1-[Bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl]-3-(but-3-enyl)imidazolium bromide

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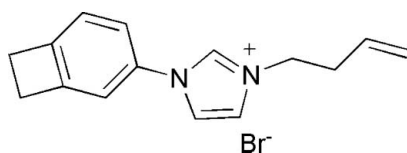Correspondence e-mail: orgxie@scu.edu.cn

In the title compound, $C_{15}H_{17}N_2^+ \cdot Br^-$, the cyclobutene and benzene rings are coplanar. The dihedral angle between the benzene and imidazolium rings is 21.2 (3)°. In the crystal structure, the $C_{15}H_{17}N_2^+$ and Br^- ions are linked into a zigzag chain along the b axis by $C-H \cdots Br$ hydrogen bonds, and weak $C-H \cdots \pi$ interactions involving the benzene ring of a screw-related cation.

Related literature

For related literature, see: Farona (1996); Tan & Arnold (1988); Zhang et al. (2005).

Experimental

Crystal data

$C_{15}H_{17}N_2^+ \cdot Br^-$
$M_r = 305.22$
Monoclinic, $P2_1/c$
$a = 9.342$ (3) Å
$b = 11.775$ (3) Å
$c = 13.695$ (7) Å
$\beta = 107.76$ (3)°

$V = 1434.7$ (10) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 2.85$ mm^{-1}
$T = 291$ (2) K
$0.30 \times 0.25 \times 0.25$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
2811 measured reflections
2665 independent reflections

1479 reflections with $I > 2\sigma(I)$
$R_{int} = 0.005$
3 standard reflections
  every 300 reflections
  intensity decay: 5.6%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.144$
$S = 0.94$
2665 reflections

166 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

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

$Cg1$ is the C1–C4/C7/C9 ring centroid.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C9—H9$\cdots$Br	0.93	2.66	3.561 (5)	165
C11—H11$\cdots$Br^i	0.93	2.87	3.697 (6)	149
C13—H13A$\cdots$Cg1^i	0.97	2.91	3.735 (7)	143

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

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe et al., 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2512).

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supporting information

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S1. Comment

Benzocyclobutene (BCB) based polymeric materials have attracted considerable attention and research interest in the area of electronic applications because of their excellent properties such as low dielectric constant, low dissipation factor, low moisture picking-up, film planarization and high thermal-stability (Farona, 1996; Tan & Arnold, 1988). A number of BCB derivatives, such as BCB-alkyne imide, bis-BCB imide, organosiloxane bridged bis-BCB have been synthesized (Zhang *et al.*, 2005). We report here the crystal structure of the title imidazolium BCB compound, (I), which was synthesized by alkylation of *N*-imidazolybenzocyclobutene and 4-bromo-1-butene.
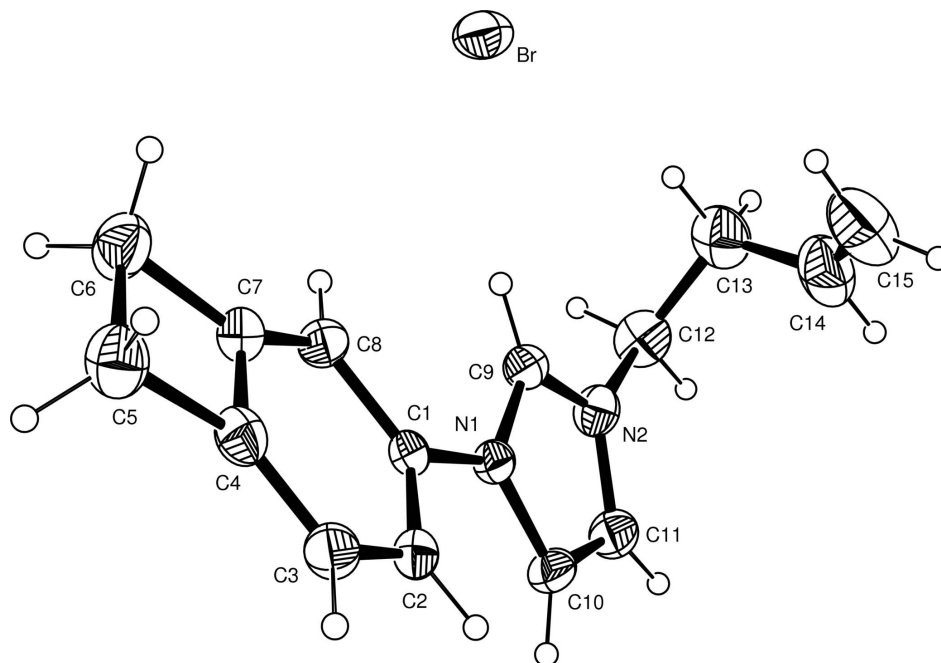
The cyclobutene and benzene rings coplanar, with a dihedral angle of 0.7 (4)°. The dihedral angle between the benzene and imidazolium rings is 21.2 (3)°. In the crystal structure of (I), the cations and the bromide ions are linked *via* C—H···Br hydrogen bonds, and weak C—H···π interactions involving the C13—H13A group and the benzene of a screw-related molecule (Table 1), forming a zigzag chain along the *b* axis.

S2. Experimental

4-(*N*-imidazolyl)benzocyclobutene (5 mmol, 850 mg) and 4-bromo-1-butene (6 mmol, 810 mg,) were placed in a two-necked round-bottomed flask under a nitrogen atmosphere and the mixture was heated at 353 K for 5 h. A light-yellow solid was obtained after the surplus 4-bromo-1-butene was removed under vacuum. Colourless crystals of compound (I) were obtained by recrystallization of the solid from methanol-ethyl ether (1:4 *v*/*v*) solution (yield: 1.278 g). [1] H NMR (400 MHz, CDCl$_3$): δ 10.80 (s, 1H), 7.74 (s, 1H), 7.61 (s, 1H), 7.49 (2 d, J = 7.6 Hz, 1H), 7.42 (s, 1H), 7.21 (d, J = 7.6 Hz, 1H), 5.86–5.95 (m, 1H), 5.11 (d, J = 14.4 Hz, 2H), 4.75 (t, 2H), 3.23 (s, 4H,), 2.76 (q, 2H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 148.22, 147.96, 135.57, 133.50, 132.65, 124.46, 123.48, 121.12, 120.97, 119.49, 116.80, 49.30, 34.60, 29.54, 29.46 p.p.m..

S3. Refinement

H atoms were positioned geometrically and refined in the riding-model approximation with C—H = 0.93 or 0.97 Å. A common free variable for U_{iso} was refined for the aromatic H atoms, and similarly for the methylene and methyl H atoms.

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

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Crystal data

$C_{15}H_{17}N_2^+ \cdot Br^-$	$F(000) = 624$
$M_r = 305.22$	$D_x = 1.413$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 24 reflections
$a = 9.342$ (3) Å	$\theta = 4.8$–9.6°
$b = 11.775$ (3) Å	$\mu = 2.85$ mm^{-1}
$c = 13.695$ (7) Å	$T = 291$ K
$\beta = 107.76$ (3)°	Block, colourless
$V = 1434.7$ (10) Å^3	$0.30 \times 0.25 \times 0.25$ mm
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.005$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.5°$, $\theta_{min} = 2.3°$
Graphite monochromator	$h = -11 \rightarrow 10$
$\omega/2\theta$ scans	$k = 0 \rightarrow 14$
2811 measured reflections	$l = -6 \rightarrow 16$
2665 independent reflections	3 standard reflections every 300 reflections
1479 reflections with $I > 2\sigma(I)$	intensity decay: 5.6%

Refinement

Refinement on F^2	$S = 0.94$
Least-squares matrix: full	2665 reflections
$R[F^2 > 2\sigma(F^2)] = 0.052$	166 parameters
$wR(F^2) = 0.144$	0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Extinction correction: *SHELXL97*,
Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Extinction coefficient: 0.029 (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}
Br	0.66118 (7)	0.64239 (5)	0.09316 (5)	0.0607 (3)
N1	0.2215 (4)	0.5236 (3)	0.1168 (3)	0.0378 (9)
N2	0.3774 (5)	0.3946 (4)	0.1964 (3)	0.0480 (11)
C1	0.1596 (5)	0.6215 (4)	0.0546 (4)	0.0367 (11)
C2	0.0346 (6)	0.6742 (5)	0.0674 (4)	0.0457 (13)
H2	−0.0101	0.6444	0.1138	0.068 (7)*
C3	−0.0254 (6)	0.7700 (5)	0.0128 (4)	0.0502 (14)
H3	−0.1096	0.8056	0.0215	0.068 (7)*
C4	0.0439 (6)	0.8108 (5)	−0.0547 (4)	0.0476 (13)
C5	0.0411 (7)	0.9018 (5)	−0.1342 (5)	0.0641 (16)
H5A	0.0602	0.9781	−0.1065	0.104 (8)*
H5B	−0.0470	0.8995	−0.1942	0.104 (8)*
C6	0.1832 (7)	0.8406 (5)	−0.1486 (5)	0.0650 (17)
H6A	0.1669	0.8072	−0.2159	0.104 (8)*
H6B	0.2742	0.8860	−0.1284	0.104 (8)*
C7	0.1696 (6)	0.7568 (5)	−0.0671 (4)	0.0462 (13)
C8	0.2313 (6)	0.6611 (4)	−0.0145 (4)	0.0449 (13)
H8	0.3147	0.6251	−0.0240	0.068 (7)*
C9	0.3604 (5)	0.4861 (4)	0.1372 (4)	0.0429 (12)
H9	0.4347	0.5185	0.1140	0.068 (7)*
C10	0.1468 (6)	0.4519 (5)	0.1661 (4)	0.0498 (14)
H10	0.0470	0.4580	0.1649	0.068 (7)*
C11	0.2442 (6)	0.3735 (5)	0.2151 (4)	0.0516 (14)
H11	0.2253	0.3148	0.2550	0.068 (7)*
C12	0.5169 (7)	0.3289 (5)	0.2334 (5)	0.0627 (17)
H12A	0.5421	0.2980	0.1751	0.104 (8)*
H12B	0.5009	0.2657	0.2743	0.104 (8)*
C13	0.6461 (7)	0.3988 (6)	0.2966 (5)	0.0742 (19)

H13A	0.7326	0.3497	0.3229	0.104 (8)*
H13B	0.6715	0.4547	0.2525	0.104 (8)*
C14	0.6175 (8)	0.4578 (8)	0.3828 (6)	0.089 (2)
H14	0.5856	0.4122	0.4276	0.068 (7)*
C15	0.6302 (9)	0.5617 (9)	0.4043 (7)	0.107 (3)
H15A	0.6616	0.6119	0.3625	0.104 (8)*
H15B	0.6082	0.5884	0.4620	0.104 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0601 (4)	0.0638 (4)	0.0677 (4)	−0.0167 (3)	0.0337 (3)	−0.0169 (3)
N1	0.040 (2)	0.041 (2)	0.033 (2)	−0.0065 (19)	0.0113 (18)	−0.0035 (19)
N2	0.057 (3)	0.042 (3)	0.045 (2)	0.003 (2)	0.016 (2)	0.005 (2)
C1	0.036 (3)	0.043 (3)	0.029 (2)	−0.007 (2)	0.006 (2)	−0.006 (2)
C2	0.040 (3)	0.064 (4)	0.033 (3)	−0.001 (3)	0.010 (2)	−0.002 (3)
C3	0.041 (3)	0.063 (4)	0.047 (3)	0.011 (3)	0.015 (3)	−0.003 (3)
C4	0.046 (3)	0.046 (3)	0.044 (3)	0.004 (3)	0.004 (3)	0.004 (3)
C5	0.069 (4)	0.059 (4)	0.061 (4)	0.015 (3)	0.014 (3)	0.011 (3)
C6	0.070 (4)	0.069 (4)	0.056 (4)	0.008 (3)	0.020 (3)	0.017 (3)
C7	0.047 (3)	0.052 (3)	0.037 (3)	−0.001 (3)	0.010 (3)	0.002 (3)
C8	0.040 (3)	0.053 (3)	0.044 (3)	0.003 (2)	0.017 (2)	−0.001 (3)
C9	0.039 (3)	0.047 (3)	0.041 (3)	0.001 (2)	0.012 (2)	0.004 (2)
C10	0.052 (3)	0.057 (3)	0.045 (3)	−0.017 (3)	0.021 (3)	−0.001 (3)
C11	0.058 (4)	0.048 (3)	0.050 (3)	−0.009 (3)	0.017 (3)	0.007 (3)
C12	0.069 (4)	0.050 (4)	0.072 (4)	0.014 (3)	0.024 (3)	0.010 (3)
C13	0.062 (4)	0.076 (5)	0.075 (5)	0.023 (4)	0.008 (4)	0.013 (4)
C14	0.075 (5)	0.110 (7)	0.067 (5)	0.015 (5)	−0.002 (4)	0.001 (5)
C15	0.078 (6)	0.118 (8)	0.104 (7)	0.006 (5)	−0.003 (5)	−0.025 (6)

Geometric parameters (Å, º)

N1—C9	1.317 (6)	C6—H6A	0.97
N1—C10	1.394 (6)	C6—H6B	0.97
N1—C1	1.444 (6)	C7—C8	1.368 (7)
N2—C9	1.329 (6)	C8—H8	0.93
N2—C11	1.367 (7)	C9—H9	0.93
N2—C12	1.466 (7)	C10—C11	1.327 (7)
C1—C2	1.380 (7)	C10—H10	0.93
C1—C8	1.396 (7)	C11—H11	0.93
C2—C3	1.374 (7)	C12—C13	1.499 (9)
C2—H2	0.93	C12—H12A	0.97
C3—C4	1.368 (7)	C12—H12B	0.97
C3—H3	0.93	C13—C14	1.463 (10)
C4—C7	1.390 (7)	C13—H13A	0.97
C4—C5	1.523 (8)	C13—H13B	0.97
C5—C6	1.576 (8)	C14—C15	1.255 (11)
C5—H5A	0.97	C14—H14	0.93

C5—H5B	0.97	C15—H15A	0.93
C6—C7	1.523 (7)	C15—H15B	0.93
C9—N1—C10	107.4 (4)	C4—C7—C6	93.2 (4)
C9—N1—C1	125.8 (4)	C7—C8—C1	114.9 (5)
C10—N1—C1	126.8 (4)	C7—C8—H8	122.6
C9—N2—C11	108.4 (5)	C1—C8—H8	122.6
C9—N2—C12	124.5 (5)	N1—C9—N2	109.2 (4)
C11—N2—C12	127.1 (5)	N1—C9—H9	125.4
C2—C1—C8	122.3 (5)	N2—C9—H9	125.4
C2—C1—N1	119.1 (4)	C11—C10—N1	107.4 (5)
C8—C1—N1	118.5 (4)	C11—C10—H10	126.3
C3—C2—C1	121.4 (5)	N1—C10—H10	126.3
C3—C2—H2	119.3	C10—C11—N2	107.6 (5)
C1—C2—H2	119.3	C10—C11—H11	126.2
C4—C3—C2	117.2 (5)	N2—C11—H11	126.2
C4—C3—H3	121.4	N2—C12—C13	112.6 (5)
C2—C3—H3	121.4	N2—C12—H12A	109.1
C3—C4—C7	121.0 (5)	C13—C12—H12A	109.1
C3—C4—C5	145.3 (5)	N2—C12—H12B	109.1
C7—C4—C5	93.7 (4)	C13—C12—H12B	109.1
C4—C5—C6	86.3 (4)	H12A—C12—H12B	107.8
C4—C5—H5A	114.3	C14—C13—C12	114.4 (6)
C6—C5—H5A	114.3	C14—C13—H13A	108.7
C4—C5—H5B	114.3	C12—C13—H13A	108.7
C6—C5—H5B	114.3	C14—C13—H13B	108.7
H5A—C5—H5B	111.4	C12—C13—H13B	108.7
C7—C6—C5	86.7 (4)	H13A—C13—H13B	107.6
C7—C6—H6A	114.2	C15—C14—C13	128.6 (9)
C5—C6—H6A	114.2	C15—C14—H14	115.7
C7—C6—H6B	114.2	C13—C14—H14	115.7
C5—C6—H6B	114.2	C14—C15—H15A	120.0
H6A—C6—H6B	111.4	C14—C15—H15B	120.0
C8—C7—C4	123.2 (5)	H15A—C15—H15B	120.0
C8—C7—C6	143.5 (5)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···Br	0.93	2.66	3.561 (5)	165
C11—H11···Br[i]	0.93	2.87	3.697 (6)	149
C13—H13A···Cg1[i]	0.97	2.91	3.735 (7)	143

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