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(*Z*)-2-Acetamido-3-(4-chlorophenyl)-acrylic acid

Qi-Jian Tian,* Hui Ouyang, Chun-Lian Tian and Yong-Dong Jiang

Key Laboratory of Hunan Forest Products and, Chemical Industry Engineering, Jishou University, Jishou 416000, People's Republic of China
Correspondence e-mail: shenyangzhou@163.com

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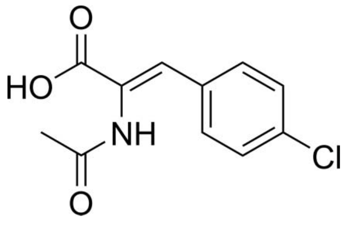
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.052; wR factor = 0.229; data-to-parameter ratio = 7.2.

In the title compound, $C_{11}H_{10}ClNO_3$, the molecule consists of a benzene ring and an acetamidoacrylic acid unit on opposite sides of the C=C double bond. In the crystal, intermolecular O—H⋯O and N—H⋯O hydrogen bonds assemble the molecules into infinite two-dimensional ribbons. These ribbons are linked into a network by intermolecular C—H⋯π contacts.

Related literature

Derivatives of 2-acetamido-3-phenylacrylic acid are key intermates in the preparations of tanshinol (Wong *et al.* 1992; Xiao, *et al.* 2008*a*), diaryl-3-hydroxy-2(5*H*)-furanones (Weber *et al.* 2002; Xiao *et al.* 2008*b*) and benzylazauracil (Chen *et al.* 1993; Xiao, *et al.* 2008*c*), which show anti-platelet aggregation, antifungal and antiviral activities, respectively.



Experimental

Crystal data

$C_{11}H_{10}ClNO_3$
M_r = 239.65
Monoclinic, $P2_1$
a = 6.2440 (12) Å
b = 7.5450 (15) Å
c = 11.813 (2) Å
β = 100.47 (3)°
V = 547.26 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 298 K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.935, T_{max} = 0.967
1160 measured reflections
1060 independent reflections
895 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.229
S = 1.01
1060 reflections
148 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1^{i}	0.86	2.09	2.933 (7)	165
O2—H2A⋯O3^{ii}	0.82	1.86	2.606 (7)	152
C3—H3⋯Cg1^{iii}	0.93	2.85	3.523 (8)	130

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y-\frac{1}{2}, -z$; (iii) $-x, y+\frac{1}{2}, -z+1$. Cg1 is the centroid of the C1–C6 ring.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2173).

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supplementary materials

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(*Z*)-2-Acetamido-3-(4-chlorophenyl)acrylic acid

Q.-J. Tian, H. Ouyang, C.-L. Tian and Y.-D. Jiang

Comment

Derivatives of 2-acetamido-3-phenylacrylic acid are key intermates for tanshinol (Wong *et al.* 1992; Xiao, *et al.* 2008*a*), diaryl-3-hydroxy-2(5*H*)-furanones (Weber *et al.* 2002; Xiao *et al.* 2008*b*) and benzylazauracil (Chen *et al.* 1993; Xiao, *et al.* 2008*c*), which show anti-platelet aggregation, antifungal and antiviral activities, respectively. In the course of our work on screening for anticancers, we synthesized the title compound and herein reported its crystal structure.

In the title compound (I), the plane of benzene ring (with mean dieviation deviation of 0.0053 Å) and the plane of hydroxy acrylic moiety (with mean deviation of 0.0049 Å) make a dihedral angle of 18.001 (97) Å. The benzene ring and the carboxy group occur on opposite side of the C8=C9 double bond with torsion angle of 179.8 (4) ° (Fig. 1). Intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) assemble the molecules into an infinite two-dimensional ribbon. This ribbons further form a network *via* C—H···pi contact (Fig. 2).

Experimental

The mixture of alpha-acetoaminocinnamic acid (2.35 g, 10 mmol) in 0.5*M* HCl (60 mL) was refluxed for 3 h. The resulting mixture was allowed to cool to room temperature and the resulting precipitate was collected by filtration. The crude product was dissolved in EtOAc and twofold volume of petroleum was added carefully. Colorless blocks of (I) suitable for single-crystal structure determination was furnished after 2 d.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H of 0.93 Å for the aromatic atoms and =CH groups, 0.96 Å for the CH_3 groups, 0.82 Å for the OH groups and 0.86 Å for the NH groups. U_{iso}(H) values were set at 1.2 times U_{eq}(C) for aromatic C double bond C groups, 1.5 times U_{eq}(C) for CH_3 and 1.5 times U_{eq}(O) for O—H groups. Because the absolute structure parameter is meaningless with a rather poor accuracy, the chemical absolute configuration could not be determined unambiguously

Figures

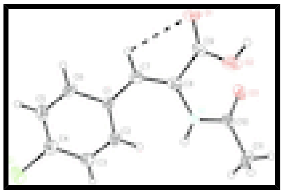

Fig. 1. A view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

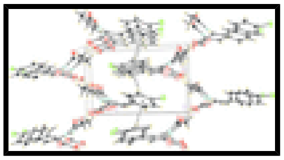

Fig. 2. An infinite two-dimensional ribbon is formed through intermolecular O—H···O hydrogen bonds. Dashed lines indicate hydrogen bonds and solid dashed lines indicate C—H···π contacts.

(*Z*)-2-Acetamido-3-(4-chlorophenyl)acrylic acid

Crystal data

$C_{11}H_{10}ClNO_3$

$M_r = 239.65$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 6.2440 (12)$ Å

$b = 7.5450 (15)$ Å

$c = 11.813 (2)$ Å

$\beta = 100.47 (3)°$

$V = 547.26 (19)$ Å^3

$Z = 2$

$F_{000} = 248$

$D_x = 1.454$ Mg m^{-3}

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

Cell parameters from 775 reflections

$\theta = 1.9–24.7°$

$\mu = 0.34$ mm^{-1}

$T = 298$ K

Block, colorless

0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.935$, $T_{max} = 0.967$

1160 measured reflections

1060 independent reflections

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

$R_{int} = 0.036$

$\theta_{max} = 25.2°$

$\theta_{min} = 1.8°$

$h = 0\rightarrow7$

$k = 0\rightarrow9$

$l = -14\rightarrow13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.052$

$wR(F^2) = 0.229$

$S = 1.01$

1060 reflections

148 parameters

1 restraint

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.32$ e Å^{-3}

$\Delta\rho_{min} = -0.35$ e Å^{-3}

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Experimental. We have re-refined our data by using 'MERG 1' instruction to avoid Friedel opposites being merged. The absolute structure parameter is still meaningless, though the data/parameter (985/148) is higher than the former (895/148).

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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
Cl	−0.0425 (4)	0.7267 (4)	0.74675 (17)	0.0628 (8)
C1	0.3337 (10)	0.6719 (8)	0.4546 (5)	0.0297 (14)
N1	0.2032 (8)	0.7163 (9)	0.1907 (4)	0.0333 (13)
H1	0.0860	0.6715	0.2071	0.06 (3)*
O1	0.7621 (7)	0.6067 (9)	0.2131 (5)	0.0529 (16)
C2	0.1323 (11)	0.7587 (9)	0.4407 (6)	0.0360 (16)
H2	0.0726	0.8045	0.3688	0.043*
O2	0.4735 (8)	0.5437 (10)	0.0782 (5)	0.0579 (17)
H2A	0.5657	0.5193	0.0393	0.087*
C3	0.0201 (13)	0.7786 (10)	0.5294 (6)	0.0422 (18)
H3	−0.1134	0.8368	0.5177	0.051*
O3	0.3510 (8)	0.9179 (9)	0.0890 (4)	0.0496 (15)
C4	0.1089 (13)	0.7102 (11)	0.6378 (6)	0.0436 (18)
C5	0.3095 (13)	0.6317 (12)	0.6558 (6)	0.0480 (19)
H5	0.3701	0.5915	0.7291	0.058*
C6	0.4220 (11)	0.6116 (11)	0.5683 (6)	0.0424 (18)
H6	0.5582	0.5577	0.5825	0.051*
C7	0.4613 (10)	0.6378 (10)	0.3662 (6)	0.0337 (14)
H7	0.6012	0.5950	0.3924	0.040*
C8	0.4067 (10)	0.6591 (10)	0.2525 (6)	0.0352 (15)
C9	0.5701 (10)	0.6022 (10)	0.1796 (6)	0.0372 (16)
C10	0.1874 (11)	0.8392 (10)	0.1065 (5)	0.0363 (15)
C11	−0.0380 (12)	0.8731 (13)	0.0403 (6)	0.050 (2)
H11A	−0.0295	0.9462	−0.0252	0.075*
H11B	−0.1227	0.9323	0.0891	0.075*
H11C	−0.1059	0.7624	0.0149	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0756 (15)	0.0751 (15)	0.0438 (10)	−0.0037 (13)	0.0266 (10)	−0.0043 (11)
C1	0.028 (3)	0.032 (3)	0.026 (3)	−0.003 (3)	−0.001 (2)	0.002 (2)
N1	0.027 (3)	0.045 (3)	0.032 (2)	0.000 (3)	0.016 (2)	0.004 (3)
O1	0.025 (2)	0.077 (4)	0.057 (3)	0.003 (3)	0.005 (2)	0.000 (3)
C2	0.041 (4)	0.033 (4)	0.034 (3)	0.008 (3)	0.006 (3)	0.004 (3)
O2	0.033 (2)	0.084 (4)	0.058 (3)	−0.001 (3)	0.012 (2)	−0.034 (4)
C3	0.037 (4)	0.046 (4)	0.045 (4)	0.004 (3)	0.011 (3)	0.000 (3)
O3	0.047 (3)	0.063 (4)	0.041 (3)	−0.013 (3)	0.015 (2)	0.014 (3)
C4	0.051 (4)	0.038 (4)	0.040 (4)	−0.008 (4)	0.003 (3)	−0.002 (3)
C5	0.059 (5)	0.054 (5)	0.030 (3)	−0.005 (4)	0.005 (3)	0.006 (3)
C6	0.038 (4)	0.046 (4)	0.039 (4)	0.012 (3)	−0.004 (3)	0.008 (3)
C7	0.027 (3)	0.035 (3)	0.040 (3)	0.002 (3)	0.006 (2)	0.002 (3)
C8	0.029 (3)	0.034 (3)	0.045 (4)	0.000 (3)	0.014 (3)	0.003 (3)
C9	0.033 (3)	0.044 (4)	0.041 (4)	−0.004 (3)	0.022 (3)	−0.002 (3)
C10	0.040 (3)	0.044 (4)	0.029 (3)	0.005 (3)	0.016 (3)	−0.003 (3)
C11	0.054 (4)	0.057 (5)	0.034 (3)	0.011 (4)	−0.006 (3)	0.006 (4)

Geometric parameters (Å, °)

Cl—C4	1.734 (8)	C3—H3	0.9300
C1—C2	1.401 (9)	O3—C10	1.231 (9)
C1—C6	1.430 (9)	C4—C5	1.367 (12)
C1—C7	1.446 (9)	C5—C6	1.360 (11)
N1—C10	1.350 (9)	C5—H5	0.9300
N1—C8	1.413 (9)	C6—H6	0.9300
N1—H1	0.8600	C7—C8	1.334 (11)
O1—C9	1.193 (8)	C7—H7	0.9300
C2—C3	1.370 (11)	C8—C9	1.512 (8)
C2—H2	0.9300	C10—C11	1.502 (10)
O2—C9	1.316 (9)	C11—H11A	0.9600
O2—H2A	0.8200	C11—H11B	0.9600
C3—C4	1.398 (11)	C11—H11C	0.9600
C2—C1—C6	116.3 (6)	C5—C6—H6	119.5
C2—C1—C7	126.8 (6)	C1—C6—H6	119.5
C6—C1—C7	116.9 (6)	C8—C7—C1	129.2 (6)
C10—N1—C8	121.9 (6)	C8—C7—H7	115.4
C10—N1—H1	119.0	C1—C7—H7	115.4
C8—N1—H1	119.0	C7—C8—N1	126.8 (6)
C3—C2—C1	122.3 (6)	C7—C8—C9	117.6 (6)
C3—C2—H2	118.8	N1—C8—C9	115.4 (6)
C1—C2—H2	118.8	O1—C9—O2	125.3 (6)
C9—O2—H2A	109.5	O1—C9—C8	123.0 (6)
C4—C3—C2	119.2 (7)	O2—C9—C8	111.7 (5)
C4—C3—H3	120.4	O3—C10—N1	120.2 (6)

C2—C3—H3	120.4	O3—C10—C11	123.9 (7)
C3—C4—C5	120.1 (7)	N1—C10—C11	115.9 (6)
C3—C4—Cl	118.3 (6)	C10—C11—H11A	109.5
C5—C4—Cl	121.6 (6)	C10—C11—H11B	109.5
C4—C5—C6	121.0 (7)	H11A—C11—H11B	109.5
C4—C5—H5	119.5	C10—C11—H11C	109.5
C6—C5—H5	119.5	H11A—C11—H11C	109.5
C5—C6—C1	121.0 (6)	H11B—C11—H11C	109.5

C6—C1—C2—C3	−2.8 (10)	C6—C1—C7—C8	170.8 (8)
C7—C1—C2—C3	177.5 (7)	C1—C7—C8—N1	−2.2 (13)
C1—C2—C3—C4	0.0 (11)	C1—C7—C8—C9	−176.5 (7)
C2—C3—C4—C5	2.9 (12)	C10—N1—C8—C7	134.8 (8)
C2—C3—C4—Cl	−176.7 (6)	C10—N1—C8—C9	−50.8 (9)
C3—C4—C5—C6	−2.9 (13)	C7—C8—C9—O1	−30.5 (11)
Cl—C4—C5—C6	176.6 (6)	N1—C8—C9—O1	154.6 (8)
C4—C5—C6—C1	0.0 (12)	C7—C8—C9—O2	147.7 (8)
C2—C1—C6—C5	2.8 (11)	N1—C8—C9—O2	−27.2 (10)
C7—C1—C6—C5	−177.5 (7)	C8—N1—C10—O3	−7.2 (11)
C2—C1—C7—C8	−9.5 (12)	C8—N1—C10—C11	174.0 (6)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.09	2.933 (7)	165
O2—H2A···O3^{ii}	0.82	1.86	2.606 (7)	152
C3—H3···Cg1^{iii}	0.93	2.85	3.523 (8)	130

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

Fig. 1

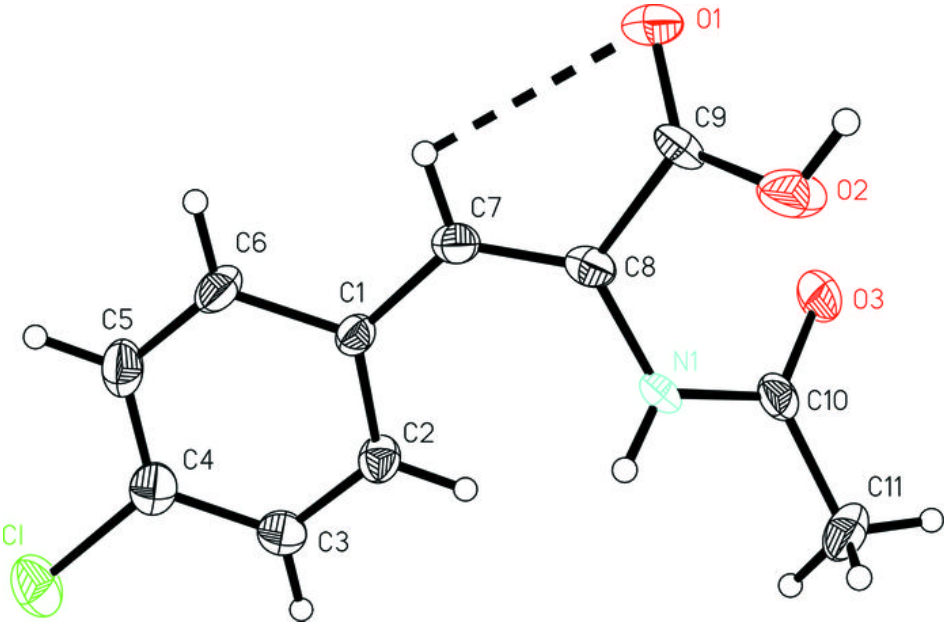

Fig. 2