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Bis(DL-aspartic acid) sulfate

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In the title compound, $2C_4H_8NO_4^+ \cdot SO_4^{2-}$, the sulfate anion connects the aspartic acid cations by strong hydrogen bonds. Both independent cations have a *gauche* II form, and the C^{γ} is *trans* to the C′ atom. An intramolecular N—H···O hydrogen bond is observed in one of the cations.

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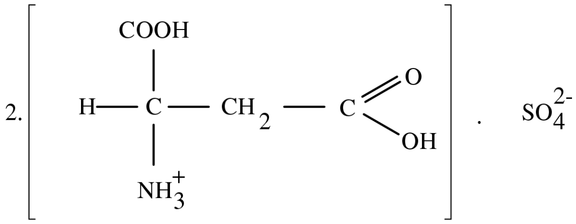
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.107
Data-to-parameter ratio = 11.8

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

Comment

Aspartic acid is an important compound among naturally occurring α-amino acids. The crystal structures of L-aspartic acid (Derissen *et al.*, 1968), DL-aspartic acid (Rao, 1973), DL-aspartic acid hydrochloride (Dawson, 1977) and DL-aspartic acid nitrate monohydrate (Asath Bahadur & Rajaram, 1994) have been reported earlier. In the present study, DL-aspartic acid reacted with sulfuric acid, (I), was investigated.



(I)

In (I), there are two independent aspartic acid cations, *A* and *B*, which have the amino N11 and N21 atoms, respectively (Fig. 1). Their geometries are similar and agree well with those of DL-aspartic acid hydrochloride (Dawson, 1977). The molecule is made up of three four-atom planes consisting of C′, C^{α}, C^{β}, C^{γ} (plane 1), α-carboxyl group (plane 2) and the β-carboxyl group (plane 3). The mean deviations of the carbon skeleton from plane 1 are 0.167 and 0.030 Å for *A* and *B*, respectively. The C^{γ} is *trans* to the C′ atom for both the molecules. The conformation angles ψ^1 are 15.4 (3) and −8.0 (3)° for *A* and *B*, respectively (Table 1). This tendency of twisting of the C—N bond from the amino carboxyl plane is found in various amino acids (Lakshiminarayanan *et al.*, 1967). The branched chain conformation angle χ^1 is in a *gauche* II form for both molecules; the N11—C12—C13—C14 and N21—C22—C23—C24 torsion angles are −85.3 (2) and −64.7 (2)°, respectively.

The sulfate anion plays a vital role in forming hydrogen bonds with both the cations and stabilizing the structure (Fig. 2). The sulfate anion, as acceptor, links the O1*B*, O1*D*, N11, O2*B*, O2*D* and N21 atoms. The O^{γ} atom of each molecule links to the amino N atom of the other molecule (Table 2). A bifurcated hydrogen bond is observed in the case of N21

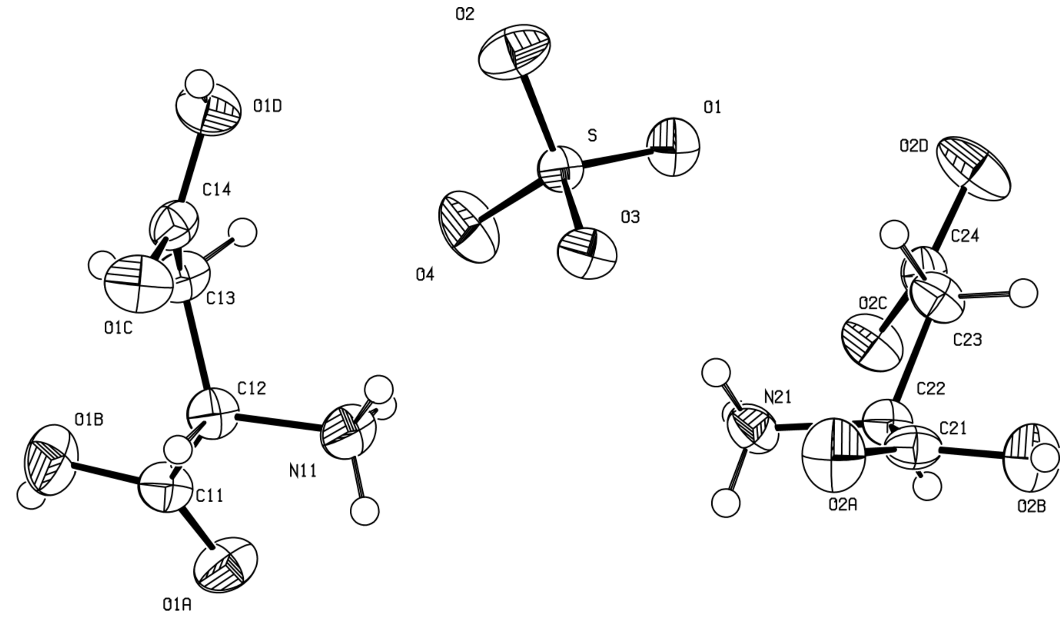


Figure 1
The molecular structures of the two independent cations and the anion of (I) showing the atomic numbering scheme and 50% probability displacement ellipsoids (Johnson, 1976).

with O1C^{v} and O2*C*, the latter being an intramolecular hydrogen bond. However, an intramolecular hydrogen bond is not favoured in molecule A, since the χ^{22} for *A* and *B* are −33.4 (3) and 3.9 (3)°, respectively. A chelated three-centre hydrogen bond is observed in the case of the amino N21 atom of molecule *B* with the sulfate O1 and O2 atoms (Jeffrey & Saenger, 1991). An infinite chain of hydrogen bonds runs along the *b* axis, linking the sulfate anion and the N21 atom.

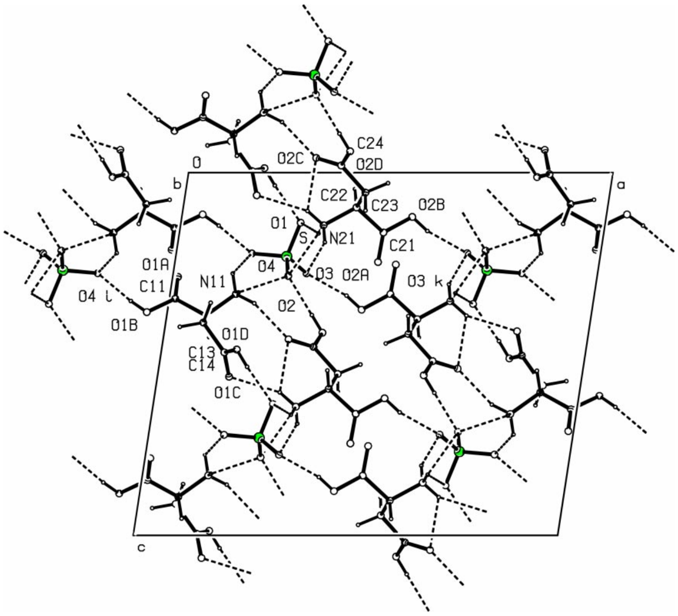


Figure 2
Packing diagram of the title compound viewed down the *b* axis

Experimental

The title compound was crystallized from an aqueous solution of DL-aspartic acid and sulfuric acid in a stoichiometric ratio of 2:1.

Crystal data

$2C_4H_8NO_4^+ \cdot SO_4^{2-}$
$M_r = 364.29$
Monoclinic, $P2_1/c$
$a = 16.774$ (4) Å
$b = 5.9697$ (9) Å
$c = 14.5276$ (18) Å
$\beta = 98.66$ (2)°
$V = 1438.1$ (4) Å^3
$Z = 4$
$D_x = 1.683$ Mg m^{-3}
$D_m = 1.674$ Mg m^{-3}
D_m measured by flotation in a mixture of bromoform and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8.9$–14.5°
$\mu = 0.30$ mm^{-1}
$T = 293$ (2) K
Needle, colorless
0.5 × 0.4 × 0.2 mm

Data collection

Enraf–Nonius sealed-tube diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.868$, $T_{max} = 0.943$
2616 measured reflections
2502 independent reflections
2113 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$
$h = -19 \rightarrow 19$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 17$
25 standard reflections every 3 reflections frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.107$
$S = 0.81$
2502 reflections
212 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 1.6327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

O1*A*—C11	1.200 (3)	O2*A*—C21	1.194 (3)
O1*B*—C11	1.311 (3)	O2*B*—C21	1.314 (3)
C14—O1*C*	1.212 (3)	C24—O2*C*	1.212 (3)
C14—O1*D*	1.325 (3)	C24—O2*D*	1.307 (3)
O1*A*—C11—C12—N11	15.4 (3)	O2*A*—C21—C22—N21	−8.0 (3)
N11—C12—C13—C14	−85.3 (2)	N21—C22—C23—C24	−64.7 (2)
C11—C12—C13—C14	152.00 (19)	C21—C22—C23—C24	175.16 (18)
C12—C13—C14—O1*C*	−33.4 (3)	C22—C23—C24—O2*C*	3.9 (3)
C12—C13—C14—O1*D*	147.60 (19)	C22—C23—C24—O2*D*	−176.05 (19)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*B*—H1*B*···O4$^{\rm i}$	0.82	1.79	2.605 (2)	174
N11—H11*A*···O2*C*$^{\rm ii}$	0.89	1.97	2.760 (2)	147
N11—H11*B*···O4$^{\rm iii}$	0.89	2.20	2.837 (3)	128
N11—H11*C*···O2	0.89	2.36	2.972 (3)	126
O1D—H1*D*···O1$^{\rm ii}$	0.82	1.85	2.673 (2)	176
O2B—H2*B*···O3$^{\rm iv}$	0.82	1.83	2.631 (2)	166
N21—H21*A*···O1*C*$^{\rm v}$	0.89	2.14	2.898 (3)	143
N21—H21*A*···O2*C*	0.89	2.29	2.899 (2)	126
N21—H21*B*···O3$^{\rm iii}$	0.89	1.96	2.785 (3)	154
N21—H21*C*···O1	0.89	2.12	2.906 (2)	147
N21—H21*C*···O2	0.89	2.28	3.010 (3)	140
O2D—H2*D*···O2$^{\rm v}$	0.82	1.85	2.669 (2)	175

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

All H atoms were fixed by geometric constraints using *HFIX* and were allowed to ride on the carrier atom.

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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