Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

DL-Aspartic acid

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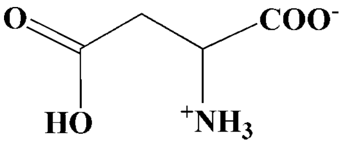
Received 27 August 2007; accepted 3 September 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.103; data-to-parameter ratio = 10.0.

A form of DL-aspartic acid, $C_4H_7NO_4$ is presented, which crystallizes in the same space group as the previously reported form but with a slightly different a axis. Its structure exhibits a three-dimensional supramolecular network of N—H···O and O—H···O bonds, which stabilizes the crystal packing.

Related literature

For related literature, see: Barfod *et al.* (1999); Casellato *et al.* (1991); Ciunik (1987); Flaig *et al.* (1998); Rao (1973); Rao *et al.* (1968); Sequeira *et al.* (1989).



Experimental

Crystal data

$C_4H_7NO_4$
M_r = 133.11
Monoclinic, $C2/c$
a = 17.5813 (2) Å
b = 7.4369 (5) Å
c = 9.1807 (3) Å
β = 116.436 (3)°
V = 1074.86 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 293 (2) K
0.18 × 0.14 × 0.06 mm

Data collection

Siemems SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.974, T_{max} = 0.991
4179 measured reflections
1105 independent reflections
986 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.07
1105 reflections
111 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O3^{i}	0.95 (3)	1.61 (3)	2.5574 (16)	176 (3)
N1—H7···O4^{ii}	0.95 (2)	1.93 (2)	2.8782 (19)	171.8 (18)
N1—H6···O4^{iii}	0.93 (2)	1.91 (2)	2.8381 (18)	177.4 (19)
N1—H5···O1^{iv}	0.90 (2)	2.07 (2)	2.8992 (18)	152.5 (19)
N1—H5···O1	0.90 (2)	2.26 (2)	2.8572 (18)	124.0 (17)

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2096).

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doi:10.1107/S1600536807043097

supplementary materials

Acta Cryst. (2007). E**63**, o4003   [**doi:10.1107/S1600536807043097**]

DL-Aspartic acid

G.-M. Wang, Z.-X. Li, C.-S. Duan and **H. Li**

Comment

Interest in D,*L*-aspartic acid has spanned several decades, from the first structural determination by Rao (1973), due to its acting as important ligand in the preparation of transition metal complexes (Ciunik, 1987; Casellato *et al.*, 1991; Barfod *et al.*, 1999). In exploring the possibility of introducing D,*L*-aspartic acid into a phosphite system, we unexpectedly obtained the title compound. To the best of our knowledge, there are only four reports on the structure of D,*L*-aspartic acid (Rao *et al.*, 1968; Rao, 1973; Sequeira *et al.*, 1989; Flaig *et al.*, 1998). The present structure of D,*L*-aspartic acid differs slightly from these previously reported ones, since it presents the same space group and a similar molecular disposition, but with an *a* parameter some 7–8% shorter.

The molecules (Fig. 1) are interconnected with each other by hydrogen bonds to form a 3-D supramolecular network (Table 1 and Fig. 2).

Experimental

The title compound was obtained by hydrothermal methods. A mixture of $Zn(CH_3COO)_2·2H_2O$ (0.16 g), H_3PO_3 (0.19 g), D,*L*-aspartic acid (0.25 g) and H_2O (8 ml), was sealed in a 25 ml Teflon-lined steel autoclave and heated under autogenous pressure at 363 K for 3 days. Then, the filtrate was kept at room temperature and colorless prism-like crystals were obtained after three weeks.

Refinement

All the H atoms were located from difference-density maps in a difference Fourier map and their positions and isotropic displacement parameters were refined.

Figures

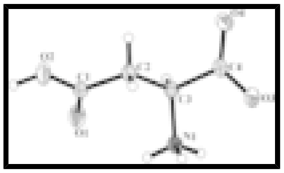

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

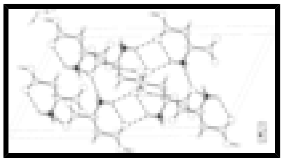

Fig. 2. Packing diagram of the title compound.

D,*L*-aspartic acid

Crystal data

$C_4H_7NO_4$
$M_r = 133.11$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 17.5813$ (2) Å
$b = 7.4369$ (5) Å
$c = 9.1807$ (3) Å
$\beta = 116.436$ (3)°
$V = 1074.86$ (9) Å^3
$Z = 8$

$F_{000} = 560$
$D_x = 1.645$ Mg m^{-3}
Mo *K*α radiation
$\lambda = 0.71073$ Å
Cell parameters from 4179 reflections
$\theta = 2.6$–26.5°
$\mu = 0.15$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.18 × 0.14 × 0.06 mm

Data collection

Siemems SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 293(2)$ K
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.974$, $T_{max} = 0.991$
4179 measured reflections

1105 independent reflections
986 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.5°$
$\theta_{min} = 2.6°$
$h = -22\rightarrow22$
$k = -9\rightarrow9$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.103$
$S = 1.07$
1105 reflections
111 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.5964P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (2)

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 $F^2 > 2\text{sigma}(F^2)$ 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_{iso}*/U_{eq}
C1	0.00684 (9)	0.2497 (2)	0.49490 (19)	0.0227 (4)
C2	0.09882 (9)	0.2473 (2)	0.52666 (19)	0.0238 (4)
H2	0.1006 (12)	0.204 (3)	0.431 (3)	0.035 (5)*
H3	0.1188 (13)	0.374 (3)	0.544 (3)	0.042 (6)*
C3	0.15383 (9)	0.1315 (2)	0.67210 (19)	0.0199 (4)
H4	0.1312 (10)	0.014 (2)	0.6575 (18)	0.016 (4)*
C4	0.24516 (9)	0.1148 (2)	0.69224 (19)	0.0205 (4)
O1	−0.01739 (7)	0.17330 (17)	0.58347 (15)	0.0342 (4)
O2	−0.04289 (8)	0.33822 (18)	0.36471 (15)	0.0348 (4)
H1	−0.1002 (18)	0.338 (4)	0.349 (3)	0.074 (8)*
O3	0.30295 (7)	0.17660 (17)	0.82024 (14)	0.0319 (4)
O4	0.25457 (7)	0.03708 (15)	0.58062 (13)	0.0270 (3)
N1	0.15498 (9)	0.2014 (2)	0.82518 (17)	0.0232 (4)
H7	0.1800 (14)	0.318 (3)	0.855 (3)	0.040 (6)*
H6	0.1879 (14)	0.126 (3)	0.911 (3)	0.041 (6)*
H5	0.1023 (14)	0.203 (3)	0.818 (3)	0.040 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0161 (8)	0.0309 (9)	0.0199 (7)	0.0015 (6)	0.0069 (6)	−0.0019 (6)
C2	0.0165 (8)	0.0321 (9)	0.0227 (8)	0.0018 (6)	0.0085 (7)	0.0036 (7)
C3	0.0153 (7)	0.0219 (8)	0.0218 (8)	−0.0023 (6)	0.0075 (6)	−0.0020 (6)
C4	0.0158 (7)	0.0196 (8)	0.0253 (8)	0.0012 (6)	0.0084 (6)	0.0024 (6)
O1	0.0183 (6)	0.0553 (8)	0.0298 (7)	0.0011 (5)	0.0116 (5)	0.0096 (6)
O2	0.0165 (6)	0.0565 (8)	0.0287 (7)	0.0073 (5)	0.0077 (5)	0.0144 (6)
O3	0.0150 (6)	0.0461 (8)	0.0311 (7)	−0.0033 (5)	0.0071 (5)	−0.0111 (5)
O4	0.0217 (6)	0.0301 (6)	0.0302 (6)	0.0014 (5)	0.0125 (5)	−0.0050 (5)
N1	0.0155 (7)	0.0322 (8)	0.0219 (7)	−0.0006 (6)	0.0083 (6)	0.0003 (6)

Geometric parameters (Å, °)

C1—O1	1.2140 (19)	C3—H4	0.945 (17)

C1—O2	1.3023 (19)	C4—O3	1.2494 (19)
C1—C2	1.510 (2)	C4—O4	1.2501 (18)
C2—C3	1.520 (2)	O2—H1	0.95 (3)
C2—H2	0.95 (2)	N1—H7	0.95 (2)
C2—H3	0.99 (2)	N1—H6	0.93 (2)
C3—N1	1.490 (2)	N1—H5	0.90 (2)
C3—C4	1.537 (2)		

O1—C1—O2	123.98 (14)	C2—C3—H4	109.7 (10)
O1—C1—C2	122.01 (14)	C4—C3—H4	107.0 (10)
O2—C1—C2	114.01 (13)	O3—C4—O4	126.31 (14)
C1—C2—C3	112.60 (13)	O3—C4—C3	116.86 (13)
C1—C2—H2	107.4 (12)	O4—C4—C3	116.81 (13)
C3—C2—H2	110.4 (12)	C1—O2—H1	112.0 (16)
C1—C2—H3	107.0 (12)	C3—N1—H7	113.5 (13)
C3—C2—H3	110.8 (12)	C3—N1—H6	109.4 (13)
H2—C2—H3	108.5 (17)	H7—N1—H6	105.5 (18)
N1—C3—C2	111.55 (13)	C3—N1—H5	110.9 (14)
N1—C3—C4	109.87 (12)	H7—N1—H5	109.6 (18)
C2—C3—C4	111.94 (13)	H6—N1—H5	107.6 (18)
N1—C3—H4	106.5 (9)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O3^{i}	0.95 (3)	1.61 (3)	2.5574 (16)	176 (3)
N1—H7···O4^{ii}	0.95 (2)	1.93 (2)	2.8782 (19)	171.8 (18)
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Symmetry codes: (i) *x*−1/2, −*y*+1/2, *z*−1/2; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) *x*, −*y*, *z*+1/2; (iv) −*x*, *y*, −*z*+3/2.

Fig. 1

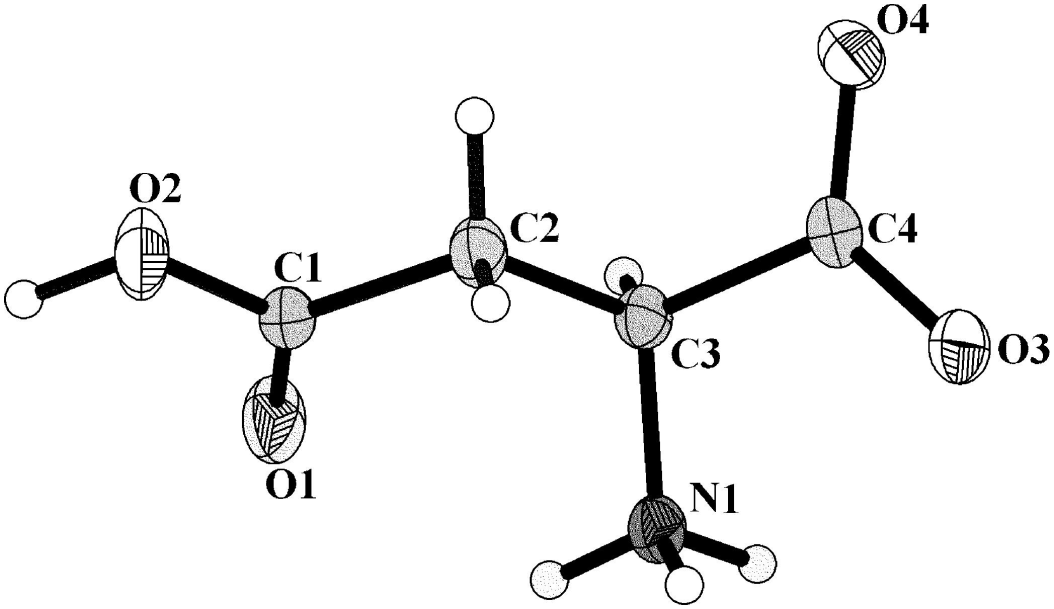

Fig. 2