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trans,trans-2,3,4-Triphenylazetidinium bromide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.042
wR factor = 0.097
Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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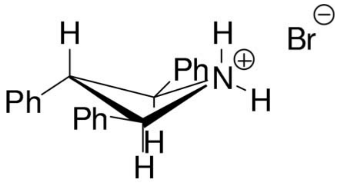
In the crystal structure of the title compound, $C_{21}H_{20}N^{+}\cdot Br^{-}$, two azetidinium cations act as hydrogen-bond donors and two bromide anions as acceptors, forming hydrogen-bonded dimers. The hydrogen-bond motif is that of the well known $R_2^4(6)$ ring. The degree of folding of the central azetidine ring is indicated by the torsion angles, one of which is 19.5 (3)°.

Comment

As part of our long-standing interest in compounds with the triphenylpropane skeleton, differently *N*-substituted diastereoisomeric 2,3,4-triphenylazetidines have been synthesized (Kurteva *et al.*, 1993; Kurteva & Lyapova, 2000, 2005). The triphenyl compounds are useful models for conformational relationship studies, owing to their symmetry and the limited number of allowed conformations. Heterocyclic systems of this type are of special interest owing to the tendency of the phenyl groups to adopt axial as opposed to equatorial positions in the conformations of the various isomers. Triphenylazetidinium bromides have been synthesized by acid hydrolysis of the corresponding benzylideneamino azides (Kurteva & Lyapova, 2005). Applying the relationships found by Abraham *et al.* (1972) and by Kingsbury *et al.* (1982) when analysing the NMR data, and comparing with known triphenyl *N*-methyl azetidines, it was suggested that in the *trans*,*trans*-isomer an *N*-substitution does not significantly influence the ring torsion angles. In contrast, quaternization results in an increase of the torsion angles and strongly biases the equilibrium towards the conformer with e,e,e phenyl groups. The number of structurally characterized azetidinium cations with a 2,3,4-substitution scheme is limited, to the best of our knowledge, to three cases [Cambridge Structural Database (Version 5.26; Allen, 2002) refcodes AZPCOH, DALWIV and YAZYON].



(I)

The title compound crystallizes in the triclinic space group $P\bar{1}$, with one azetidinium cation and one bromide anion in the asymmetric unit. The C—N and C—C distances in the cation are in the expected ranges (Table 1). The puckering angle, C4—N1—C2—C3, of the azetidinium ring is 19.5 (3)°. The

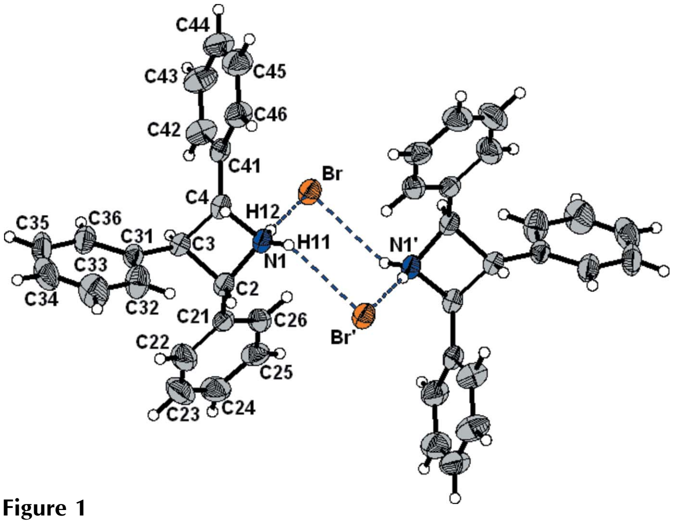


Figure 1
Hydrogen-bonded dimeric structure of the title compound. H atoms are drawn with an arbitrary radii and displacement ellipsoids are drawn at the 50% probability level. N1′, Br′ and unlabelled atoms are related by ($-x$, $1 - y$, $1 - z$).

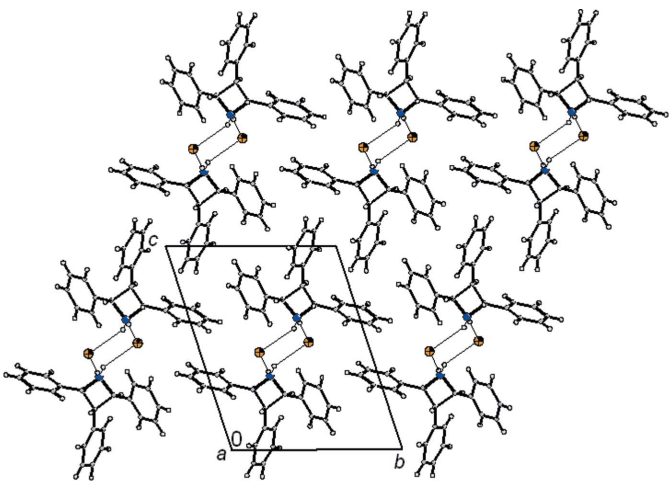


Figure 2
Packing diagram of the title compound, viewed along [$\bar{1}$00]. Dashed lines indicate hydrogen bonds.

C—N—C, C—C—C and C—C—N angles of the azetidinium ring are close to 90°. Two azetidinium cations act as hydrogen bond donors and two bromide anions as acceptors, forming hydrogen-bonded dimers (Table 2). The hydrogen-bond motif is that of the well known $R_2^4(6)$ ring (Fig. 1) (Etter *et al.*, 1990). These dimers are packed in the *bc* plane with the phenyl substituents geared with neighbouring ones to give an excellent fit (Fig. 2).

Experimental

The synthesis and spectroscopic characterization were reported within the chemical context before this structure determination was undertaken (Kurteva & Lyapova, 2005). Diffraction quality single crystals of the title compound have been grown from ethanol solutions at room temperature within several weeks.

Crystal data

$C_{21}H_{20}N^+\cdot Br^-$
$M_r = 366.29$
Triclinic, $P\bar{1}$
$a = 6.0658$ (6) Å
$b = 10.9797$ (11) Å
$c = 13.8090$ (13) Å
$\alpha = 107.500$ (9)°
$\beta = 90.460$ (8)°
$\gamma = 92.119$ (8)°
$V = 876.35$ (15) Å^3
$Z = 2$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.35$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.30 × 0.15 × 0.15 mm

Data collection

Stoe Stadi CCD diffractometer
ω scans
Absorption correction: none
9652 measured reflections
3062 independent reflections
2719 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.060$
$\theta_{\max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.097$
$S = 1.02$
3062 reflections
229 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$\Delta\rho_{\min} = -1.02$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0058 (15)

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.513 (4)	C3—C31	1.501 (5)
N1—C4	1.536 (4)	C3—C4	1.538 (5)
C2—C21	1.494 (5)	C4—C41	1.498 (5)
C2—C3	1.551 (4)		
C2—N1—C4	89.0 (2)	C4—C3—C2	87.6 (2)
N1—C2—C3	88.5 (2)		
C4—N1—C2—C3	19.5 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H12···Br	0.82 (4)	2.51 (4)	3.317 (3)	170 (4)
N1—H11···Br$^{\rm i}$	0.84 (5)	2.70 (5)	3.331 (3)	133 (4)

Symmetry code: (i) $-x, -y + 1, -z + 1$.

The atomic coordinates of the H atoms belonging to NH_2 and the Csp^3—H groups were refined freely; their $U_{\rm iso}$ values were refined individually. H atoms belonging to the phenyl groups were included in the refinement, riding on their attached C atoms, with $U_{\rm iso}$(H) set to $1.2U_{\rm eq}$(carrier atom) and C—H = 0.93 Å. The deepest difference electron density hole is located 0.96 Å from Br.

Data collection: *CrysAlis CCD* (Kuma Diffraction, 2000); cell refinement: *CrysAlis RED* (Kuma Diffraction, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

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