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N-Acryloylaspartic acid

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.157
Data-to-parameter ratio = 14.6

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

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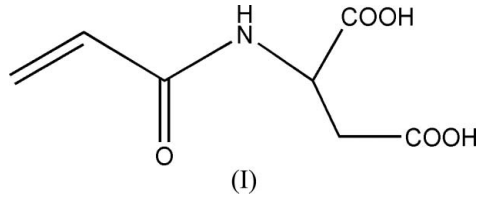
The title compound, $C_7H_9NO_5$, was prepared by the nucleophilic substitution reaction of acryloyl chloride with aspartic acid. The asymmetric unit contains two molecules. In the crystal structure, intermolecular N—H···O and O—H···O hydrogen bonds link the molecules into a three-dimensional network, which may be effective in stabilizing the crystal structure.

Comment

The title compound, (I), is an important intermediate and also a free radical addition monomer for the syntheses of radiation-sensitive (Heilmann & Palensky, 1981), hydropholic (Heilmann & Rasmussen, 1984) and pressure-sensitive (Heilmann, 1979) polymers. The crystal structure determination of (I) has been carried out in order to elucidate the molecular conformation. We report here the synthesis and the crystal structure of (I).



(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The asymmetric unit contains two molecules. In each molecule, parts *A* (O1/N1/C1–C4), *B* (O2/O3/C4/C5), *C* (O4/O5/C4/C6/C7) and *D* (N2/O6/C8–C11), *E* (O7/O8/C11/C12) and *F* (O9/O10/C11/C13/C14) are each nearly planar, with maximum r.m.s. deviations of 0.0407 (3) (for C4), 0.0111 (4) (for C5), 0.0687 (4) (for C6), 0.0974 (4) (for C9), 0.0114 (3) (for C12) and 0.0209 (4) Å (for C13), respectively. The dihedral angles between the planes are: *A*/*B* = 37.38 (3)°, *A*/*C* = 68.70 (4)°, *B*/*C* = 87.49 (3)°, *D*/*E* = 38.24 (3)°, *D*/*F* = 65.11 (4)° and *E*/*F* = 79.90 (4)°.

As can be seen from the packing digram (Fig. 2), intermolecular N—H···O and O—H···O hydrogen bonds (Table 1) link molecules into a three-dimensional network, which may be effective in stabilizing the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

For the preparation of the title compound, acryloyl chloride (10 ml, 0.22 mol) containing diphenylpicrylhydrazyl polymerization inhibitor

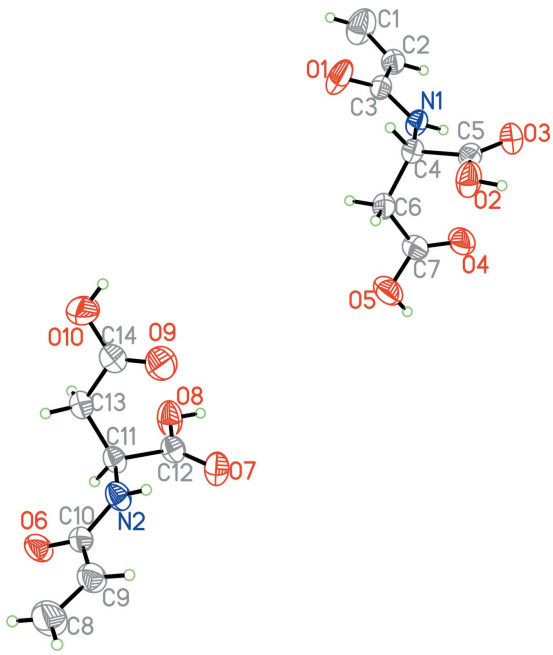


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

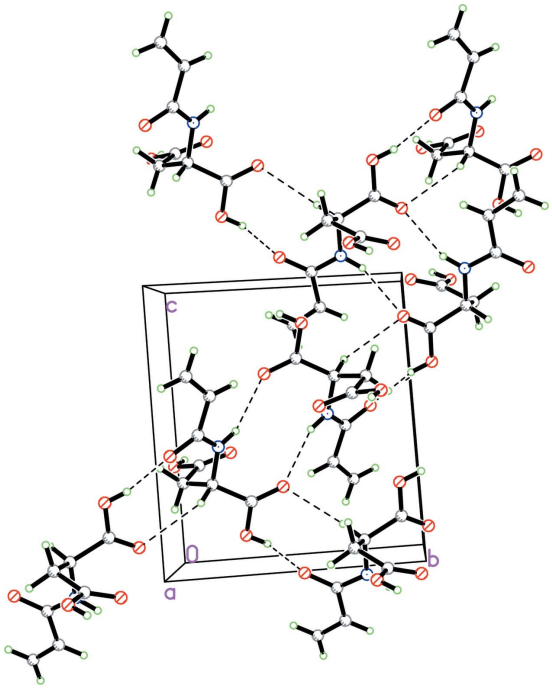


Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(0.01%) and aqueous sodium hydroxide solution (0.8 g, 0.02 mol, in 10 ml water) were simultaneously added dropwise over a 30 min period to a well stirred aqueous solution of DL-aspartic acid (2.7 g, 0.02 mol, in 60 ml water) and sodium hydroxide (1.6 g, 0.04 mol, in 20 ml water). The solution was stirred for a further 1 h at 273 K in an ice–water bath, and the pH of the reaction mixture was maintained at 2 by the addition of HCl (6 *M*) and extracted with ethyl acetate. The ethyl acetate solution was dried with anhydrous magnesium sulfate and the resulting solid was crystallized from ethyl acetate (yield 1.35 g, 36.1%; m.p. 430.5 K).

Crystal data

$C_7H_9NO_5$
M_r = 187.15
Triclinic, $P\overline{1}$
a = 8.1560 (16) Å
b = 10.079 (2) Å
c = 11.084 (2) Å
α = 90.16 (3)°
β = 94.17 (3)°
γ = 104.99 (3)°
V = 877.6 (3) Å^3
Z = 4
D_x = 1.424 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 294 (2) K
Plate, colorless
0.40 × 0.30 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.953, T_{max} = 0.988
3434 measured reflections
3433 independent reflections
2448 reflections with $I > 2\sigma(I)$
R_{int} = 0.008
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.157
S = 1.00
3433 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2 + 0.2474P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2C···O6^{i}	0.82	1.85	2.655 (3)	166
O5—H5A···O4^{ii}	0.82	1.86	2.681 (3)	177
N1—H1A···O3^{iii}	0.86	2.24	3.053 (3)	159
O8—H8D···O1^{iv}	0.82	1.79	2.590 (3)	164
O10—H10A···O6^{v}	0.82	1.93	2.719 (3)	162
N2—H2B···O7^{vi}	0.86	2.28	3.102 (3)	159

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

H atoms were positioned geometrically, with O—H = 0.82 Å, N—H = 0.86 Å and C—H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,N,O), where x = 1.5 for OH, and x = 1.2 for all other H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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