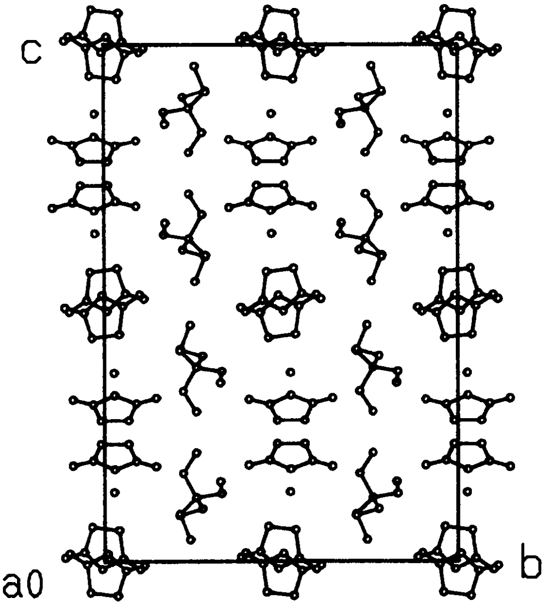

Fig. 2. Unit-cell contents projected on (100). All atoms are drawn with the same small arbitrary radius.

Experimental

Crystal data

$C_8H_{20}N^+.Br^-.2C_4H_5NO_2$
$M_r = 408.34$
Orthorhombic
Pbca
$a = 7.5137$ (9) Å
$b = 19.178$ (2) Å
$c = 27.731$ (6) Å
$V = 3996$ (1) Å^3
$Z = 8$
$D_x = 1.36$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 4.7$–$17.2°$
$\mu = 2.057$ mm^{-1}
$T = 298$ K
Needle
0.16 × 0.11 × 0.11 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration from crystal shape
$T_{min} = 0.7663$, $T_{max} = 0.8150$
4194 measured reflections
3486 independent reflections
659 observed reflections $[I > 3\sigma(I)]$

$R_{int} = 0.0074$ based on the standard reflections
$\theta_{max} = 25°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 33$
3 standard reflections
frequency: 60 min
intensity variation: linear decay by 3% (linear correction)

Refinement

Refinement on F
Final $R = 0.036$
$wR = 0.041$
$S = 1.08$
659 reflections
137 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + (0.025F_o)^2]$
$(\Delta/\sigma)_{max} = 0.046$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	0.1675 (2)	0.47334 (7)	0.63423 (5)	0.0465 (4)
N1	0.5808 (14)	0.4714 (10)	0.6784 (3)	0.044 (4)
C1	0.657 (2)	0.5317 (11)	0.6956 (4)	0.041 (4)
C2	0.811 (2)	0.5100 (7)	0.7277 (5)	0.047 (5)
C3	0.814 (2)	0.4313 (7)	0.7264 (5)	0.049 (4)
C4	0.661 (3)	0.4096 (9)	0.6939 (5)	0.045 (4)
O1	0.6047 (15)	0.5900 (6)	0.6861 (4)	0.067 (6)
O4	0.6174 (14)	0.3526 (5)	0.6838 (4)	0.064 (5)
N2	0.235 (2)	0.4928 (6)	0.5145 (4)	0.047 (5)
C5	0.309 (2)	0.5511 (8)	0.4941 (5)	0.044 (4)
C6	0.315 (2)	0.5404 (7)	0.4400 (4)	0.049 (4)
C7	0.251 (2)	0.4643 (8)	0.4334 (5)	0.061 (5)
C8	0.188 (3)	0.4402 (9)	0.4820 (6)	0.058 (5)
O5	0.365 (2)	0.5991 (5)	0.5161 (3)	0.067 (5)
O8	0.123 (2)	0.3860 (6)	0.4923 (4)	0.092 (6)
N3	0.2103 (14)	0.2611 (5)	0.1235 (3)	0.036 (5)
C31	0.191 (2)	0.2225 (7)	0.1704 (5)	0.048 (4)
C32	0.083 (2)	0.2584 (9)	0.2093 (6)	0.082 (6)
C33	0.034 (2)	0.2793 (8)	0.1018 (5)	0.049 (4)
C34	−0.082 (2)	0.2163 (9)	0.0901 (6)	0.064 (5)
C35	0.317 (2)	0.2136 (7)	0.0917 (5)	0.040 (4)
C36	0.366 (2)	0.2426 (7)	0.0428 (5)	0.061 (5)
C37	0.301 (2)	0.3308 (6)	0.1307 (5)	0.048 (4)
C38	0.482 (2)	0.3279 (9)	0.1529 (5)	0.083 (6)

Table 2. *Selected bond distances* (Å) *and angles* (°) *for the bromide ion and the succinimide molecules*

Br···N1	3.338 (11)	C5—O5	1.18 (2)
Br···N2	3.380 (10)	C5—C6	1.51 (2)
Br···H1	2.44	C6—C7	1.55 (2)
Br···H2	2.43	C7—C8	1.50 (2)
N1—C1	1.38 (3)	C8—O8	1.18 (2)
N1—C4	1.40 (2)	N3—C31	1.50 (2)
C1—O1	1.21 (2)	N3—C33	1.50 (2)
C1—C2	1.52 (2)	N3—C35	1.50 (2)
C2—C3	1.51 (2)	N3—C37	1.52 (2)
C3—C4	1.52 (2)	C31—C32	1.52 (2)
C4—O4	1.18 (2)	C33—C34	1.52 (2)
N2—C5	1.37 (2)	C35—C36	1.51 (2)
N2—C8	1.40 (2)	C37—C38	1.49 (2)
N1···Br···N2	102.8 (3)	N1—H1···Br	158
H1···Br···H2	96	N2—H2···Br	178

The structure was solved by direct methods (*MULTAN*87; Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987). Some of the H atoms were found in $\Delta\rho$ maps, the others were placed in calculated positions (C—H and N—H distances 0.95 Å). All were assigned an isotropic displacement parameter of 4.0 Å^2, which is an approximate average of the displacement parameters of the corresponding C and N atoms. Br, N and O atoms were refined with anisotropic displacement parameters. The H atoms were not refined. The system of computer programs used for this study is described by Lundgren (1982).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71314 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1042]

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Refinement of the Molecular Structure of Benzhydrylamine Hydrochloride

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Abstract

The molecular conformation of the title compound, α-phenylbenzylammonium chloride, is helical C_2 with ring twist angles of 57.8 (3) and 68.3 (2)°. Pairs of doubly hydrogen-bonded molecules, connected by hydrogen bonds, form infinite chains along **b**.

Comment

Benzhydrylamine inhibits smooth-muscle spasms (Bruno Blanch, 1990); some of its derivatives obtained by arylic and/or aminic mono- and di-substitution present greater biological activity than the parent compound. Comparison of the molecular structure of the title compound and its derivatives would help towards understanding the structure-activity relationship. Consequently, a study of the structure of benzhydrylamine hydrochloride (BH), a salt used in activity determination, was performed (Rivero, Punte, Rigotti, Goeta, Quinteros Rivero, Vega & Bruno Blanch, 1991) but all attempts to obtain an *R* factor smaller than 0.07 failed. A thorough analysis of the information was conducted; an instability of the equipment was found that mimics crystal decay. Therefore, to provide a more accurate starting model to analyze the influence of substituents on molecular geometry, a freshly grown single crystal was used to perform the present study.

Intramolecular bond distances and angles in BH do not deviate notably from the expected values. The aromatic rings are planar within experimental error. The bridge angle, Φ, is 113.7 (3)°, in agreement with that in diphenylmethane (DPM) [112.5 (6)° (Barnes, Paton, Damewood & Mislow, 1981)]. The dihedral angles subtended between the least-squares planes of the two rings and the central plane defined by C1*A*—C7—C1*B*, Φ_1 and Φ_2, are 57.8 (3) and 68.3 (2)°, respectively. The angle between the rings, Φ_{12}, is 97.7 (2)°. Barnes, Paton, Damewood & Mislow (1981) found, from data retrieved from the Cambridge Structural Database, that

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