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Key indicators

Single-crystal X-ray study
T = 299 K
 Mean σ (C–C) = 0.010 Å
R factor = 0.054
wR factor = 0.134
 Data-to-parameter ratio = 19.7

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

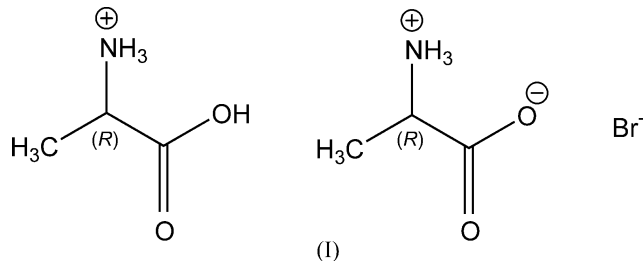
D-Alanine alaninium bromide

The title compound, $C_3H_7NO_2^+ \cdot Br^- \cdot C_3H_8NO_2$, crystallizes from an aqueous solution of D-alanine and racemic 2,3-dibromosuccinic acid. The structure includes alaninium cations and neutral zwitterionic alanine, and displays N–H···Br, N–H···O and O–H···O hydrogen bonding.

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Comment

We are currently studying the structural chemistry of substituted succinic acids such as dibromosuccinic acid. In an attempt to crystallize enantiomerically pure 2,3-dibromosuccinic acid (2,3-DBSA), an aqueous solution of D-alanine and the racemic acid was allowed to evaporate slowly at room temperature. The crystallization product was the title compound, (I), probably formed as the result of an elimination reaction. It has been observed previously that HBr is readily eliminated from racemic 2,3-DBSA, even at room temperature and in the presence of rather weak bases (Fischer, 2006*a,b*).



The structure of (I) includes neutral zwitterionic D-alanine, D-alaninium cations and bromide anions (Fig. 1). The

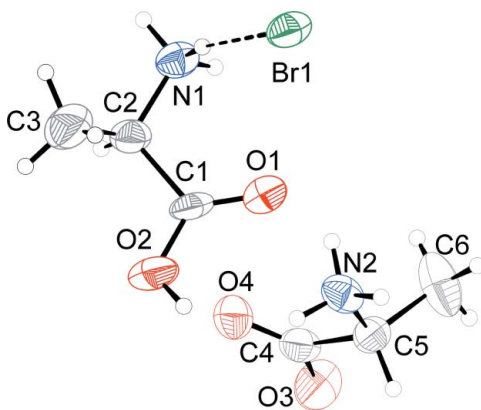


Figure 1
 The asymmetric unit of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. The dashed line denotes an N–H···Br hydrogen bond.

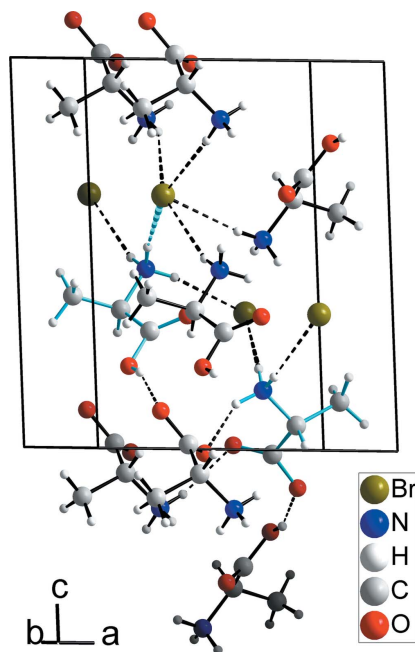


Figure 2
The hydrogen-bonding pattern in (I). H bonds are shown as dashed lines. Components of the asymmetric unit are indicated by light-blue bonds.

geometry of the two alanine species is unexceptional. All six H atoms bound to N are involved in hydrogen bonds, five of which are N—H···Br and one N—H···O (Table 1). Additionally, one O—H···O hydrogen bond is present (Table 1). The hydrogen bonds lead to the formation of a complex three-dimensional network (Fig. 2).

Co-crystallization of alaninium cations and neutral (zwitterionic) alanine in the presence of a suitable anion has been observed previously for nitrate (Ramos Silva *et al.*, 2001). In the alanine/nitrate system, alaninium nitrate (1:1) has also been prepared and structurally characterized (Bahadur & Rajaram, 1995). As far as alaninium halides are concerned, only DL-alanine hydrochloride (Trotter, 1962) and L-alanine hydrochloride (di Blasio *et al.* 1977) have so far been characterized. The question of whether alaninium halides exhibit the same behaviour as the nitrate will be the subject of further investigations.

Experimental

The synthesis of racemic 2,3-dibromosuccinic acid has been described previously (Fischer, 2006c). 133 mg of the acid were dissolved together with 43 mg of D-alanine (Sigma) in 5.52 g of demineralized water. The solution was left to stand for evaporation, and the title compound crystallized within a couple of days.

Crystal data

$C_3H_7NO_2^+ \cdot Br^- \cdot C_3H_8NO_2$	$Z = 2$
$M_r = 259.10$	$D_x = 1.561 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 9.485 (4) \text{ \AA}$	$\mu = 3.72 \text{ mm}^{-1}$
$b = 5.1678 (6) \text{ \AA}$	$T = 299 \text{ K}$
$c = 11.258 (2) \text{ \AA}$	Plate, colourless
$\beta = 92.97 (3)^\circ$	$0.31 \times 0.19 \times 0.06 \text{ mm}$
$V = 551.1 (3) \text{ \AA}^3$	

Data collection

Bruker-Nonius KappaCCD diffractometer	6831 measured reflections
φ and ω scans	2422 independent reflections
Absorption correction: numerical (HABITUS; Herrendorf & Bärnighausen, 1997)	2127 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.378$, $T_{\max} = 0.774$	$R_{\text{int}} = 0.070$
	$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2 + 1.5036P]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.134$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.15$	$\Delta\rho_{\max} = 0.80 \text{ e \AA}^{-3}$
2422 reflections	$\Delta\rho_{\min} = -0.49 \text{ e \AA}^{-3}$
123 parameters	Absolute structure: Flack (1983), 1033 Friedel pairs
H-atom parameters constrained	Flack parameter: 0.07 (3)

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A···Br1	0.89	2.52	3.399 (9)	170
N1—H1B···Br1 ⁱ	0.89	2.59	3.426 (9)	156
N1—H1C···Br1 ⁱⁱ	0.89	2.54	3.408 (5)	167
N2—H2A···Br1 ⁱⁱ	0.89	2.60	3.484 (9)	174
N2—H2B···Br1 ⁱⁱⁱ	0.89	2.60	3.424 (8)	155
N2—H2C···O4 ^{iv}	0.89	2.03	2.802 (7)	144
O2—H2D···O3 ^{iv}	0.82	1.69	2.431 (8)	150

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

H atoms were visible in difference Fourier maps, but were placed in idealized positions with N—H = 0.89 \AA , C—H = 0.98 \AA , $C_{\text{methyl}}\text{—H} = 0.96 \text{ \AA}$ and O—H = 0.82 \AA . They were allowed to ride during subsequent refinement with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$. The methyl and NH_3 groups were allowed to rotate about their local threefold axes.

Data collection: COLLECT (Nonius, 1999); cell refinement: DIRAX/LSQ (Duisenberg, 1992); data reduction: EVALCCD (Duisenberg *et al.*, 2003); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); 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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