

***N*-(Carboxymethyl)aspartic acid**

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Key indicators

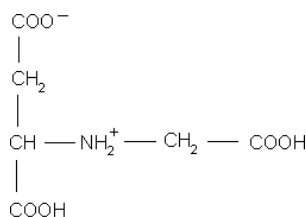
Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 R factor = 0.033
 wR factor = 0.082
 Data-to-parameter ratio = 10.0

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

The title molecule, $\text{C}_6\text{H}_9\text{NO}_6$, exists in a zwitterionic form. The transfer of the H atom to the N atom involves two carboxyl groups and, as a result, their H atoms are disordered. The crystal structure is stabilized by $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Comment

The structure of the title compound, (I), has been analysed as part of a general study of the chemistry of aspartic acid derivatives as potential ligands in coordination compounds (Pavelčík & Majer, 1978). The title compound is a potential tetradentate chelating agent that is isomeric with and chemically quite similar to NTA (nitrilotriacetic acid), but the compound is chiral as in EDDS (*N,N*-ethylenediamine-disuccinic acid) (Scarborough & Voet, 1976).



(I)

The molecular structure and atom-numbering scheme are shown in Fig. 1. In the crystal, the title molecule exists in a zwitterionic form with a carboxyl H atom transferred to the N atom. In 50% of the population, the H atom is transferred from O2, whereas in the remaining population, it is transferred from O4. This is evident from the C—O and C=O bond distances observed for the carboxyl groups (Table 1). In the solid state, intermolecular $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules to form a three-dimensional network (Table 2).

Experimental

The title compound was prepared by dissolving maleic anhydride and glycine in an aqueous solution of NaOH. The solution was refluxed for 48 h. The pH was adjusted to 7 with HCl. The resulting solution was filtered off and the pH was adjusted with HCl to 2. After several days, colourless crystals were obtained.

Crystal data

$\text{C}_6\text{H}_9\text{NO}_6$
 $M_r = 191.14$
 Monoclinic, $P2_1/n$
 $a = 5.3430 (5) \text{ \AA}$
 $b = 16.033 (2) \text{ \AA}$
 $c = 9.0895 (12) \text{ \AA}$
 $\beta = 95.384 (12)^\circ$
 $V = 775.2 (2) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.638 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 36 reflections
 $\theta = 16.9\text{--}25.0^\circ$
 $\mu = 0.15 \text{ mm}^{-1}$
 $T = 120 (2) \text{ K}$
 Prism, colourless
 $0.60 \times 0.40 \times 0.30 \text{ mm}$

Data collection

Kuma KM-4 four-circle κ -axis diffractometer with Oxford Cryosystems Cryostream cooler (Cosier & Glazer, 1986)

ω scans

Absorption correction: none
2969 measured reflections
1368 independent reflections
1179 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$
 $\theta_{\text{max}} = 25.1^\circ$
 $h = -6 \rightarrow 0$
 $k = -19 \rightarrow 19$
 $l = -10 \rightarrow 10$
3 standard reflections every 200 reflections
intensity decay: <2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.082$
 $S = 1.09$

1368 reflections
137 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.2243P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{\AA}^{-3}$

Table 1

Selected interatomic distances (Å).

O1—C2	1.2268 (18)	O5—C7	1.2097 (18)
O2—C2	1.2750 (18)	O6—C7	1.3115 (19)
O3—C4	1.2387 (18)	N5—C6	1.4797 (19)
O4—C4	1.2813 (18)	N5—C1	1.5055 (18)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2—H2 \cdots O2 ⁱ	0.91 (5)	1.52 (6)	2.429 (2)	174 (6)
O4—H4 \cdots O4 ⁱⁱ	0.83 (4)	1.60 (4)	2.433 (2)	177 (4)
O6—H6 \cdots O3 ⁱⁱⁱ	0.94 (3)	1.58 (2)	2.517 (2)	170 (2)
N5—H5A \cdots O4	0.91	2.09	2.751 (2)	128
N5—H5A \cdots O5 ^{iv}	0.91	2.32	3.040 (2)	136
N5—H5B \cdots O6 ^v	0.91	2.47	3.302 (2)	152
C3—H3B \cdots O2 ^{vi}	0.95	2.43	3.249 (2)	144
C6—H6A \cdots O1 ^{vii}	0.95	2.31	3.227 (2)	162

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

Initially, all the H atoms were located from a difference Fourier map and the NH group was found to be protonated. The C—O and C=O distances in the carboxyl groups showed that the H atom might be transferred from two carboxyl groups (C2/O1/O2/H2 and C4/O3/O4/H4), indicating disorder of H2 and H4. In the difference map, H2 and H4 were found to lie on centres of inversion such that two inversion related molecules share H2 and H4. However, refinement using this model resulted in a very long O—H distance of 1.21 Å. The U_{iso} values of both H2 and H4 were found to be relatively high at 0.07 \AA^2 . Hence, H2 and H4 were placed at calculated positions and

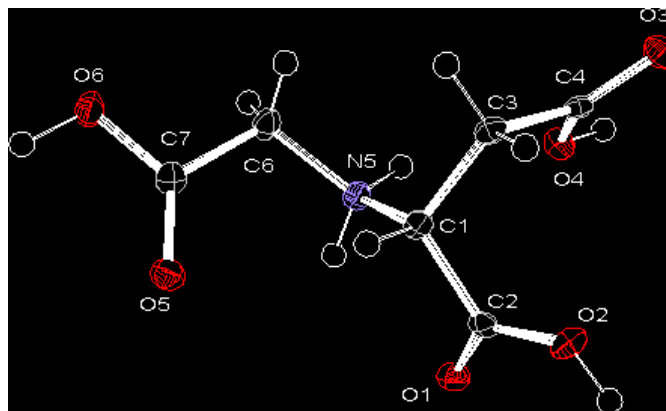


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms attached to O2 and O4 are disordered with half-occupancy.

their occupancies were initially refined and finally fixed at 0.5. At this stage, no peaks were observed at the centres of inversion. Even if the positional constraints were released, the H2 and H4 positions did not move to the inversion centres. Now the U_{iso} values of H2 and H4 showed values comparable to other H atoms. Another refinement model with three atoms (N5, O2 and O4) sharing four H atoms (H2, H4, H5A and H5B) was investigated. The refinement using this model resulted in nearly full occupancies for H5A and H5B and partial occupancies for H2 and H4, as in the previous model. Hence the earlier model was retained. During the final stages of the refinement, the H atoms attached to C and N atoms were fixed geometrically and allowed to ride on the parent atoms.

Data collection: *KM-4 CCD System Software* (Kuma, 1998); cell refinement: *KM-4 CCD System Software*; data reduction: *KM-4 CCD System Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson & Burnett, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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