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N-Acryloylglycylglycine monohydrate

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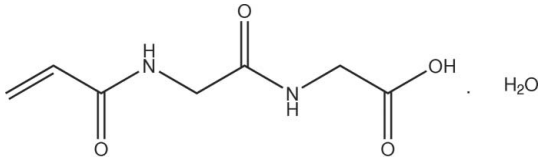
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.053; wR factor = 0.163; data-to-parameter ratio = 14.8.

The title compound [systematic name: 2-(2-acrylamidoacetamido)acetic acid monohydrate], $C_7H_{10}N_2O_4\cdot H_2O$, was prepared by the nucleophilic substitution reaction of acryloyl chloride with glycylglycine. Excluding H atoms, the main chain of the molecule is nearly planar. In the crystal structure, O—H···O, N—H···O and C—H···O hydrogen bonds link the molecules into a three-dimensional network.

Related literature

For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_7H_{10}N_2O_4\cdot H_2O$
M_r = 204.19
Monoclinic, $P2_1/n$
a = 13.332 (3) Å
b = 5.149 (1) Å
c = 14.458 (3) Å
β = 103.09 (3)°
V = 966.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 294 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.954, T_{max} = 0.988
1959 measured reflections
1875 independent reflections
1424 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.163
S = 1.03
1875 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
OW—HW*A*···O1^{i}	0.86	2.05	2.755 (3)	140
N1—H1*A*···O2^{ii}	0.86	2.32	3.150 (2)	163
N2—H2*A*···O4^{i}	0.86	2.13	2.968 (2)	166
C2—H2*B*···O2^{ii}	0.93	2.47	3.305 (3)	149

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); 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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2358).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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N-Acryloylglycylglycine monohydrate

X. Gao, C. Wu, H. Wang and J. Wang

Comment

N-acryloylglycylglycine(I) is one of the useful synthetic intermediates and free radical addition monomers. The crystal structure determination of the title compound, (I), has been carried out in order to elucidate its molecular conformation.

In the molecule of the title compound, (I), (Fig.1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The main chain of the molecule is nearly planar.

In the crystal structure, O—H···O, N—H···O and C—H···O hydrogen bonds (Table 1) link the molecules into a three dimensional network, in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, acryloyl chloride (1.1 ml) containing diphenylpicrylhydrazyl polymerization inhibitor (0.01%) and sodium hydroxide solution [0.61 g, in H_2O (5 ml)] were added dropwise simultaneously over a 30 min period to a well stirred aqueous solution of glycylglycine [2.0 g, in H_2O (30 ml)] and sodium hydroxide [0.61 g, in H_2O (5 ml)], and then stirred 1 h more. The reaction of the mixture was kept at 273 K in an ice-water bath. The solution was acidified to pH = 2 with 6 N HCl and the resulting solid was filtered off and crystallized from ethanol (95%) (yield; 63%, m.p. 421–423 K).

Refinement

H atoms (for H_2O) were located in difference syntheses and constrained to ride on their parent atom [O—H = 0.8543, 0.8553 Å and U_{iso}(H) = 1.2U_{eq}(O)]. The remaining H atoms were positioned geometrically, with N—H = 0.86 Å (for NH), O—H = 0.82 Å (for OH) and C—H = 0.93, 0.93 and 0.97 Å, for aromatic and methylene H atoms and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*N*,*O*), where x = 1.5 for OH H and x = 1.2 for all other H atoms.

Figures

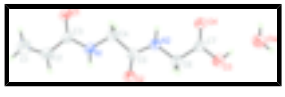

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level

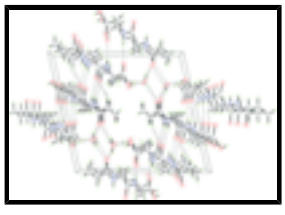

Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

2-(2-acrylamidoacetamido)acetic acid monohydrate

Crystal data

$C_7H_{10}N_2O_4 \cdot H_2O$	$F_{000} = 432$
$M_r = 204.19$	$D_x = 1.403$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 422 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 13.332$ (3) Å	Cell parameters from 25 reflections
$b = 5.149$ (1) Å	$\theta = 9$–13º
$c = 14.458$ (3) Å	$\mu = 0.12$ mm^{-1}
$\beta = 103.09$ (3)º	$T = 294$ (2) K
$V = 966.7$ (4) Å^3	Block, colorless
$Z = 4$	0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.052$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.9$º
Monochromator: graphite	$\theta_{min} = 1.9$º
$T = 294$(2) K	$h = 0 \rightarrow 16$
$\omega/2\theta$ scans	$k = 0 \rightarrow 6$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -17 \rightarrow 17$
$T_{min} = 0.954$, $T_{max} = 0.988$	3 standard reflections
1959 measured reflections	every 120 min
1875 independent reflections	intensity decay: none
1424 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.163$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
1875 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}
127 parameters	Extinction correction: SHELXL97
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.028 (5)
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 $F^2 > 2\text{sigma}(F^2)$ 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}
OW	0.23119 (13)	1.4675 (4)	0.32683 (15)	0.0778 (7)
HWA	0.1700	1.5204	0.3028	0.093*
HWB	0.2821	1.5271	0.3067	0.093*
O1	−0.09533 (11)	0.1547 (3)	0.75924 (12)	0.0549 (5)
O2	0.22255 (11)	0.4246 (3)	0.68108 (11)	0.0471 (4)
O3	0.26156 (12)	1.1329 (4)	0.45810 (13)	0.0596 (5)
H3A	0.2427	1.2454	0.4178	0.089*
O4	0.09464 (11)	1.1214 (3)	0.46083 (12)	0.0524 (5)
N1	0.06626 (12)	0.2081 (4)	0.73869 (12)	0.0394 (4)
H1A	0.1293	0.1580	0.7550	0.047*
N2	0.12573 (12)	0.7266 (3)	0.59002 (12)	0.0401 (5)
H2A	0.0655	0.7923	0.5697	0.048*
C1	−0.0148 (2)	−0.2397 (8)	0.8963 (3)	0.1003 (14)
H1B	−0.0855	−0.2131	0.8852	0.120*
H1C	0.0166	−0.3632	0.9403	0.120*
C2	0.03980 (18)	−0.1049 (5)	0.85048 (17)	0.0520 (6)
H2B	0.1103	−0.1356	0.8630	0.062*
C3	−0.00320 (16)	0.0949 (4)	0.77944 (15)	0.0392 (5)
C4	0.03959 (15)	0.4106 (4)	0.66844 (15)	0.0408 (5)
H4A	0.0015	0.5463	0.6919	0.049*
H4B	−0.0036	0.3403	0.6107	0.049*
C5	0.13773 (14)	0.5225 (4)	0.64742 (14)	0.0352 (5)
C6	0.21375 (16)	0.8398 (5)	0.56131 (17)	0.0463 (6)
H6A	0.2508	0.7044	0.5363	0.056*
H6B	0.2601	0.9146	0.6164	0.056*
C7	0.18165 (15)	1.0451 (4)	0.48762 (15)	0.0386 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
OW	0.0528 (11)	0.0893 (15)	0.1042 (16)	0.0224 (10)	0.0447 (10)	0.0482 (13)
O1	0.0335 (8)	0.0620 (11)	0.0732 (11)	0.0044 (7)	0.0204 (7)	0.0196 (9)

O2	0.0321 (8)	0.0464 (9)	0.0635 (10)	0.0015 (7)	0.0121 (7)	0.0119 (8)
O3	0.0356 (9)	0.0713 (12)	0.0752 (12)	0.0008 (8)	0.0194 (8)	0.0283 (10)
O4	0.0324 (9)	0.0629 (11)	0.0634 (10)	0.0051 (7)	0.0144 (7)	0.0182 (8)
N1	0.0292 (9)	0.0432 (10)	0.0472 (9)	−0.0002 (7)	0.0116 (7)	0.0040 (8)
N2	0.0290 (9)	0.0410 (10)	0.0515 (10)	0.0013 (7)	0.0116 (7)	0.0072 (8)
C1	0.0625 (19)	0.128 (3)	0.117 (3)	0.017 (2)	0.0358 (19)	0.078 (3)
C2	0.0392 (12)	0.0551 (15)	0.0633 (15)	0.0032 (10)	0.0147 (11)	0.0164 (12)
C3	0.0325 (10)	0.0421 (12)	0.0456 (11)	−0.0012 (9)	0.0141 (9)	0.0001 (9)
C4	0.0332 (11)	0.0448 (12)	0.0457 (11)	−0.0024 (9)	0.0114 (9)	0.0029 (10)
C5	0.0295 (10)	0.0375 (11)	0.0389 (10)	−0.0028 (8)	0.0088 (8)	−0.0052 (9)
C6	0.0320 (11)	0.0467 (13)	0.0614 (13)	0.0004 (9)	0.0130 (9)	0.0124 (11)
C7	0.0308 (10)	0.0410 (11)	0.0453 (12)	−0.0047 (9)	0.0116 (8)	−0.0014 (9)

Geometric parameters (Å, °)

OW—HWA	0.8553	N2—H2A	0.8600
OW—HWB	0.8543	C1—C2	1.292 (4)
O1—C3	1.235 (3)	C1—H1B	0.9300
O2—C5	1.233 (2)	C1—H1C	0.9300
O3—C7	1.314 (2)	C2—C3	1.475 (3)
O3—H3A	0.8200	C2—H2B	0.9300
O4—C7	1.203 (2)	C4—C5	1.522 (3)
N1—C3	1.338 (3)	C4—H4A	0.9700
N1—C4	1.443 (3)	C4—H4B	0.9700
N1—H1A	0.8600	C6—C7	1.493 (3)
N2—C5	1.326 (3)	C6—H6A	0.9700
N2—C6	1.452 (3)	C6—H6B	0.9700
HWA—OW—HWB	120.4	N1—C4—H4A	109.8
C3—N1—C4	122.67 (17)	C5—C4—H4A	109.8
C3—N1—H1A	118.7	N1—C4—H4B	109.8
C4—N1—H1A	118.7	C5—C4—H4B	109.8
C2—C1—H1B	120.0	H4A—C4—H4B	108.3
C2—C1—H1C	120.0	O2—C5—N2	122.73 (18)
H1B—C1—H1C	120.0	O2—C5—C4	121.30 (19)
C5—N2—C6	120.40 (17)	N2—C5—C4	115.96 (17)
C5—N2—H2A	119.8	N2—C6—C7	111.62 (18)
C6—N2—H2A	119.8	N2—C6—H6A	109.3
C1—C2—C3	123.8 (2)	C7—C6—H6A	109.3
C1—C2—H2B	118.1	N2—C6—H6B	109.3
C3—C2—H2B	118.1	C7—C6—H6B	109.3
C7—O3—H3A	109.5	H6A—C6—H6B	108.0
O1—C3—N1	122.5 (2)	O4—C7—O3	125.2 (2)
O1—C3—C2	123.19 (19)	O4—C7—C6	124.11 (19)
N1—C3—C2	114.31 (18)	O3—C7—C6	110.66 (18)
N1—C4—C5	109.16 (16)		
C4—N1—C3—O1	0.5 (3)	C6—N2—C5—C4	−176.70 (19)
C4—N1—C3—C2	−179.1 (2)	N1—C4—C5—O2	6.8 (3)
C1—C2—C3—O1	1.7 (5)	N1—C4—C5—N2	−174.33 (17)
C1—C2—C3—N1	−178.6 (3)	C5—N2—C6—C7	172.89 (18)

C3—N1—C4—C5	171.95 (18)	N2—C6—C7—O4	5.4 (3)
C6—N2—C5—O2	2.2 (3)	N2—C6—C7—O3	−175.83 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
OW—HWA···O1^{i}	0.86	2.05	2.755 (3)	140
N1—H1A···O2^{ii}	0.86	2.32	3.150 (2)	163
N2—H2A···O4^{i}	0.86	2.13	2.968 (2)	166
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Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) −*x*+1/2, *y*−1/2, −*z*+3/2.

Fig. 1

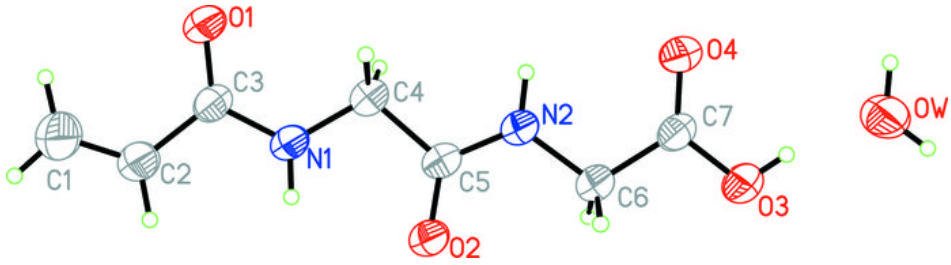

Fig. 2