1366 reflections
216 parameters
H atoms riding on attached C or N
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} = 0.0181$

Extinction coefficient: 32.7 (22)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	−0.11100 (9)	0.7296 (2)	0.46747 (5)	0.0349 (6)
Cl(2)	−0.52314 (9)	0.5846 (2)	0.27248 (5)	0.0374 (6)
N(11)	0.0557 (4)	0.8273 (6)	0.3595 (2)	0.0455 (2)
N(12)	0.2237 (4)	0.7725 (6)	0.3083 (2)	0.0429 (2)
N(21)	−0.2828 (4)	0.4288 (6)	0.3745 (2)	0.0427 (2)
N(22)	−0.1522 (3)	0.2014 (6)	0.4318 (2)	0.0408 (2)
C(11)	0.1618 (4)	0.7311 (7)	0.3551 (2)	0.0366 (3)
C(12)	0.2060 (4)	0.5734 (7)	0.4039 (2)	0.0395 (3)
C(13)	0.3382 (4)	0.6236 (9)	0.4410 (2)	0.0557 (3)
C(14)	0.2051 (5)	0.3697 (8)	0.3713 (2)	0.0576 (3)
C(21)	−0.2525 (4)	0.2440 (7)	0.3884 (2)	0.0377 (3)
C(22)	−0.3310 (4)	0.0758 (7)	0.3553 (2)	0.0415 (3)
C(23)	−0.3302 (5)	0.0793 (8)	0.2834 (2)	0.0551 (3)
C(24)	−0.4628 (5)	0.0770 (9)	0.3722 (3)	0.0587 (4)

Table 2. *Geometric parameters* (Å, °)

N(11)—C(11)	1.320 (5)	Cl(1)···N(11)	3.20 (2)
N(12)—C(11)	1.315 (5)	Cl(1)···N(21)	3.15 (2)
N(21)—C(21)	1.299 (6)	Cl(1)···N(22)	3.62 (2)
N(22)—C(21)	1.301 (5)	Cl(1)···N(22$^{\rm i}$)	3.26 (2)
C(11)—C(12)	1.486 (6)	Cl(1)···N(22$^{\rm ii}$)	3.22 (2)
C(12)—C(13)	1.517 (6)	Cl(2)···N(11$^{\rm iii}$)	3.23 (2)
C(12)—C(14)	1.526 (6)	Cl(2)···N(12$^{\rm iv}$)	3.19 (2)
C(21)—C(22)	1.495 (6)	Cl(2)···N(12$^{\rm iii}$)	3.24 (2)
C(22)—C(23)	1.514 (6)	Cl(2)···N(21)	3.20 (2)
C(22)—C(24)	1.513 (6)		
N(11)—C(11)—N(12)	120.4 (4)	N(21)—C(21)—N(22)	120.3 (4)
N(11)—C(11)—C(12)	117.7 (4)	N(21)—C(21)—C(22)	121.2 (4)
N(12)—C(11)—C(12)	121.9 (4)	N(22)—C(21)—C(22)	118.5 (4)
C(11)—C(12)—C(13)	110.0 (4)	C(21)—C(22)—C(23)	110.3 (4)
C(11)—C(12)—C(14)	110.4 (4)	C(21)—C(22)—C(24)	110.6 (4)
C(13)—C(12)—C(14)	110.9 (4)	C(23)—C(22)—C(24)	114.4 (4)

Symmetry codes: (i) $x, 1+y, z$; (ii) $-x, 1-y, 1-z$; (iii) $-\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $x-1, y, z$.

Data collection: Rigaku *AFC*-7R software. Cell refinement: Rigaku *AFC*-7R software. Data reduction: Rigaku *AFC*-7R software. Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Molecular graphics: *CAMERON* (Pearce & Watkin, 1993). Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diisopropylammonium Bromide

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Abstract

In the title compound, $C_6H_{16}N^+.Br^-$, each diisopropylammonium cation interacts with the bromide anion *via* two hydrogen bonds leading to an infinite one-dimensional chain of molecules in the crystal. The cation has an approximate twofold axis of symmetry.

Comment

Diisopropylammonium bromide (I) was obtained in the course of studying the reaction of $BrRe(CO)_5$ with lithium dialkylamides with the intention of generating lithium carbamoylmetallates as precursors for amino-

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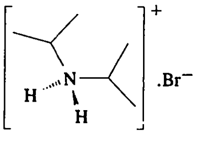

(I)

carbene complexes (Dötz *et al.*, 1983). The cation in (I) has an approximate C_2 symmetry (Fig. 1). Two hydrogen bonds are found between the diisopropylammonium cation and the bromide anion, forming a one-dimensional chain in the direction of the crystallographic *b* axis (Fig. 2). The hydrogen-bond geometry is given in Table 3. A similar hydrogen-bond pattern is found in the isostructural, though not isomorphous, diisopropylammonium chloride (Prince, Miller, Fronczek & Gandour, 1990). Diisopropylammonium cations and anions packed in the crystal through networks of hydrogen-bond interactions are also found in bis(diisopropylammonium)tetrabromocuprate(II) (Fernandez, Moran, Gutierrez-Rios, Foces-Foces & Cano, 1987) and in the diisopropylammonium salts of a carbamoylplatinate (Dell'Amico, Calderazzo & Pelizzi, 1979) and a carbonylferrate (Chen, Cheng & Hseu, 1987). The N—C and C—C distances of (I) compare favourably with distances found in diisopropylammonium diisopropyldithiocarbamate (Wahlberg, 1978) and the diisopropylammonium salt of a titanium(III) *N*,*N*-dialkylcarbamato complex (Dell'Amico, Calderazzo, Giurlani & Pelizzi, 1987).

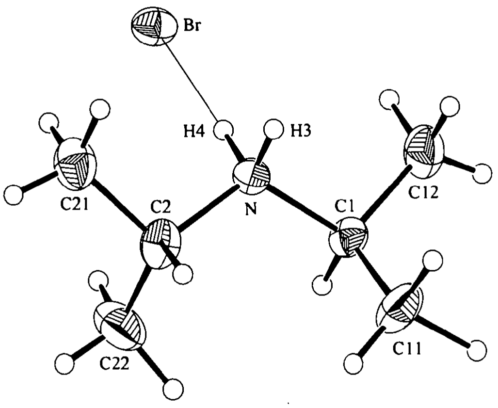

Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1994) drawing of $[C_6H_{16}N]Br$ with displacement ellipsoids drawn at the 50% probability level.

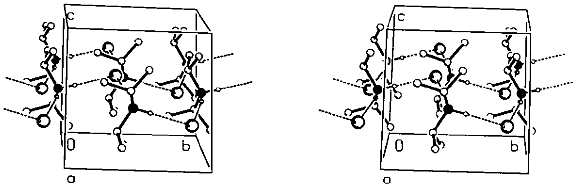

Fig. 2. Stereoview (*PLUTON92*; Spek, 1992) of the unit cell of $C_6H_6N^+.Br^-$.

Experimental

The title compound has been isolated as a by-product after the reaction of $BrRe(CO)_5$ with lithium diisopropylamide in thf at room temperature. Colourless needles of (I) could be obtained upon cooling a saturated thf solution to 233 K.

Crystal data

$C_6H_{16}N^+.Br^-$
$M_r = 182.11$
Monoclinic
$P2_1$
$a = 7.7989 (4)$ Å
$b = 8.0660 (11)$ Å
$c = 7.8631 (10)$ Å
$\beta = 116.336 (13)°$
$V = 443.30 (9)$ Å^3
$Z = 2$
$D_x = 1.364$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 27 reflections
$\theta = 13–17.4°$
$\mu = 4.556$ mm^{-1}
$T = 180 (2)$ K
Needle
0.57 × 0.33 × 0.08 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω–2θ scans
Absorption correction: empirical, ψ-scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.8002$, $T_{max} = 0.9984$
2760 measured reflections
1374 independent reflections
1188 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0279$
$\theta_{max} = 29.99°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0220$
$wR(F^2) = 0.0468$
$S = 1.020$
1332 reflections
139 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0207P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.424$ e Å^{-3}
$\Delta\rho_{min} = -0.306$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.003 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.33 (3)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	0.14997 (3)	0.8454	0.36481 (4)	0.02746 (9)
N	0.1682 (3)	0.4431 (5)	0.4772 (4)	0.0205 (5)
C1	0.3636 (5)	0.4135 (7)	0.6394 (5)	0.0240 (7)
C11	0.3851 (7)	0.2351 (7)	0.7030 (8)	0.0378 (11)
C12	0.3855 (6)	0.5329 (7)	0.7970 (6)	0.0361 (8)
C2	0.1122 (4)	0.3426 (13)	0.2975 (4)	0.0277 (6)
C21	−0.0873 (5)	0.3966 (6)	0.1579 (5)	0.0347 (8)
C22	0.2571 (6)	0.3653 (15)	0.2211 (6)	0.045 (2)

Table 2. *Geometric parameters (Å, °)*

N—C1	1.510 (5)	C1—C12	1.518 (6)
N—C2	1.516 (7)	C2—C22	1.508 (5)
C1—C11	1.508 (7)	C2—C21	1.516 (5)
C1—N—C2	117.9 (3)	C22—C2—N	110.4 (5)
C11—C1—N	110.7 (3)	C22—C2—C21	112.7 (4)
C11—C1—C12	112.3 (4)	N—C2—C21	107.5 (5)
N—C1—C12	107.2 (3)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H(3)···Br^{i}	0.82 (4)	2.51 (4)	3.325 (3)	173 (4)
N—H(4)···Br	0.81 (4)	2.55 (4)	3.350 (4)	171 (4)

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

The *SHELX* input files were prepared using the program *UTILITY* (Pickardt, 1994). All non-H-atoms were refined anisotropically and all H atoms were refined isotropically. The crystal was a racemic twin consisting of two individuals in the ratio 2:1, as established with a *TWIN*/*BASF* refinement (*SHELXL*93; Sheldrick, 1993).

Data collection: *Stadi*-4 *Software* (Stoe & Cie, 1995*a*). Cell refinement: *Stadi*-4 *Software*. Data reduction: *X-RED*, (Stoe & Cie, 1995*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *PLUTON*92 (Spek, 1992), *PLATON* (Spek, 1990), *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Substituted *cis*,*trans*,*cis*,*cis*-[4.5.5.5]Fenestrene

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(Received 22 December 1995; accepted 18 March 1996)

Abstract

The title compound, *rel*-(1*S*,4*R*,7*R*,9*S*)-tetracyclo-[5.4.1.0^{4,12}.0^{9,12}]dodec-10-enyl-1-acetic acid, $C_{14}H_{18}O_2$, has been prepared and its structure elucidated. The geometry of the central C(C)$_4$ substructure shows a considerable distortion from an ideal tetrahedral arrangement towards planarity, with two opposite bond angles of 119.2 (2) and 134.9 (2)°. The other bridgehead bond angle of the *trans*-bicyclo[3.3.0]octane subunit is also large, with a value of 128.0 (2)°.

Comment

Tetracyclic compounds, where four small rings share a common C atom, are of considerable interest for their propensity towards planarizing distortions at the central C atom, leading to two opposite bond angles markedly larger than 109.47° (Luef & Keese, 1992, 1993). In our systematic search for fenestranes with strong planarizing distortions, it was found that the simultaneous incorporation of *trans*-bicyclo[3.3.0]octane subunits and bridgehead substituents led to enlarged opposite bond angles at the central C atom. In order to verify this computational result and to compare the structure of (3*b*) with that of the structurally similar *cis*,*trans*,*cis*,*cis*-[4.5.5.5]fenestranediol, (5) (Hirschi, Luef, Gerber & Keese, 1992), the known compound (2*a*) was prepared from (1) by a new route, transformed *via* (2*b*) into (3*a*) by a previously published method (Grieco, Brandes, McCann & Clark, 1989) and oxidized with Jones reagent (acetone, 298 K) to compound (3*b*).