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N-Acryloylphenylalanine

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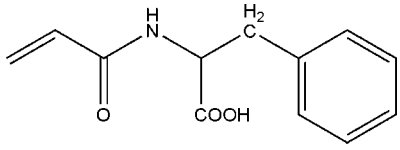
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.009 Å; R factor = 0.063; wR factor = 0.161; data-to-parameter ratio = 7.5.

The title compound, $C_{12}H_{13}NO_3$, was prepared by the nucleophilic substitution reaction of acryloyl chloride with glycylglycine. In the crystal structure, intermolecular N—H···O, O–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

$C_{12}H_{13}NO_3$
M_r = 219.23
Monoclinic, $P2_1$
a = 6.0050 (12) Å
b = 7.5820 (15) Å
c = 12.512 (3) Å
β = 98.58 (3)°
V = 563.3 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 291 (2) K
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
1195 measured reflections
1088 independent reflections
940 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.161
S = 1.00
1088 reflections
145 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O2^{i}	0.86	2.30	3.036 (6)	144
O1—H1B···O3^{ii}	0.82	1.84	2.614 (6)	156
C12—H12B···O1^{iii}	0.93	2.60	3.178 (8)	121

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

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, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: HK2488).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o1483 [doi:10.1107/S1600536808020849]

N-Acryloylphenylalanine

Cong-Ren Wu, Xiao-Feng Gao, Hai-Bo Wang, Dong Jin and Jin-Tang Wang

S1. Comment

N-Acryloylphenylalanie is one of the useful synthetic intermediates and free radical addition monomers. The crystal structure determination of the title compound has been carried out in order to elucidate the molecular conformation. We report herein its synthesis and crystal structure.

In the molecule of the title compound (Fig. 1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

In the crystal structure, intermolecular N-H···O, O-H···O and C-H···O hydrogen bonds (Table 1) link the molecules into a three dimensional network (Fig. 2), in which they may be effective in stabilization of the structure.

S2. Experimental

For the preparation of the title compound, to a well stirred solutions of phenylalanie (2.5 g) in H_2O (30 ml) and sodium hydroxide (0.66 g) in H_2O (5 ml), acryloyl chloride (1.34 ml) containing diphenylpicrylhydrazyl polymerization inhibitor (0.01%) and sodium hydroxide solution (0.66 g) in H_2O (5 ml) were added dropwise simultaneously over a 30 min period and the stirring was continued for another 1 h. The reaction mixture was kept at 273 K in an ice-water bath. The solution was acidified to pH = 2 with HCl (6 N). The resulting solid was filtered off, and crystallized from ethanol (95%) (yield; 61%, m.p.401-403 K).

S3. Refinement

H atoms were positioned geometrically, with O-H = 0.82 Å (for OH), N-H = 0.86 Å (for NH) and C-H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H, respectively, 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.

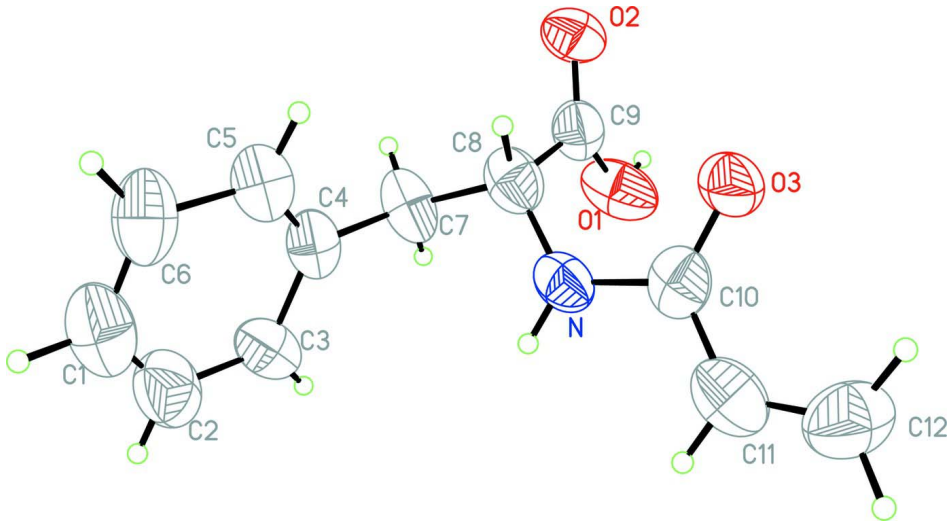

Figure 1

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

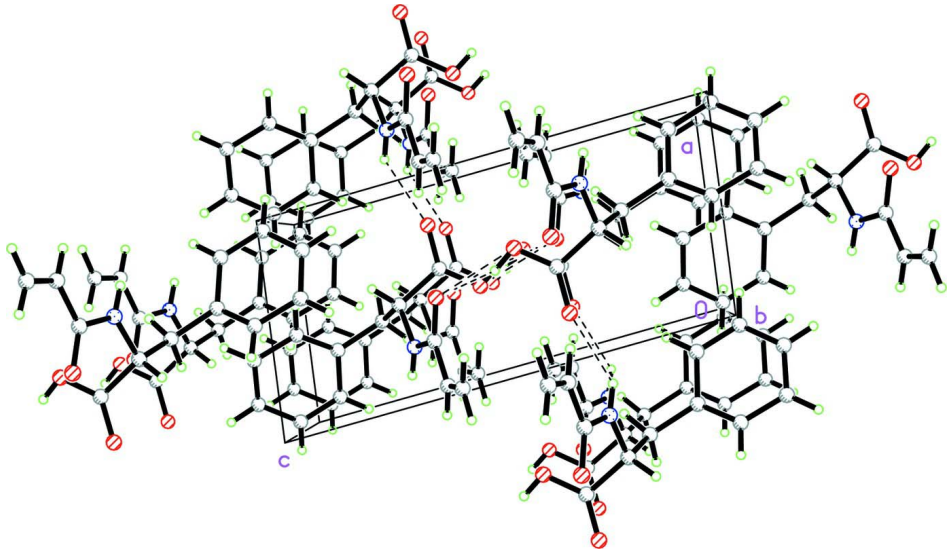

Figure 2

A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

N-Acryloylphenylalanine

Crystal data

$C_{12}H_{13}NO_3$
$M_r = 219.23$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.0050\ (12)$ Å
$b = 7.5820\ (15)$ Å
$c = 12.512\ (3)$ Å
$\beta = 98.58\ (3)°$
$V = 563.3\ (2)$ Å^3
$Z = 2$
$F(000) = 232$
$D_x = 1.293$ Mg m^{-3}
Melting point: 402 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–14°$

μ = 0.09 mm^{-1}
T = 291 K

Block, colorless
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
1195 measured reflections

1088 independent reflections
940 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.2°, θ_{min} = 1.7°
h = −7→7
k = 0→9
l = 0→14
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.161
S = 1.01
1088 reflections
145 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.62P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.028 (5)

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}
N	0.7481 (7)	0.7059 (7)	0.2952 (3)	0.0555 (11)
H0A	0.8730	0.7469	0.2798	0.067*
O1	0.5602 (6)	0.8820 (8)	0.4513 (3)	0.0843 (15)
H1B	0.4871	0.9128	0.4985	0.126*
C1	0.8147 (14)	0.9763 (11)	−0.0922 (6)	0.086 (2)
H1A	0.8611	0.9777	−0.1599	0.103*
O2	0.2165 (6)	0.8618 (7)	0.3516 (3)	0.0700 (11)
C2	0.9519 (13)	1.0469 (10)	−0.0068 (7)	0.084 (2)
H2A	1.0883	1.0979	−0.0164	0.101*
O3	0.5572 (6)	0.4855 (7)	0.3658 (3)	0.0632 (11)
C3	0.8883 (9)	1.0423 (9)	0.0922 (5)	0.0699 (16)

H3A	0.9839	1.0857	0.1517	0.084*
C4	0.6681 (9)	0.9687 (8)	0.1060 (4)	0.0592 (13)
C5	0.5445 (10)	0.8933 (9)	0.0224 (4)	0.0670 (15)
H5A	0.4140	0.8336	0.0325	0.080*
C6	0.6059 (12)	0.9014 (10)	−0.0818 (5)	0.0801 (19)
H6A	0.5110	0.8584	−0.1417	0.096*
C7	0.5920 (12)	0.9795 (10)	0.2154 (5)	0.0743 (17)
H7A	0.4576	1.0520	0.2085	0.089*
H7B	0.7079	1.0400	0.2642	0.089*
C8	0.5411 (10)	0.8052 (8)	0.2676 (4)	0.0608 (15)
H8A	0.4362	0.7369	0.2159	0.073*
C9	0.4281 (9)	0.8489 (9)	0.3655 (4)	0.0634 (15)
C10	0.7431 (9)	0.5410 (8)	0.3477 (3)	0.0566 (14)
C11	0.9502 (11)	0.4520 (10)	0.3691 (4)	0.0701 (18)
H11A	1.0794	0.5112	0.3564	0.084*
C12	0.9710 (12)	0.2919 (10)	0.4055 (6)	0.083 (2)
H12A	0.8446	0.2297	0.4189	0.099*
H12B	1.1124	0.2392	0.4183	0.099*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.055 (2)	0.065 (3)	0.049 (2)	−0.007 (2)	0.0183 (18)	−0.002 (2)
O1	0.076 (3)	0.118 (4)	0.066 (2)	−0.021 (3)	0.033 (2)	−0.036 (3)
C1	0.118 (5)	0.070 (4)	0.076 (4)	0.005 (5)	0.036 (4)	0.008 (4)
O2	0.0515 (19)	0.084 (3)	0.078 (2)	0.001 (2)	0.0231 (17)	0.001 (3)
C2	0.093 (5)	0.074 (5)	0.090 (4)	0.000 (4)	0.029 (4)	0.023 (4)
O3	0.0551 (19)	0.090 (3)	0.0452 (18)	−0.009 (2)	0.0088 (14)	0.010 (2)
C3	0.062 (3)	0.074 (4)	0.078 (4)	−0.003 (3)	0.026 (3)	0.000 (3)
C4	0.077 (3)	0.052 (3)	0.052 (3)	0.004 (3)	0.020 (2)	0.005 (3)
C5	0.081 (4)	0.063 (4)	0.059 (3)	−0.003 (3)	0.019 (3)	0.004 (3)
C6	0.116 (5)	0.077 (5)	0.048 (3)	0.001 (4)	0.017 (3)	−0.004 (3)
C7	0.099 (4)	0.066 (4)	0.064 (3)	−0.017 (4)	0.032 (3)	0.002 (3)
C8	0.072 (3)	0.064 (4)	0.048 (3)	−0.012 (3)	0.016 (2)	−0.006 (3)
C9	0.068 (3)	0.068 (4)	0.055 (3)	−0.006 (3)	0.011 (2)	0.010 (3)
C10	0.073 (3)	0.069 (4)	0.030 (2)	0.003 (3)	0.011 (2)	−0.006 (2)
C11	0.083 (4)	0.080 (5)	0.053 (3)	−0.010 (4)	0.031 (3)	−0.014 (3)
C12	0.067 (4)	0.063 (4)	0.115 (6)	0.003 (3)	0.003 (4)	−0.014 (4)

Geometric parameters (Å, º)

N—C10	1.414 (8)	C4—C7	1.509 (7)
N—C8	1.451 (7)	C5—C6	1.408 (8)
N—H0A	0.8600	C5—H5A	0.9300
O1—C9	1.261 (6)	C6—H6A	0.9300
O1—H1B	0.8200	C7—C8	1.525 (9)
C1—C2	1.358 (11)	C7—H7A	0.9700
C1—C6	1.400 (10)	C7—H7B	0.9700

C1—H1A	0.9300	C8—C9	1.523 (7)
O2—C9	1.261 (6)	C8—H8A	0.9800
C2—C3	1.350 (10)	C10—C11	1.405 (9)
C2—H2A	0.9300	C11—C12	1.296 (10)
O3—C10	1.245 (6)	C11—H11A	0.9300
C3—C4	1.468 (8)	C12—H12A	0.9300
C3—H3A	0.9300	C12—H12B	0.9300
C4—C5	1.319 (8)		

C10—N—C8	119.5 (4)	C4—C7—H7A	108.1
C10—N—H0A	120.2	C8—C7—H7A	108.1
C8—N—H0A	120.2	C4—C7—H7B	108.1
C9—O1—H1B	109.5	C8—C7—H7B	108.1
C2—C1—C6	122.2 (6)	H7A—C7—H7B	107.3
C2—C1—H1A	118.9	N—C8—C9	112.9 (5)
C6—C1—H1A	118.9	N—C8—C7	109.4 (5)
C3—C2—C1	119.4 (7)	C9—C8—C7	107.3 (5)
C3—C2—H2A	120.3	N—C8—H8A	109.0
C1—C2—H2A	120.3	C9—C8—H8A	109.0
C2—C3—C4	120.0 (6)	C7—C8—H8A	109.0
C2—C3—H3A	120.0	O2—C9—O1	126.5 (5)
C4—C3—H3A	120.0	O2—C9—C8	117.8 (5)
C5—C4—C3	118.8 (5)	O1—C9—C8	115.4 (5)
C5—C4—C7	122.2 (5)	O3—C10—C11	126.5 (6)
C3—C4—C7	119.0 (5)	O3—C10—N	117.7 (5)
C4—C5—C6	121.4 (6)	C11—C10—N	115.7 (5)
C4—C5—H5A	119.3	C12—C11—C10	123.6 (7)
C6—C5—H5A	119.3	C12—C11—H11A	118.2
C1—C6—C5	117.7 (6)	C10—C11—H11A	118.2
C1—C6—H6A	121.1	C11—C12—H12A	120.0
C5—C6—H6A	121.1	C11—C12—H12B	120.0
C4—C7—C8	116.7 (6)	H12A—C12—H12B	120.0

C6—C1—C2—C3	1.3 (12)	C10—N—C8—C7	178.2 (4)
C1—C2—C3—C4	−2.8 (11)	C4—C7—C8—N	67.3 (7)
C2—C3—C4—C5	6.2 (10)	C4—C7—C8—C9	−169.9 (5)
C2—C3—C4—C7	−174.7 (7)	N—C8—C9—O2	−149.5 (6)
C3—C4—C5—C6	−8.0 (10)	C7—C8—C9—O2	89.9 (7)
C7—C4—C5—C6	172.9 (7)	N—C8—C9—O1	35.8 (8)
C2—C1—C6—C5	−2.9 (12)	C7—C8—C9—O1	−84.9 (7)
C4—C5—C6—C1	6.5 (10)	C8—N—C10—O3	1.8 (6)
C5—C4—C7—C8	58.1 (9)	C8—N—C10—C11	178.5 (4)
C3—C4—C7—C8	−121.1 (7)	O3—C10—C11—C12	4.3 (9)
C10—N—C8—C9	58.7 (6)	N—C10—C11—C12	−172.1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O2^{i}	0.86	2.30	3.036 (6)	144
O1—H1*B*···O3^{ii}	0.82	1.84	2.614 (6)	156
C12—H12*B*···O1^{iii}	0.93	2.60	3.178 (8)	121

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) −*x*+2, *y*−1/2, −*z*+1.