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N-Benzyl-1-(dimethylamino)-2-methyl-1-oxopropan-2-aminium bromide

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.006 Å
R factor = 0.058
wR factor = 0.108
Data-to-parameter ratio = 21.0

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

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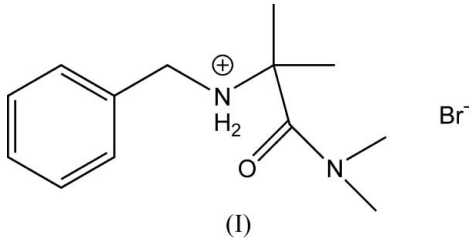
The title compound, $C_{13}H_{21}N_2O^+\cdot Br^-$, was crystallized from a CH_2Cl_2 solution of N^2,N^2-dibenzyl-*N*,*N*,2-trimethylalaninamide and BBr_3. The geometry of the cation is unexceptional. Intermolecular N—H···O and N—H···Br hydrogen bonds link two cations and two anions into a hydrogen-bonded cluster. The crystal packing is further stabilized by van der Waals forces.

Comment

We are currently studying the reactions of different amine-amides such as N^2,N^2-dibenzyl-*N*,*N*,2-trimethylalaninamide and boron halides. In such a synthesis involving BBr_3, we obtained single crystals of the title compound, (I) (Fig. 1). Here we present its crystal structure.



(I)

The geometry of the *N*-benzyl-1-(dimethylamino)-2-methyl-1-oxopropan-2-aminium ion is unexceptional. In the crystal structure, two amino H atoms are involved in hydrogen bonds, where the $O_{carbonyl}$ atom and the bromide ion act as acceptors (Table 1). These hydrogen bonds link two cations and two anions into a hydrogen-bonded cluster. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Hydrogen bonding between NH hydrogens and bromide is a common feature in the structural chemistry of aminium bromides. The H···Br distance of 2.28 Å observed in (I) is of the same order of magnitude as in similar compounds of this type. A few examples of simple structures of this type are 1,7-heptanediamine dihydrobromide (Brisson & Brisse, 1984*a*), 1,8-octanediamine dihydrobromide (Brisson & Brisse, 1984*b*; Baur & Tillmanns, 1986), iminodiacetic acid hydrobromide (Oskarsson, 1976) and tert-butylammonium bromide (Shabazi *et al.*, 1992).

Experimental

A solution of 0.075 mmol of N^2,N^2-dibenzyl-*N*,*N*,2-trimethylalaninamide in 2 ml of CH_2Cl_2 under dry nitrogen was treated with one equivalent of BBr_3 (1 ml solution in CH_2Cl_2) and the resulting solution was overlayered with 4 ml of dry hexane at 296 K and stored at 253 K. After two months, small colourless blocks of (I) had formed.

Crystal data

$C_{13}H_{21}N_2O^+\cdot Br^-$
M_r = 301.23
Orthorhombic, *Pbca*
a = 15.7845 (8) Å
b = 10.6592 (14) Å
c = 16.872 (2) Å
V = 2838.7 (5) Å^3
Z = 8
D_x = 1.410 Mg m^{-3}
Mo *Kα* radiation
μ = 2.88 mm^{-1}
T = 299 K
Block, colourless
0.33 × 0.30 × 0.27 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ scans
Absorption correction: numerical (*HABITUS*; Herrendorf & Bärnighausen, 1997)
T_{min} = 0.476, T_{max} = 0.567
18982 measured reflections
3239 independent reflections
1828 reflections with $I > 2\sigma(I)$
R_{int} = 0.099
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.108
S = 1.07
3239 reflections
154 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.014P)^2 + 6.423P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1	0.94	2.28	3.201 (3)	166
N1—H1*B*···O1^{i}	0.93	2.09	2.927 (4)	151

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

H atoms bonded to N1 and C7 were located in a difference Fourier map with distances N—H = 0.93–0.94 Å and C—H = 1.00 Å. The remaining H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (aliphatic). All H atoms were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) or U_{iso}(H) = 1.5U_{eq}(methyl C).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *MAXUS* (Mackay *et al.*, 1999).

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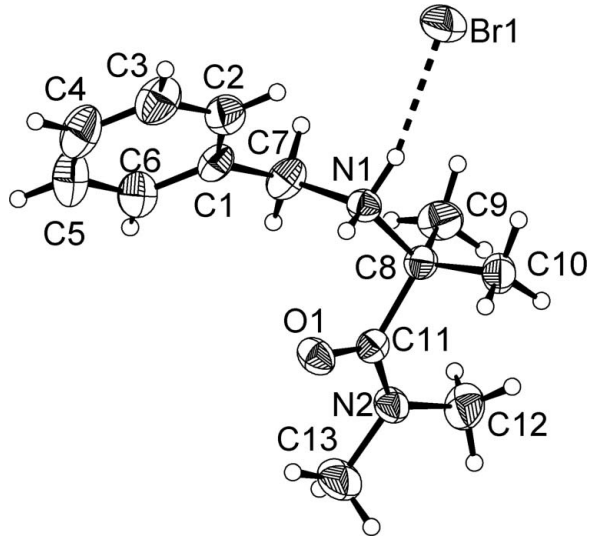

Figure 1
View of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level. The dashed line denotes a hydrogen bond.

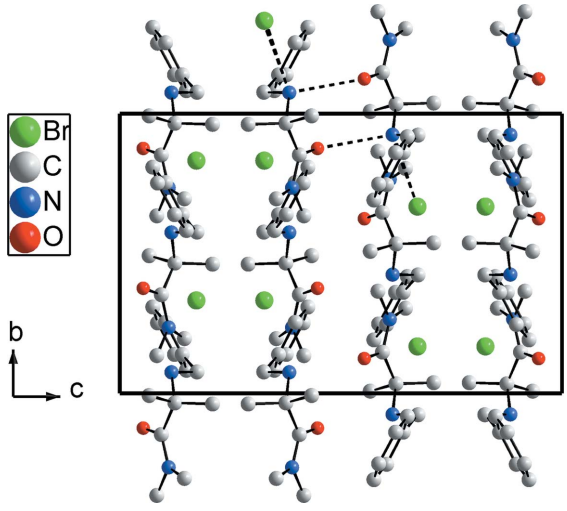

Figure 2
The crystal packing, viewed along the *a* axis. Hydrogen bonds within one hydrogen-bonded cluster are indicated by dashed lines. H atoms have been omitted for clarity.

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