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β-Alanine β-alaninium picrate

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In the title compound, $C_3H_7NO_2\cdot C_3H_8NO_2^+\cdot C_6H_2N_3O_7^-$, each β-alanine residue forms an asymmetric O—H···O hydrogen bond with its inversion-related partner [O···O = 2.477 (4) and 2.472 (4) Å], involving disorder of carboxylic acid and carboxylate groups. The crystal structure is further stabilized by N—H···O hydrogen bonds.

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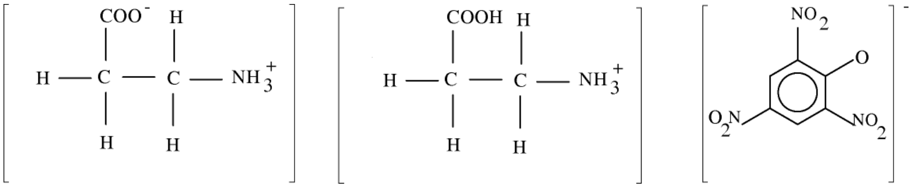
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.193
Data-to-parameter ratio = 14.3

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

Comment

The present work was undertaken to study the behaviour of hydrogen bonding in amino acid–organic acid complexes. In this case, one proton is released by the organic acid and is shared by two amino acid molecules in disordered hydrogen bonds across inversion centers. This results in short, asymmetric O—H···O hydrogen bonds. This type of bond is observed in complexes such as triglycinium sulfate (Kay, 1977), L-phenylalanine L-phenylalaninium perchlorate (Srinivasan & Rajaram, 1997), hydrogen bis[L-lysinium(2+)] dichloride perchlorate (Srinivasan *et al.*, 2001*a*), L-lysine L-lysinium dichloride nitrate (Srinivasan *et al.*, 2001*b*), L-phenylalanine–nitric acid (2/1) (Srinivasan *et al.*, 2001*c*), bis(L-proline) hydrogen perchlorate (Pandiarajan *et al.*, 2002) and bis(β-alanine) hydrogen nitrate (Sridhar *et al.*, 2001). A similar structure, L-phenylalanine L-phenylalaninium formate (Görbitz & Etter, 1992), has also been reported.



(I)

The asymmetric unit of the title compound contains two β-alanine residues and a picrate anion. The backbone conformation angles ψ^1 and ψ^2 are *cis* and *trans*, respectively, for β-alanine residue I, while for residue II, ψ^1 and ψ^2 are *trans* and *cis*, respectively (Fig. 1). In general, the carbonyl O atom is *cis* and the carboxyl O atom is in a *trans* configuration. However, in the case of β-alanine residue II, the conformation is reversed, as found in L-argininium dinitrate (Ramaswamy *et al.*, 2001). The straight-chain conformation angle χ^1 is in *gauche* II form for residue I, while it is in *trans* form for residue II.

The picrate anion plays a vital role in forming hydrogen bonds. The degree of twisting of the nitro groups from the benzene mean plane does not depend upon the C—N bond distances (Soriano-Garcia *et al.*, 1978). The nitro groups are not coplanar with the benzene ring of the picrate anion [O2—

N1—C1—C6 −30.2 (4)°, C2—C3—N2—O4 14.9 (4)°, C6—C5—N3—O6 20.0 (4)°].

The proton dissociated from the picric acid is disordered and is bonded to the carboxyl group of both the alanine residues (50% each). The geometry of the carboxyl groups also confirms the protonation. Each alanine residue is bonded to its inversion-related alanine residue through strong O—H⋯O hydrogen bonds. The picrate anion is linked to the alanine residues through N—H⋯O hydrogen bonds involving the deprotonated oxygen and nitro O atoms. The amino N atom in residue I links two symmetry-related residues II through N—H⋯O hydrogen bonds involving the carboxyl groups. Similarly, the amino N atom in residue II links two symmetry-related residues I. In residue I, an intramolecular N—H⋯O hydrogen bond is observed. A class II hydrogen-bonding pattern is observed in *β*-alanine residue I, which involves two two-centered and one three-centered hydrogen bonds. In the case of residue II, a class III hydrogen-bonding pattern is observed, with one two-centered and two three-centered hydrogen bonds. No classical head-to-tail sequence is observed in the present structure. The O atoms of the picrate anion which do not take part in hydrogen bonding have large U_{eq} values.

Experimental

The title compound was crystallized by slow evaporation of an equimolar solution of *β*-alanine and picric acid.

Crystal data

$C_3H_7NO_2 \cdot C_3H_8NO_2^+ \cdot C_6H_2N_3O_7^-$
$M_r = 407.31$
Triclinic, $P\overline{1}$
$a = 4.9515$ (4) Å
$b = 11.725$ (2) Å
$c = 14.931$ (2) Å
$\alpha = 78.00$ (1)°
$\beta = 83.75$ (1)°
$\gamma = 82.45$ (1)°
$V = 837.5$ (2) Å^3
$Z = 2$
$D_x = 1.615$ Mg m^{-3}
$D_m = 1.611$ Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetrachloride and xylene
Mo *Kα* radiation
Cell parameters from 25 reflections
$\theta = 9.9$–14.1°
$\mu = 0.14$ mm^{-1}
$T = 293$ (2) K
Needle, yellow
0.40 × 0.20 × 0.15 mm

Data collection

Nonius MACH3 four-circle diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.966$, $T_{max} = 0.979$
4194 measured reflections
3646 independent reflections
2537 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.0°$
$h = -6 \rightarrow 6$
$k = -1 \rightarrow 14$
$l = -18 \rightarrow 19$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.193$
$S = 1.03$
3646 reflections
255 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1203P)^2 + 0.2829P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

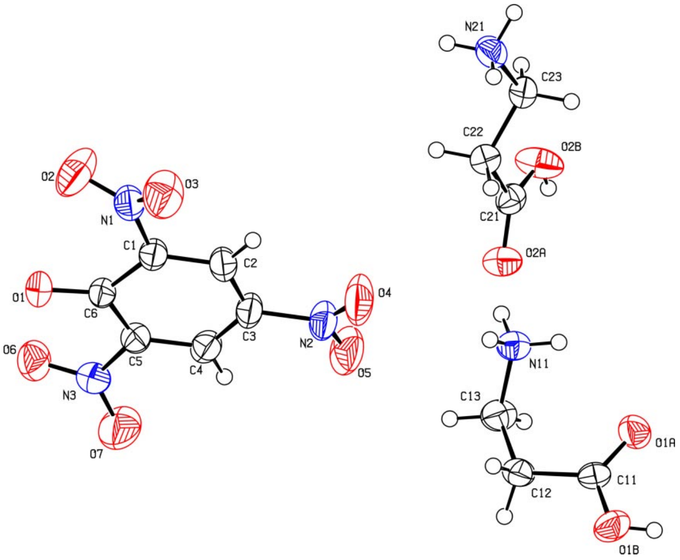


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids (Johnson, 1976) and the atom-numbering scheme. H atoms attached to atoms O1*B* and O2*B* each have an occupancy of 0.50.

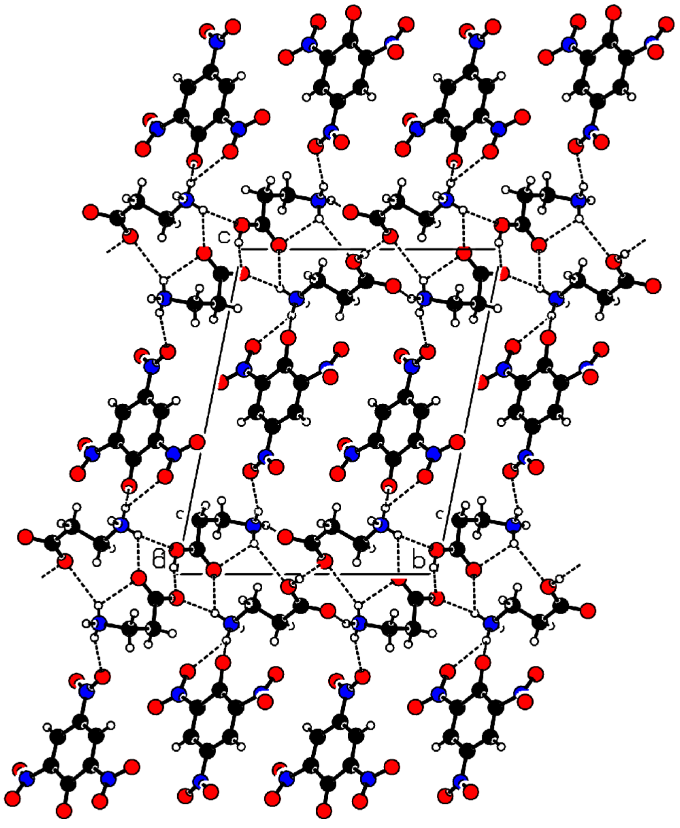


Figure 2
The packing of the molecules of (I), viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

Acta Cryst. (2004). E**60**, o1630–o1632 K. Anitha *et al.* • $C_3H_7NO_2 \cdot C_3H_8NO_2^+ \cdot C_6H_2N_3O_7^-$ **o1631**

Table 1
Selected geometric parameters (Å, °).

O1*A*—C11	1.225 (3)	O2*A*—C21	1.222 (3)
O1*B*—C11	1.289 (3)	O2*B*—C21	1.295 (3)
O1*A*—C11—O1*B*	123.9 (2)	O2*A*—C21—O2*B*	122.9 (2)
O1*A*—C11—C12	121.6 (2)	O2*A*—C21—C22	120.8 (2)
O1*B*—C11—C12	114.5 (2)	O2*B*—C21—C22	116.3 (2)
O2—N1—C1—C6	−30.2 (4)	C11—C12—C13—N11	−62.8 (3)
O4—N2—C3—C2	14.9 (4)	O2*A*—C21—C22—C23	−157.1 (2)
O6—N3—C5—C6	20.0 (4)	O2*B*—C21—C22—C23	25.2 (3)
O1*A*—C11—C12—C13	5.3 (4)	C21—C22—C23—N21	−174.6 (2)
O1*B*—C11—C12—C13	−176.6 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O1*B*^{i}	0.82	1.70	2.477 (4)	157
N11—H11*A*···O2*A*^{ii}	0.89	1.93	2.775 (3)	159
N11—H11*B*···O1*A*	0.89	2.28	2.885 (3)	126
N11—H11*B*···O2*B*^{iii}	0.89	2.48	3.188 (3)	137
N11—H11*C*···O5	0.89	2.09	2.904 (3)	153
O2*B*—H2*B*···O2B^{iv}	0.82	1.66	2.472 (4)	170
N21—H21*A*···O1*B*^{v}	0.89	2.19	2.894 (3)	136
N21—H21*A*···O1*A*^{vi}	0.89	2.27	2.862 (3)	124
N21—H21*B*···O1	0.89	2.03	2.830 (3)	148
N21—H21*B*···O6	0.89	2.28	2.919 (3)	128
N21—H21*C*···O1^{vii}	0.89	2.63	3.281 (3)	131

Symmetry codes: (i) $3 - x, -y, -z$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $x, y, z - 1$; (iv) $2 - x, 1 - y, 2 - z$; (v) $2 - x, -y, 1 - z$; (vi) $x - 1, y, 1 + z$; (vii) $x - 1, y, z$.

The H atom dissociated from the picric acid as a proton is disordered and is bonded to the carboxyl group of both the alanine molecules (50% each, as required by the centrosymmetric arrangement for the disordered O---H···O hydrogen bonding). All the H atoms were placed in geometrically calculated positions [O—H = 0.82 Å, N—H = 0.89 Å and C—H = 0.93–0.97 Å] and included in the refinement in the riding-model approximation, with $U_{\rm iso}$ equal to $1.2U_{\rm eq}$ of the carrier atom ($1.5U_{\rm eq}$ for methyl and NH$_3$ H atoms).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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