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3-(Phenylcarbamoyl)acrylic acid

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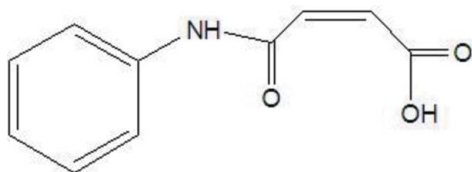
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.049; wR factor = 0.157; data-to-parameter ratio = 17.1.

In the title compound, $\text{C}_{10}\text{H}_9\text{NO}_3$, the dihedral angle between the phenyl ring and the amide group is $10.8(2)^\circ$. The $\text{C}=\text{O}$ and $\text{O}-\text{H}$ bonds of the carboxyl group adopt an *anti* orientation and an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond closes an $S(7)$ ring. In the crystal, $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into $C(7)$ chains propagating in $[101]$. The packing is consolidated by $\text{C}-\text{H}\cdots\text{O}$ interactions, generating sheets aligned at an angle of *ca* 60° with the bc plane

Related literature

For background to carboxylic acids in supramolecular chemistry, see: Gossel *et al.* (2006). For a related structure, see: Jin *et al.* (2010).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{NO}_3$
 $M_r = 191.18$
 Monoclinic, $P2_1/n$
 $a = 7.2396(8)$ Å

$b = 10.5918(11)$ Å
 $c = 11.7718(15)$ Å
 $\beta = 99.122(1)^\circ$
 $V = 891.25(18)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹

$T = 298$ K
 $0.38 \times 0.36 \times 0.33$ mm

Data collection

Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
 $T_{\min} = 0.960$, $T_{\max} = 0.965$

5497 measured reflections
 2190 independent reflections
 1356 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.157$
 $S = 1.03$
 2190 reflections

128 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, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O3}$	0.82	1.68	2.4947 (19)	175
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{ii}}$	0.86	2.04	2.885 (2)	169
$\text{C9}-\text{H9}\cdots\text{O3}^{\text{ii}}$	0.93	2.51	3.389 (2)	157
$\text{C10}-\text{H10}\cdots\text{O2}^{\text{ii}}$	0.93	2.58	3.335 (2)	138

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; 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: HB6935).

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supporting information

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3-(Phenylcarbamoyl)acrylic acid

Shouwen Jin, Yanfei Huang, Shuaishuai Wei, Yong Zhou and Yingping Zhou

S1. Comment

The carboxylic acid contains the important hydrogen bonding functional group for crystal engineering (Grossel *et al.*, 2006). As an extension of our study concentrating on hydrogen bonded assembly of organic acid and organic base (Jin *et al.*, 2010), herein we report the crystal structure of 3-phenylcarbamoyl-acrylic acid.

The single-crystal of the title compound (Fig.1) with the formula $C_{10}H_9NO_3$ was obtained by recrystallization of 3-phenylcarbamoyl-acrylic acid and 2-methylquinoline from a methanol solution. However the 2-methylquinoline molecules do not appear in the title compound. X-ray diffraction analysis indicated that the asymmetric unit of the structure contains one molecule. The conformations of the amide oxygen and the carbonyl oxygen of the acid segments are anti to each other and the amide oxygen is anti to the H atom on the olefinic group, while the carbonyl oxygen of the acid is *syn* to the CH at the olefinic group. Thus there existed intramolecular O—H \cdots O hydrogen bond producing a $S_1^1(7)$ ring.

The dihedral angle between the phenyl ring and the amide group in the molecule is $10.8(2)^\circ$.

Two adjacent 3-phenylcarbamoyl-acrylic acids were joined together *via* the N—H \cdots O, and CH—O interactions to form a dimer. Both carboxylic acids in the dimer were almost perpendicular with each other. In the dimer there are hydrogen-bonded ring motifs with descriptors of $R_2^1(6)$, and $R_2^2(8)$. The two ring motifs were fused together by the N—H \cdots O hydrogen bond. The neighboring carboxylic dimers were linked together through the CH—O associations between the benzene CH and the carbonyl group with C—O distance of 3.389 Å to form one-dimensional chain. The one-dimensional chains were combined together by the interchain CH—O, and N—H \cdots O interactions to form two-dimensional sheet extending at the direction that made a dihedral angle of *ca* 60° with the *bc* plane (Fig. 2). Two two-dimensional sheets were further held together by the intersheet C— π interactions with C \cdots C $_{\pi}$ distance of 3.369 Å to generate two-dimensional double sheet structure.

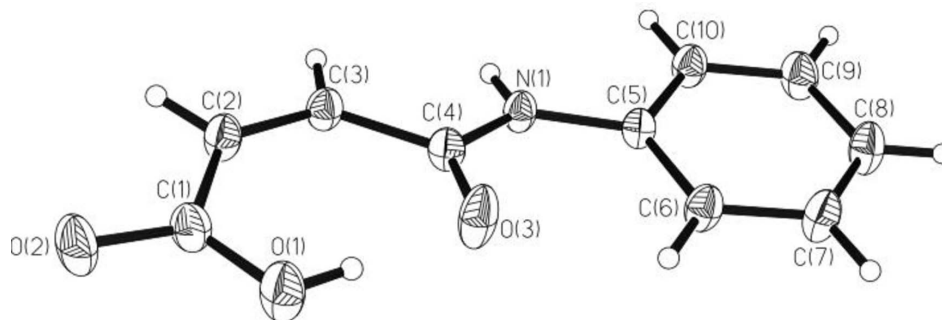
S2. Experimental

Crystals of 3-phenylcarbamoyl-acrylic acid were formed by slow evaporation of its methanol solution at room temperature. 3-phenylcarbamoyl-acrylic acid (19.1 mg, 0.10 mmol) was dissolved in 4 ml of methanol, and 2-methylquinoline (14.3 mg, 0.1 mmol) was added to the methanol solution. The solution was then filtered into a test tube and left standing at room temperature. After about one week colorless blocks crystals were obtained.

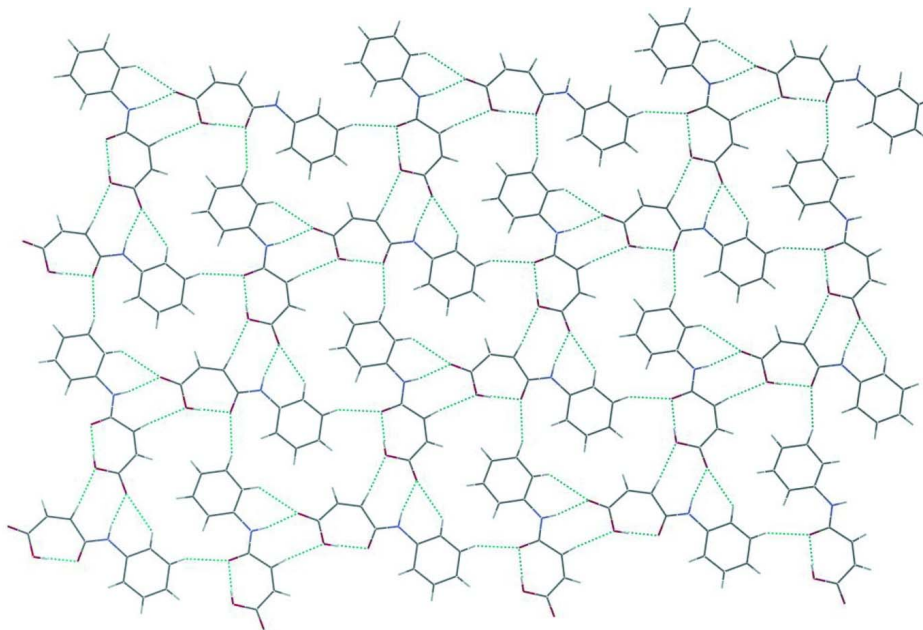
S3. Refinement

H atoms bonded to O, and N atoms were located in a difference Fourier map and refined isotropically.

Other H atoms were positioned geometrically with C—H = 0.93 Å for aromatic, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.

**Figure 1**

The structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

**Figure 2**

Two-dimensional sheet structure formed through hydrogen bonds.

3-(Phenylcarbamoyl)acrylic acid

Crystal data

$C_{10}H_9NO_3$

$M_r = 191.18$

Monoclinic, $P2_1/n$

$a = 7.2396$ (8) Å

$b = 10.5918$ (11) Å

$c = 11.7718$ (15) Å

$\beta = 99.122$ (1)°

$V = 891.25$ (18) Å³

$Z = 4$

$F(000) = 400$

$D_x = 1.425$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

$\mu = 0.11$ mm⁻¹

$T = 298$ K

BLOCK, colorless

$0.38 \times 0.36 \times 0.33$ mm

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2002)

$T_{\min} = 0.960$, $T_{\max} = 0.965$

5497 measured reflections

2190 independent reflections

1356 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 2.6^\circ$

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 8$

$l = -15 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.157$

$S = 1.03$

2190 reflections

128 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2 + 0.0617P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL*,

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.020 (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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.0940 (2)	-0.19833 (13)	0.83751 (12)	0.0561 (5)
H1	1.0527	-0.1373	0.7987	0.084*
N1	0.7880 (2)	0.00622 (13)	0.53533 (12)	0.0340 (4)
H1A	0.7299	-0.0337	0.4767	0.041*
O2	1.0652 (2)	-0.40422 (14)	0.83105 (12)	0.0546 (4)
C4	0.8638 (3)	-0.06557 (17)	0.62392 (15)	0.0348 (4)
O3	0.9581 (2)	-0.02077 (13)	0.71212 (13)	0.0585 (5)
C3	0.8248 (3)	-0.20179 (16)	0.60914 (15)	0.0358 (4)
H3	0.7460	-0.2246	0.5421	0.043*
C5	0.7910 (2)	0.14022 (16)	0.52529 (14)	0.0326 (4)
C10	0.6725 (3)	0.19311 (17)	0.43353 (16)	0.0401 (5)
H10	0.5983	0.1413	0.3811	0.048*
C6	0.9055 (3)	0.21772 (18)	0.60146 (16)	0.0414 (5)
H6	0.9883	0.1831	0.6619	0.050*
C2	0.8880 (3)	-0.29664 (17)	0.67949 (16)	0.0395 (5)

H2	0.8378	-0.3748	0.6554	0.047*
C1	1.0230 (3)	-0.30190 (18)	0.78871 (16)	0.0395 (5)
C9	0.6645 (3)	0.32210 (18)	0.41997 (17)	0.0476 (5)
H9	0.5842	0.3573	0.3586	0.057*
C7	0.8946 (3)	0.34771 (19)	0.58618 (17)	0.0487 (5)
H7	0.9698	0.4001	0.6374	0.058*
C8	0.7752 (3)	0.39960 (19)	0.49705 (18)	0.0496 (6)
H8	0.7683	0.4868	0.4882	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0747 (11)	0.0394 (9)	0.0446 (9)	0.0003 (7)	-0.0197 (7)	0.0041 (6)
N1	0.0425 (9)	0.0265 (8)	0.0302 (8)	-0.0006 (6)	-0.0026 (6)	-0.0013 (6)
O2	0.0712 (11)	0.0410 (9)	0.0462 (9)	0.0107 (7)	-0.0073 (7)	0.0127 (7)
C4	0.0423 (10)	0.0309 (10)	0.0290 (9)	0.0009 (8)	-0.0007 (7)	-0.0001 (7)
O3	0.0913 (12)	0.0311 (8)	0.0420 (8)	-0.0062 (7)	-0.0235 (7)	0.0000 (6)
C3	0.0426 (10)	0.0298 (10)	0.0324 (9)	-0.0020 (8)	-0.0023 (7)	-0.0014 (7)
C5	0.0402 (10)	0.0272 (9)	0.0298 (9)	0.0003 (7)	0.0043 (7)	0.0001 (7)
C10	0.0482 (11)	0.0320 (10)	0.0367 (10)	-0.0003 (8)	-0.0038 (8)	0.0016 (8)
C6	0.0533 (12)	0.0332 (11)	0.0346 (10)	-0.0037 (8)	-0.0022 (8)	0.0009 (8)
C2	0.0492 (12)	0.0294 (10)	0.0375 (10)	-0.0016 (8)	-0.0006 (8)	-0.0006 (8)
C1	0.0468 (12)	0.0357 (11)	0.0347 (10)	0.0038 (8)	0.0022 (8)	0.0047 (8)
C9	0.0596 (13)	0.0371 (11)	0.0414 (11)	0.0067 (10)	-0.0061 (9)	0.0071 (9)
C7	0.0679 (15)	0.0329 (11)	0.0420 (11)	-0.0073 (10)	-0.0018 (10)	-0.0023 (9)
C8	0.0715 (15)	0.0297 (11)	0.0459 (12)	0.0000 (10)	0.0041 (11)	0.0037 (9)

Geometric parameters (Å, °)

O1—C1	1.305 (2)	C10—C9	1.376 (2)
O1—H1	0.8200	C10—H10	0.9300
N1—C4	1.336 (2)	C6—C7	1.389 (3)
N1—C5	1.425 (2)	C6—H6	0.9300
N1—H1A	0.8600	C2—C1	1.488 (3)
O2—C1	1.212 (2)	C2—H2	0.9300
C4—O3	1.243 (2)	C9—C8	1.382 (3)
C4—C3	1.475 (2)	C9—H9	0.9300
C3—C2	1.336 (3)	C7—C8	1.365 (3)
C3—H3	0.9300	C7—H7	0.9300
C5—C10	1.387 (3)	C8—H8	0.9300
C5—C6	1.389 (3)		
C1—O1—H1	109.5	C5—C6—H6	120.4
C4—N1—C5	128.47 (15)	C7—C6—H6	120.4
C4—N1—H1A	115.8	C3—C2—C1	132.65 (18)
C5—N1—H1A	115.8	C3—C2—H2	113.7
O3—C4—N1	122.54 (17)	C1—C2—H2	113.7
O3—C4—C3	122.77 (17)	O2—C1—O1	120.98 (18)

N1—C4—C3	114.68 (15)	O2—C1—C2	118.50 (18)
C2—C3—C4	128.48 (17)	O1—C1—C2	120.51 (16)
C2—C3—H3	115.8	C10—C9—C8	120.30 (19)
C4—C3—H3	115.8	C10—C9—H9	119.9
C10—C5—C6	119.76 (17)	C8—C9—H9	119.9
C10—C5—N1	116.85 (16)	C8—C7—C6	120.97 (19)
C6—C5—N1	123.40 (16)	C8—C7—H7	119.5
C9—C10—C5	120.07 (18)	C6—C7—H7	119.5
C9—C10—H10	120.0	C7—C8—C9	119.76 (19)
C5—C10—H10	120.0	C7—C8—H8	120.1
C5—C6—C7	119.11 (18)	C9—C8—H8	120.1
C5—N1—C4—O3	-3.0 (3)	N1—C5—C6—C7	-178.30 (17)
C5—N1—C4—C3	176.37 (16)	C4—C3—C2—C1	-4.5 (4)
O3—C4—C3—C2	-4.1 (3)	C3—C2—C1—O2	-174.8 (2)
N1—C4—C3—C2	176.5 (2)	C3—C2—C1—O1	5.2 (3)
C4—N1—C5—C10	-168.15 (18)	C5—C10—C9—C8	0.4 (3)
C4—N1—C5—C6	12.2 (3)	C5—C6—C7—C8	-0.8 (3)
C6—C5—C10—C9	-1.9 (3)	C6—C7—C8—C9	-0.7 (3)
N1—C5—C10—C9	178.43 (17)	C10—C9—C8—C7	0.8 (3)
C10—C5—C6—C7	2.0 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O3	0.82	1.68	2.4947 (19)	175
N1—H1A \cdots O2 ⁱ	0.86	2.04	2.885 (2)	169
C9—H9 \cdots O3 ⁱⁱ	0.93	2.51	3.389 (2)	157
C10—H10 \cdots O2 ⁱ	0.93	2.58	3.335 (2)	138

Symmetry codes: (i) $x-1/2, -y-1/2, z-1/2$; (ii) $x-1/2, -y+1/2, z-1/2$.