L-Aspartic acid nitrate–L-aspartic acid (1/1)

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L-Aspartic acid nitrate–L-aspartic acid (1/1)

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In the title compound, $C_4H_8NO_4^+\cdot C_4H_7NO_4\cdot NO_3^-$, the cation and neutral molecule are connected by an asymmetric hydrogen bond. The cation, residue 1, exists in a *gauche* I conformation, whereas the neutral molecule, residue 2, exhibits a *gauche* II conformation. A *syn–syn* orientation is also observed in this structure and residue 1 is involved in a straight (*S*1) head-to-tail sequence. The structure is stabilized by both inter- and intramolecular N—H⋯O hydrogen bonding.

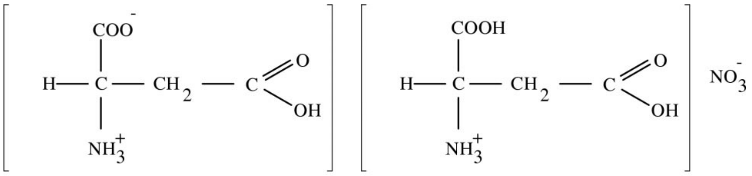
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.041
wR factor = 0.118
Data-to-parameter ratio = 7.4

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

Comment

Aspartic acid is a non-essential amino acid, widely distributed in proteins, which plays a major role in the energy cycle of the human body. The crystal structures of L-aspartic acid (Derissen *et al.*, 1968), DL-aspartic acid (Rao, 1973; Sequeira *et al.*, 1989), DL-aspartic acid nitrate monohydrate (Asath Bahadur & Rajaram, 1995) and bis(DL-aspartic acid) sulfate (Srinivasan *et al.*, 2001) have been reported. In the present paper, the crystal structure of the product, (I), of the reaction of L-aspartic acid with nitric acid is reported.



(I)

The asymmetric unit of (I) contains one protonated aspartic acid molecule (residue 1), one neutral aspartic acid molecule (residue 2) and one nitrate anion. Superposition of residue 1 on residue 2 results in an r.m.s. deviation of the constituent atoms of 1.088 Å. Examination of the coordinates suggests that the two residues might be related by a pseudo-inversion centre. The unsymmetrical carboxyl bond distances and angles [1.217 (7)/1.297(6) Å and 122.3 (5)/109.9(5)°] of residue 1 clearly indicate protonation of the carboxyl group, whilst, in the case of residue 2, the equality of C—O bond distances [1.240 (7)/1.250(6) Å] and O—C—C bond angles [117.4 (5)/115.5(5)°] represent the deprotonated carboxylate group (Table 1).

The backbone conformation angle ψ^1 is the *cis* form for both residues [O1*A*—C11—C12—N1 −7.0 (7)° and O2*A*—C21—C22—N21 −1.7 (7)°]. The deviations of the amino atoms N11 and N21 from the planar carboxyl groups at C11 and C22 are 0.198 (8) and 0.044 (1) Å, respectively. This non-planarity of the amino nitrogen and carboxyl group is also found in other amino acids (Lakshminarayanan *et al.*, 1967).

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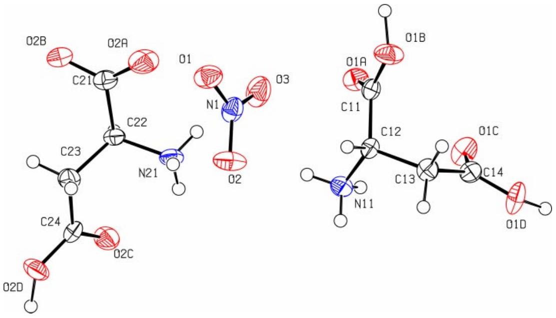

Figure 1
The molecular structure, with the atom-numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976).

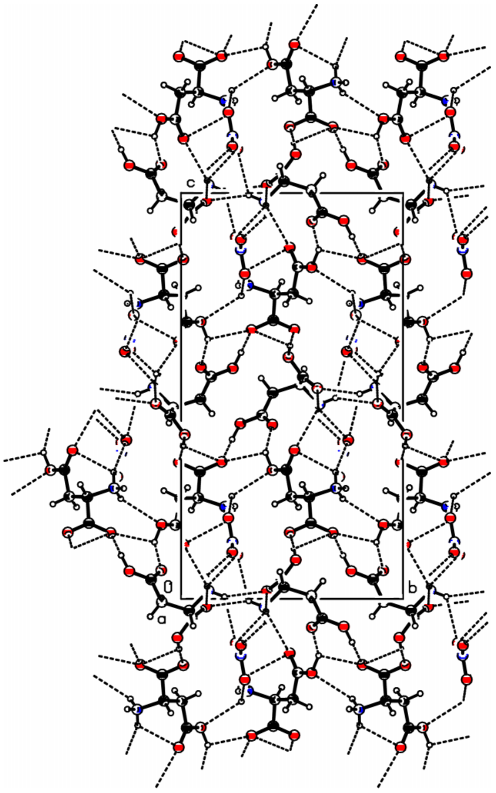

Figure 2
Packing diagram of the title molecule, viewed down the *a* axis.

The side-chain conformation angle χ^1 is in a *gauche* I form [66.1 (6)°] for residue 1 and a *gauche* II form [−64.8 (6)°] for residue 2. The branched chain conformation angles, χ^{11} and χ^{21}, are in *cis* and *trans* forms [10.0 (8)/−5.5 (8) and −170.8 (5)/174.3(5)°] for both residues. In residue 1, the C^γ atom C14 is in the *gauche* II [−58.1 (6)°] conformation with respect to C11, while, in the case of residue 2, the C^γ atom C24 is *trans* [170.9 (5)°] with respect to C21.

The average N—O and O—N—O values are 1.248 Å and 120°, respectively, clearly showing the nearly ideal trigonal symmetry of the anion, which plays a vital role in hydrogen bonding and the resulting stabilization of the structure.

The aspartic acid cation and neutral aspartic acid molecule are linked, by strong O—H···O hydrogen bonding, to form dimers. This hydrogen bond may be termed an asymmetric hydrogen bond, since the H atom is closer to one of the O atoms of the carboxyl group (Olovsson *et al.*, 2001). Atom H1*B* is in a *syn* orientation with respect to both donor carboxyl group and acceptor carboxylate group; the torsion angles H1*B*—O1*B*—C11—O1*A* and H1*B*—O2*B*^{i}—C21^{i}—O2*A*^{i} [symmetry code: (i) $1 + x, y, z$] are 13 (5) and 29 (3)°, respectively. This type of *syn–syn* orientation is also found in betaine betainium oxalate (Rodrigues *et al.*, 2001). The β-carboxyl group of residue 1 forms a strong O—H···O hydrogen bond with the α-carboxylate group of residue 2. In the case of residue 2, the β-carboxyl group forms a rather strong O—H···O hydrogen bond with the carbonyl O atom of the β-carboxyl group of residue 1.

The amino N atom of both residues forms N—H···O hydrogen bonds with the nitrate anion, and the α- and β-carboxyl groups. Three-centred hydrogen bonding is observed in both residues. Interestingly, in residue 1, three such three-centred bonds are observed, leading to a class IV hydrogen-bonding pattern (Jeffrey & Saenger, 1991). The frequency of such class IV hydrogen-bonding patterns is very low. A class II hydrogen-bonding pattern is observed in residue 2, having two two-centred and one three-centred hydrogen bonds. In both residues, intramolecular N—H···O hydrogen bonding is present, involving the amino nitrogen and the carbonyl oxygen of a carboxylic acid group. A straight (*S*1) head-to-tail sequence is observed in residue 1, connecting two amino acids separated by a unit translation (Vijayan, 1988). Each aspartic acid residue is linked by the nitrate anion through N—H···O hydrogen bonding, forming a chain running along the *a* axis: (*a*) O2($x - 1, y, z$)···H11*B*—N11—H11*A*···O3($x - 2, y, z$) and (*b*) O1($-\frac{1}{2} - x, 2 - y, z - \frac{1}{2}$)···H21*A*—N21—H21*B*···O3($\frac{1}{2} - x, 2 - y, z - \frac{1}{2}$).

Experimental

The title compound was crystallized by slow evaporation of an aqueous solution of L-aspartic acid and nitric acid in a 2:1 stoichiometric ratio.

Crystal data

$C_4H_8NO_4^+ \cdot C_4H_7NO_4 \cdot NO_3^-$
$M_r = 329.23$
Orthorhombic, $P2_12_12_1$
$a = 5.5840$ (7) Å
$b = 11.491$ (3) Å
$c = 21.043$ (5) Å
$V = 1350.2$ (5) Å^3
$Z = 4$
$D_x = 1.620$ Mg m^{-3}
$D_m = 1.58$ Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetrachloride and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.0$–14.7°
$\mu = 0.15$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.5 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.932$, $T_{max} = 0.968$
1640 measured reflections
1578 independent reflections
929 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 6$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 24$
3 standard reflections
frequency: 60 min
intensity decay: none

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Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.118$
$S = 1.02$
1578 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.0829P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.011 (3)

Table 1
Selected geometric parameters (Å, °).

O1*A*—C11	1.217 (7)	O2*A*—C21	1.240 (7)
O1*B*—C11	1.297 (6)	O2*B*—C21	1.250 (6)
C14—O1*C*	1.215 (6)	C24—O2*C*	1.208 (6)
C14—O1*D*	1.306 (6)	C24—O2*D*	1.319 (7)
O1*A*—C11—C12	122.3 (5)	O2*A*—C21—C22	117.4 (5)
O1*B*—C11—C12	109.9 (5)	O2*B*—C21—C22	115.5 (5)
O1*A*—C11—C12—N11	−7.0 (7)	O2*A*—C21—C22—N21	−1.7 (7)
N11—C12—C13—C14	66.1 (6)	N21—C22—C23—C24	−64.8 (6)
C11—C12—C13—C14	−58.1 (6)	C21—C22—C23—C24	170.9 (5)
C12—C13—C14—O1*C*	10.0 (8)	C22—C23—C24—O2*C*	−5.5 (8)
C12—C13—C14—O1*D*	−170.8 (5)	C22—C23—C24—O2*D*	174.3 (5)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O2*B*$^{\text{i}}$	1.05 (8)	1.47 (8)	2.512 (5)	175 (8)
N11—H11*A*···O1*A*$^{\text{ii}}$	0.89	2.39	3.126 (6)	140
N11—H11*A*···O3$^{\text{iii}}$	0.89	2.50	3.050 (6)	121
N11—H11*B*···O2$^{\text{iv}}$	0.89	2.12	2.793 (6)	131
N11—H11*B*···O1*C*	0.89	2.63	3.170 (6)	120
N11—H11*C*···O1*A*$^{\text{v}}$	0.89	2.25	3.018 (5)	145
N11—H11*C*···O3$^{\text{vi}}$	0.89	2.58	3.192 (6)	127
O1*D*—H1*D*···O2*A*$^{\text{vi}}$	0.96 (6)	1.68 (6)	2.594 (5)	156 (6)
N21—H21*A*···O1$^{\text{vii}}$	0.89	2.20	2.892 (6)	134
N21—H21*B*···O3$^{\text{viii}}$	0.89	2.34	3.155 (6)	152
N21—H21*B*···O2*C*	0.89	2.47	3.040 (5)	122
N21—H21*C*···O2*D*$^{\text{ix}}$	0.89	2.53	3.111 (6)	124
O2*D*—H2*D*···O1*C*$^{\text{x}}$	1.06 (6)	1.74 (6)	2.699 (5)	148 (5)

Symmetry codes: (i) $1+x, y, z$; (ii) $x-1, y, z$; (iii) $x, y-1, z$; (iv) $1+x, y-1, z$; (v) $x-\frac{1}{2}, -\frac{1}{2}-y, 1-z$; (vi) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$; (vii) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; (viii) $\frac{3}{2}-x, 1-y, z-\frac{1}{2}$; (ix) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$; (x) $\frac{5}{2}-x, -y, z-\frac{1}{2}$.

The carboxyl H atoms were located in a difference Fourier synthesis and refined isotropically [O—H = 0.96 (6)–1.06 (6) Å]. All other H atoms were placed in geometrically calculated positions and included in the refinement in a riding-model approximation, with U_{iso} equal to $1.2U_{\text{eq}}$ of the carrier atom (1.5 U_{eq} for methyl and H atoms attached to nitrogen).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1999); software used to prepare material for publication: *SHELXL*97.

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supporting information

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B. Sridhar, N. Srinivasan and R. K. Rajaram

S1. Comment

Aspartic acid is a non-essential amino acid, widely distributed in proteins, which plays a major role in the energy cycle of the human body. The crystal structures of *L*-aspartic acid (Derissen *et al.*, 1968), DL-aspartic acid (Rao, 1973; Sequeira *et al.*, 1989), DL-aspartic acid nitrate monohydrate (Asath Bahdur & Rajaram, 1995) and bis(DL-aspartic acid) sulfate (Srinivasan *et al.*, 2001) have been reported. In the present paper, the crystal structure of the product, (I), of *L*-aspartic acid reacted with nitric acid is reported.

The asymmetric unit of (I) contains one aspartic acid cation (residue 1), one neutral aspartic acid molecule (residue 2) and one nitrate anion. Superposition of residue 1 on residue 2 results in an r.m.s. deviation of the constituent atoms of 1.088 Å. Examination of the coordinates suggests that the two residues might be related by a pseudo-inversion centre. The unsymmetrical carboxyl bond distances and angles [1.217 (7)/1.297 (6) Å and 122.3 (5)/109.9 (5)°] of residue 1 clearly indicate protonation of the carboxyl group, whilst, in the case of residue 2, the equality of C—O bond distances [1.240 (7)/1.250 (6) Å] and O—C—C bond angles [117.4 (5)/115.5 (5)°] represent the deprotonated carboxylate group (Table 1).

The backbone conformation angle ψ^1 is the *cis* form for both residues [O1A—C11—C12—N1 − 7.0 (7)° and O2A—C21—C22—N21 − 1.7 (7)°]. The deviations of the amino atoms N11 and N21 from the planar carboxyl groups at C11 and C22 are 0.198 (8) and 0.044 (1) Å, respectively. This non-planarity of the amino nitrogen and carboxyl group is also found in other amino acids (Lakshminarayanan *et al.*, 1967). The side-chain conformation angle χ^1 is in a *gauche* I form [66.1 (6)°] for residue 1 and a *gauche* II form [−64.8 (6)°] for residue 2. The branched chain conformation angles, χ^{11} and χ^{21}, are in *cis* and *trans* forms [10.0 (8)/-5.5 (8) and −170.8 (5)/174.3 (5)°] for both residues. In residue 1, the C^γ atom C14 is in the *gauche* II [−58.1 (6)°] conformation with respect to C11, while, in the case of residue 2, the C^γ atom C24 is *trans* [170.9 (5)°] with respect to C21.

The average N—O and O—N—O values are 1.248 Å and 120°, respectively, clearly showing the nearly ideal trigonal symmetry of the anion, which plays a vital role in hydrogen bonding and the resulting stabilization of the structure.

The aspartic acid cation and neutral aspartic acid molecule are linked, by strong O—H···O hydrogen bonding, to form dimers. This hydrogen bond may be termed an asymmetric hydrogen bond, since the H atom is closer to one of the O atoms of the carboxyl group (Olovsson *et al.*, 2001). Atom H1B is in a *syn* orientation with respect to both donor carboxyl group and acceptor carboxylate group; the torsion angles H1B—O1B—C11—O1A and H1B—O2B^{i}—C21^{i}—O2A^{i} [symmetry code: (i) $1 + x, y, z$] are 13 (5) and 29 (3)°, respectively. This type of *syn*–syn orientation is also found in betaine betainium oxalate (Rodrigues *et al.*, 2001). The β-carboxyl group of residue 1 forms a strong O—H···O hydrogen bond with the α-carboxylate group of residue 2. In the case of residue 2, the β-carboxyl group forms a rather strong O—H···O hydrogen bond with the carbonyl O atom of the β-carboxyl group of residue 1.

The amino N atom of both residues forms N—H···O hydrogen bonds with the nitrate anion, and the α- and β-carboxyl groups. Three-centered hydrogen bonding is observed in both residues. Interestingly, in residue 1, three such three-centered bonds are observed, leading to a class IV hydrogen-bonding pattern (Jeffrey & Saenger, 1991). The frequency of such class IV hydrogen-bonding pattern is very low. A class II hydrogen-bonding pattern is observed in residue 2, having two two-centered and one three-centered hydrogen bonds. In both residues, intramolecular N—H···O hydrogen bonding is present, involving the amino nitrogen and the carbonyl oxygen of a carboxylic acid group. A straight (S1) head-to-tail sequence is observed in residue 1, connecting two amino acids separated by a unit translation (Vijayan, 1988). Each aspartic acid residue is linked by the nitrate anion through N—H···O hydrogen bonding, forming a chain running along the *a* axis: (*a*) ($x - 1, y, z$)O2···H11B—N11—H11A···O3($x - 2, y, z$) and (*b*) ($-1/2 - x, 2 - y, z - 1/2$)O1···H21A—N21—H21B···O3($1/2 - x, 2 - y, z - 1/2$).

S2. Experimental

The title compound was crystallized by slow evaporation of an aqueous solution of *L*-aspartic acid and nitric acid in a 2:1 stoichiometric ratio.

S3. Refinement

The carboxyl H atoms were located in a difference Fourier synthesis and refined isotropically [O—H = 0.96 (6)–1.06 (6) Å]. All other H atoms were placed in geometrically calculated positions and included in the refinement in a riding-model approximation, with U_{iso} equal to $1.2U_{eq}$ of the carrier atom (1.5 U_{eq} for methyl and H atoms attached to nitrogen).

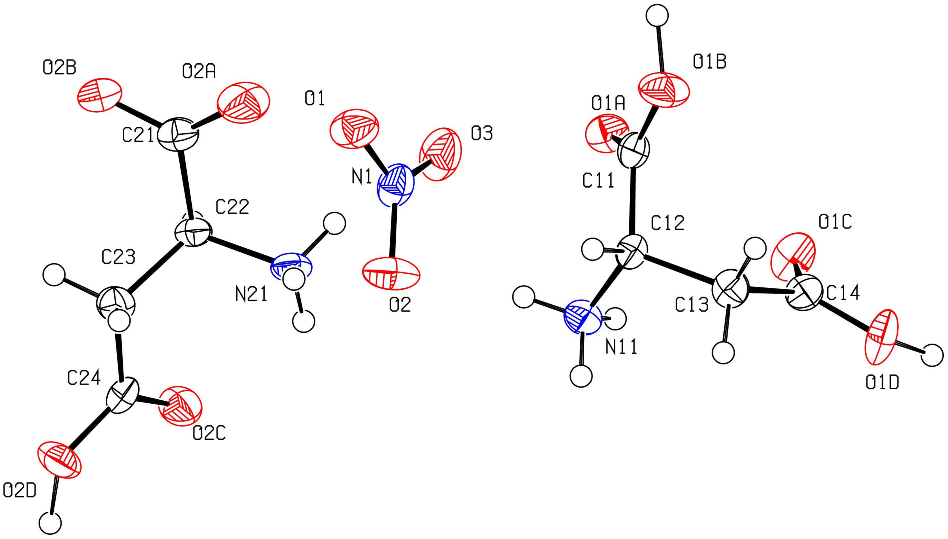

Figure 1

The molecular structure, with the atom numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976).

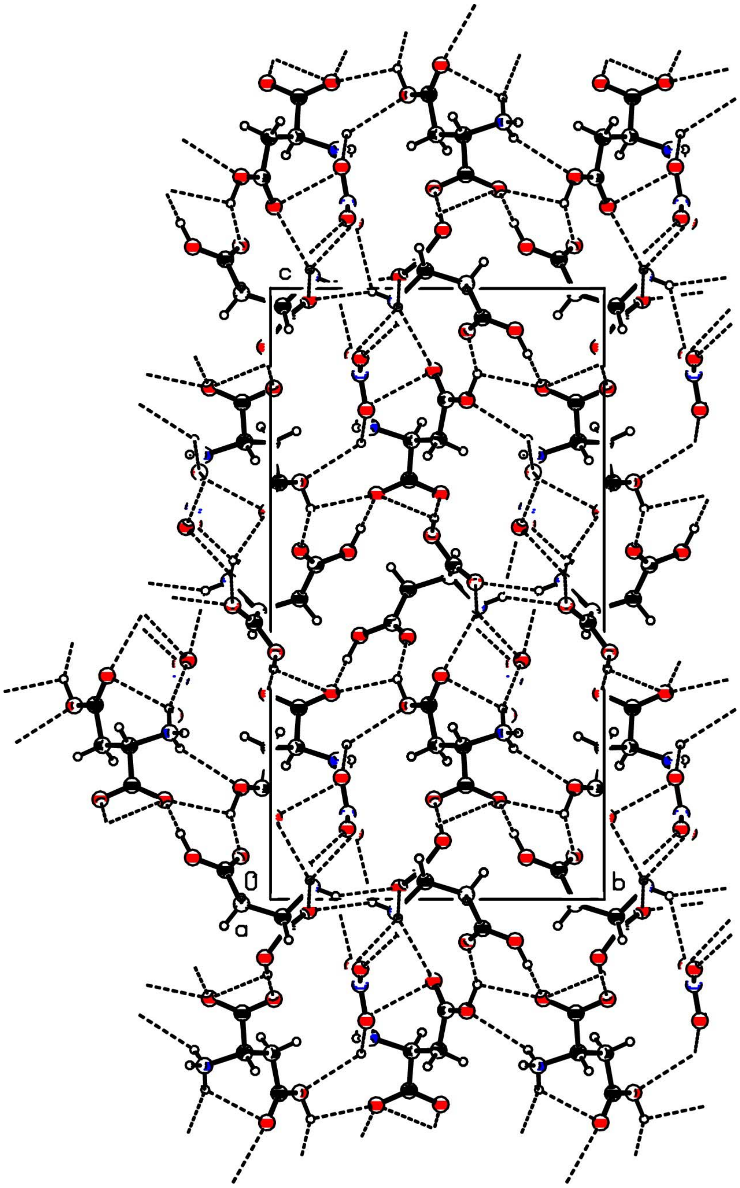

Figure 2

Packing diagram of the title molecule, viewed down the *a* axis.

Bis(*L*-aspartic acid) nitrate

Crystal data

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$M_r = 329.23$
Orthorhombic, $P2_12_12_1$
$a = 5.5840$ (7) Å
$b = 11.491$ (3) Å
$c = 21.043$ (5) Å
$V = 1350.2$ (5) Å^3
$Z = 4$
$F(000) = 688$

$D_x = 1.620$ Mg m^{-3}
$D_m = 1.58$ Mg m^{-3}
D_m measured by flotation in mixture of carbon tetrachloride and xylene
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.0$–$14.8°$
$\mu = 0.15$ mm^{-1}
$T = 293$ K

Needle, colourless	0.5 × 0.2 × 0.2 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	1578 independent reflections
Radiation source: fine-focus sealed tube	929 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\rm int} = 0.020$
ω–2θ scans	$\theta_{\rm max} = 25.0°$, $\theta_{\rm min} = 1.9°$
Absorption correction: ψ scan (North et al., 1968)	$h = 0\rightarrow6$
$T_{\rm min} = 0.932$, $T_{\rm max} = 0.968$	$k = -1\rightarrow13$
1640 measured reflections	$l = -1\rightarrow24$
	3 standard reflections every 60 min
	intensity decay: none

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.0829P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\rm max} < 0.001$
$S = 1.02$	$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
1578 reflections	$\Delta\rho_{\rm min} = -0.30$ e Å^{-3}
212 parameters	Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.011 (3)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

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	$U_{\rm iso}$*/$U_{\rm eq}$
O1A	1.2219 (7)	−0.1138 (3)	0.48094 (17)	0.0314 (10)
O1B	1.1335 (8)	0.0165 (4)	0.40417 (18)	0.0396 (11)
H1B	1.289 (15)	0.009 (8)	0.376 (3)	0.11 (3)*
C11	1.0862 (11)	−0.0487 (5)	0.4529 (2)	0.0280 (14)
C12	0.8217 (10)	−0.0363 (4)	0.4712 (2)	0.0213 (13)
H12	0.7266	−0.0545	0.4334	0.026*
N11	0.7613 (8)	−0.1242 (4)	0.5204 (2)	0.0286 (12)
H11A	0.6072	−0.1179	0.5307	0.043*
H11B	0.8509	−0.1123	0.5548	0.043*
H11C	0.7894	−0.1952	0.5052	0.043*
C13	0.7539 (11)	0.0870 (4)	0.4922 (2)	0.0273 (13)
H13A	0.7759	0.1390	0.4563	0.033*

H13B	0.5849	0.0876	0.5029	0.033*
C14	0.8914 (11)	0.1353 (5)	0.5478 (2)	0.0270 (14)
O1C	1.0679 (7)	0.0892 (4)	0.57031 (16)	0.0375 (11)
O1D	0.8000 (8)	0.2330 (3)	0.56814 (19)	0.0414 (12)
H1D	0.849 (11)	0.270 (5)	0.607 (3)	0.05 (2)*
O2A	0.5429 (9)	0.1827 (4)	0.33741 (18)	0.0469 (13)
O2B	0.4999 (8)	−0.0102 (4)	0.33645 (18)	0.0402 (11)
C21	0.5667 (10)	0.0847 (5)	0.3135 (2)	0.0283 (13)
C22	0.6907 (9)	0.0773 (5)	0.2487 (2)	0.0240 (13)
H22	0.5733	0.0462	0.2186	0.029*
N21	0.7516 (8)	0.1980 (4)	0.22727 (19)	0.0268 (11)
H21A	0.6204	0.2420	0.2281	0.040*
H21B	0.8092	0.1955	0.1879	0.040*
H21C	0.8613	0.2283	0.2531	0.040*
C23	0.9042 (11)	−0.0038 (5)	0.2478 (2)	0.0322 (14)
H23A	1.0300	0.0291	0.2740	0.039*
H23B	0.8578	−0.0776	0.2665	0.039*
C24	1.0026 (11)	−0.0258 (5)	0.1819 (2)	0.0277 (14)
O2C	0.9084 (8)	0.0106 (3)	0.13420 (17)	0.0367 (11)
O2D	1.2010 (7)	−0.0881 (4)	0.18295 (17)	0.0404 (11)
H2D	1.274 (13)	−0.120 (6)	0.140 (3)	0.07 (2)*
N1	0.2416 (10)	0.7685 (4)	0.6397 (2)	0.0316 (12)
O1	0.2544 (7)	0.7870 (3)	0.69917 (17)	0.0397 (11)
O2	0.0374 (7)	0.7696 (4)	0.61466 (19)	0.0447 (12)
O3	0.4204 (8)	0.7467 (4)	0.60902 (19)	0.0471 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.024 (2)	0.039 (2)	0.031 (2)	0.004 (2)	−0.004 (2)	−0.0002 (19)
O1B	0.039 (3)	0.042 (2)	0.038 (2)	0.002 (2)	0.014 (2)	0.015 (2)
C11	0.032 (4)	0.030 (3)	0.022 (3)	−0.007 (3)	0.002 (3)	−0.011 (3)
C12	0.027 (3)	0.019 (3)	0.018 (3)	0.003 (3)	−0.001 (2)	0.001 (2)
N11	0.022 (3)	0.031 (3)	0.032 (2)	−0.003 (3)	0.001 (3)	−0.001 (2)
C13	0.025 (3)	0.031 (3)	0.026 (3)	0.001 (4)	−0.005 (3)	−0.002 (3)
C14	0.031 (4)	0.031 (3)	0.020 (3)	−0.001 (3)	0.000 (3)	0.000 (3)
O1C	0.032 (3)	0.045 (2)	0.036 (2)	0.011 (2)	−0.014 (2)	−0.009 (2)
O1D	0.050 (3)	0.028 (2)	0.045 (2)	0.009 (2)	−0.013 (2)	−0.018 (2)
O2A	0.061 (3)	0.043 (3)	0.037 (2)	−0.007 (2)	0.019 (2)	−0.015 (2)
O2B	0.040 (3)	0.043 (2)	0.038 (2)	0.003 (2)	0.017 (2)	0.007 (2)
C21	0.025 (3)	0.037 (3)	0.023 (3)	−0.001 (3)	0.002 (3)	0.000 (3)
C22	0.019 (3)	0.029 (3)	0.024 (3)	−0.001 (3)	0.005 (3)	0.001 (3)
N21	0.019 (3)	0.031 (3)	0.031 (2)	−0.005 (3)	0.007 (2)	−0.004 (2)
C23	0.032 (3)	0.037 (3)	0.028 (3)	0.008 (3)	0.005 (3)	0.005 (3)
C24	0.033 (4)	0.025 (3)	0.026 (3)	−0.003 (3)	0.004 (3)	0.008 (3)
O2C	0.041 (3)	0.044 (2)	0.024 (2)	0.010 (2)	0.003 (2)	0.001 (2)
O2D	0.041 (3)	0.051 (3)	0.029 (2)	0.025 (2)	0.009 (2)	0.004 (2)
N1	0.028 (3)	0.030 (3)	0.037 (3)	−0.006 (3)	0.006 (3)	−0.007 (2)

O1	0.028 (2)	0.055 (3)	0.036 (2)	−0.001 (2)	0.000 (2)	−0.007 (2)
O2	0.028 (3)	0.061 (3)	0.046 (2)	−0.003 (2)	−0.013 (2)	0.012 (2)
O3	0.040 (3)	0.047 (3)	0.054 (3)	−0.002 (2)	0.011 (3)	−0.019 (2)

Geometric parameters (Å, º)

O1A—C11	1.217 (7)	O2B—C21	1.250 (6)
O1B—C11	1.297 (6)	C21—C22	1.531 (7)
O1B—H1B	1.05 (8)	C22—N21	1.498 (7)
C11—C12	1.533 (8)	C22—C23	1.513 (7)
C12—N11	1.485 (6)	C22—H22	0.9800
C12—C13	1.532 (7)	N21—H21A	0.8900
C12—H12	0.98	N21—H21B	0.8900
N11—H11A	0.89	N21—H21C	0.8900
N11—H11B	0.8900	C23—C24	1.512 (7)
N11—H11C	0.8900	C23—H23A	0.9700
C13—C14	1.505 (7)	C23—H23B	0.9700
C13—H13A	0.9700	C24—O2C	1.208 (6)
C13—H13B	0.9700	C24—O2D	1.319 (7)
C14—O1C	1.215 (6)	O2D—H2D	1.06 (6)
C14—O1D	1.306 (6)	N1—O3	1.215 (6)
O1D—H1D	0.96 (6)	N1—O2	1.256 (6)
O2A—C21	1.240 (7)	N1—O1	1.272 (5)
C11—O1B—H1B	124 (5)	O2A—C21—C22	117.4 (5)
O1A—C11—O1B	127.7 (6)	O2B—C21—C22	115.5 (5)
O1A—C11—C12	122.3 (5)	N21—C22—C23	112.8 (4)
O1B—C11—C12	109.9 (5)	N21—C22—C21	108.6 (4)
N11—C12—C13	111.9 (4)	C23—C22—C21	113.7 (4)
N11—C12—C11	109.3 (4)	N21—C22—H22	107.1
C13—C12—C11	113.4 (5)	C23—C22—H22	107.1
N11—C12—H12	107.3	C21—C22—H22	107.1
C13—C12—H12	107.3	C22—N21—H21A	109.5
C11—C12—H12	107.3	C22—N21—H21B	109.5
C12—N11—H11A	109.5	H21A—N21—H21B	109.5
C12—N11—H11B	109.5	C22—N21—H21C	109.5
H11A—N11—H11B	109.5	H21A—N21—H21C	109.5
C12—N11—H11C	109.5	H21B—N21—H21C	109.5
H11A—N11—H11C	109.5	C24—C23—C22	113.7 (5)
H11B—N11—H11C	109.5	C24—C23—H23A	108.8
C14—C13—C12	116.0 (5)	C22—C23—H23A	108.8
C14—C13—H13A	108.3	C24—C23—H23B	108.8
C12—C13—H13A	108.3	C22—C23—H23B	108.8
C14—C13—H13B	108.3	H23A—C23—H23B	107.7
C12—C13—H13B	108.3	O2C—C24—O2D	124.6 (5)
H13A—C13—H13B	107.4	O2C—C24—C23	123.1 (5)
O1C—C14—O1D	124.3 (5)	O2D—C24—C23	112.4 (5)
O1C—C14—C13	123.8 (5)	C24—O2D—H2D	120 (4)

O1D—C14—C13	111.8 (5)	O3—N1—O2	121.7 (5)
C14—O1D—H1D	123 (4)	O3—N1—O1	120.8 (6)
O2A—C21—O2B	127.1 (5)	O2—N1—O1	117.5 (5)

O1A—C11—C12—N11	−7.0 (7)	O2A—C21—C22—N21	−1.7 (7)
O1B—C11—C12—N11	171.4 (4)	O2B—C21—C22—N21	178.2 (5)
O1A—C11—C12—C13	118.6 (6)	O2A—C21—C22—C23	124.8 (6)
O1B—C11—C12—C13	−63.0 (5)	O2B—C21—C22—C23	−55.3 (7)
N11—C12—C13—C14	66.1 (6)	N21—C22—C23—C24	−64.8 (6)
C11—C12—C13—C14	−58.1 (6)	C21—C22—C23—C24	170.9 (5)
C12—C13—C14—O1C	10.0 (8)	C22—C23—C24—O2C	−5.5 (8)
C12—C13—C14—O1D	−170.8 (5)	C22—C23—C24—O2D	174.3 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O2*B*^{i}	1.05 (8)	1.47 (8)	2.512 (5)	175 (8)
N11—H11*A*···O1*A*^{ii}	0.89	2.39	3.126 (6)	140
N11—H11*A*···O3^{iii}	0.89	2.50	3.050 (6)	121
N11—H11*B*···O2^{iv}	0.89	2.12	2.793 (6)	131
N11—H11*B*···O1*C*	0.89	2.63	3.170 (6)	120
N11—H11*C*···O1*A*^{v}	0.89	2.25	3.018 (5)	145
N11—H11*C*···O3^{vi}	0.89	2.58	3.192 (6)	127
O1*D*—H1*D*···O2*A*^{vi}	0.96 (6)	1.68 (6)	2.594 (5)	156 (6)
N21—H21*A*···O1^{vii}	0.89	2.20	2.892 (6)	134
N21—H21*B*···O3^{viii}	0.89	2.34	3.155 (6)	152
N21—H21*B*···O2*C*	0.89	2.47	3.040 (5)	122
N21—H21*C*···O2*D*^{ix}	0.89	2.53	3.111 (6)	124
O2*D*—H2*D*···O1*C*^{x}	1.06 (6)	1.74 (6)	2.699 (5)	148 (5)

Symmetry codes: (i) $x+1, y, z$; (ii) $x-1, y, z$; (iii) $x, y-1, z$; (iv) $x+1, y-1, z$; (v) $x-1/2, -y-1/2, -z+1$; (vi) $x+1/2, -y+1/2, -z+1$; (vii) $-x+1/2, -y+1, z-1/2$; (viii) $-x+3/2, -y+1, z-1/2$; (ix) $-x+2, y+1/2, -z+1/2$; (x) $-x+5/2, -y, z-1/2$.