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Methyl 2-(1,3-dioxoisoindolin-2-yl)-acrylate

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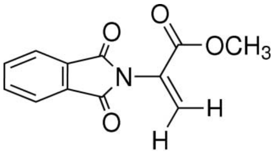
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 12.7.

In the title compound, $C_{12}H_9NO_4$, an important dehydroamino acid, the acrylate C=C double bond is not parallel to the adjacent carbonyl group and an *s-trans* configuration is also observed.

Related literature

For related literature, see: Cativiela *et al.* (2000); Clausen *et al.* (2002); Schmidt *et al.* (1988); Trost & Dake, (1997); Wirth (1997); Osborn *et al.* (1966). For related structures, see: Ajò *et al.* (1984; 1979); Busetti *et al.* (1984; 1986).



Experimental

Crystal data

$C_{12}H_9NO_4$
M_r = 231.20
Triclinic, $P\overline{1}$
a = 6.5179 (3) Å
b = 7.4817 (4) Å
c = 11.7322 (6) Å
α = 80.954 (2)°
β = 78.866 (2)°
γ = 76.723 (2)°
V = 542.52 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.20 × 0.18 × 0.15 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.984
2932 measured reflections
1984 independent reflections
1497 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.04
1984 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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: HG2354).

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supplementary materials

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Methyl 2-(1,3-dioxoisoindolin-2-yl)acrylate

Y.-W. Wang and **Y. Peng**

Comment

Optical active nonproteinogenic amino acids (Clausen *et al.*, 2002) are valuable compounds of high interest not only due to their remarkable pharmacological and biological activities but also for their role as a topographic probe for investigation of bioactive conformations of peptides and the mechanisms of enzyme reactions. Consequently, efficient and convenient methods for the preparation of optically pure enantiomers of amino acids are be general interest (Cativiela & Diaz-de-Villegas, 2000; Wirth, 1997). It is noteworthy that extraordinary progress has been made in the asymmetric hydrogenation of dehydroamino acids (Osborn *et al.*, 1966; Schmidt *et al.*, 1988), which serve as important precursors of saturated ones.

The title compound belong to one of dehydroamino acids mentioned above. Its molecular structure is shown in Fig.1 and can be prepared conveniently through nucleophilic addition of phthalimide to propiolate according to the precedent procedure (Trost & Dake, 1997). The exceptionally large difference ($\Delta\ \delta$= 0.68 p.p.m.) between the chemical shifts of the two protons (H_a and H_b) on the double bond suggest that the chemical environments (*e.g.*, deshielding effect of the phthalimidyl group) of them are considerably different. As shown in Fig. 1, An s-*trans* conformation is observed, which was discussed in detail other groups previously (Ajò *et al.*, 1984; Ajò *et al.*, 1979; Busetti, *et al.*, 1984; Busetti, *et al.*, 1986). The double bond (C9–C12) is not parallel with the carbonyl (C10–O1) group with the C12-C9_C10-O1 torsion angle −142.3 (2)° while the double bond (C9–C12) lies out of the plane of the phthalimidyl group with the torsion angle C7—N1—C9—C12 44.7 (3)°.

Experimental

To a solution of phthalimide (740 mg, 5 mmol), triphenylphosphine (130 mg, 0.5 mmol) and sodium acetate (210 mg, 2.5 mmol) in 10 ml of toluene at 378 K were added sequentially acetic acid (0.14 ml, 2.5 mmol) and methyl propiolate (420 mg, 5 mmol). After 18 h, the reaction mixture was cooled and directly subjected to chromatograph on silica gel (Hexane: EtOAc = 2: 1) to yield 928 mg (80% yield) of the title compound. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 7.94–7.90 (m, 2H), 7.83–7.77 (m, 2H), 6.69 (s, 1H), 6.01 (s, 1H), 3.82 (s, 3H) p.p.m.; ^{13}C NMR (50 MHz, $CDCl_3$): δ = 166.2, 162.6, 134.2 (2 C), 131.7, 129.0, 127.9 (2 C), 123.8 (2 C), 52.7 p.p.m.. Single crystals suitable for X-ray determination were obtained by slow evaporation of a EtOAc solution over a period of several days.

Refinement

All H atoms were placed geometrically (C—H values were set to 0.96 and 0.93 Å for atoms CH_3, CH_2, and CH (phenyl), respectively) and refined with a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Figures

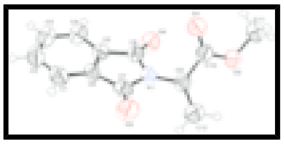

Fig. 1. The independent components of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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Crystal data

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$M_r = 231.20$
Triclinic, $P\bar{1}$
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$a = 6.5179$ (3) Å
$b = 7.4817$ (4) Å
$c = 11.7322$ (6) Å
$\alpha = 80.954$ (2)°
$\beta = 78.866$ (2)°
$\gamma = 76.723$ (2)°
$V = 542.52$ (5) Å^3

$Z = 2$
$F_{000} = 240$
$D_x = 1.415$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 1079 reflections
$\theta = 2.8$–23.7°
$\mu = 0.11$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.20 × 0.18 × 0.15 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 294(2)$ K
phi and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{\min} = 0.979$, $T_{\max} = 0.984$
2932 measured reflections

1984 independent reflections
1497 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.016$
$\theta_{\max} = 25.5$°
$\theta_{\min} = 1.8$°
$h = -6 \rightarrow 7$
$k = -9 \rightarrow 8$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.114$
$S = 1.04$
1984 reflections

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.1136P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

156 parameters	Extinction correction: SHELXL, $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.044 (7)
Secondary atom site location: difference Fourier map	

Special details

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*	$U_{iso}*/U_{eq}$
C5	0.3823 (3)	0.8001 (2)	0.03710 (15)	0.0475 (4)
C6	0.5949 (3)	0.7126 (2)	0.02643 (15)	0.0471 (4)
C1	0.7069 (3)	0.6511 (3)	−0.07723 (18)	0.0594 (5)
H1	0.8502	0.5927	−0.0841	0.071*
C2	0.5971 (4)	0.6799 (3)	−0.17070 (18)	0.0667 (6)
H2	0.6677	0.6396	−0.2418	0.080*
C4	0.2737 (3)	0.8285 (3)	−0.05580 (18)	0.0618 (5)
H4	0.1303	0.8869	−0.0489	0.074*
C3	0.3854 (4)	0.7671 (3)	−0.15998 (18)	0.0673 (6)
H3	0.3157	0.7852	−0.2242	0.081*
C7	0.6660 (3)	0.6964 (3)	0.14049 (16)	0.0503 (5)
C8	0.3091 (3)	0.8473 (2)	0.15854 (16)	0.0485 (4)
N1	0.4864 (2)	0.7786 (2)	0.21650 (12)	0.0483 (4)
C9	0.4784 (3)	0.7876 (3)	0.33739 (16)	0.0512 (5)
C10	0.2984 (3)	0.7159 (3)	0.41433 (16)	0.0560 (5)
C11	0.0631 (4)	0.7296 (4)	0.59378 (18)	0.0740 (7)
H11A	−0.0473	0.7213	0.5525	0.111*
H11B	0.0054	0.8140	0.6513	0.111*
H11C	0.1182	0.6097	0.6318	0.111*
O2	0.2339 (2)	0.7960 (2)	0.51181 (11)	0.0633 (4)
O1	0.2230 (3)	0.5971 (3)	0.39155 (13)	0.0908 (6)
O4	0.1388 (2)	0.9302 (2)	0.20287 (13)	0.0667 (4)
O3	0.8377 (2)	0.6270 (2)	0.16799 (13)	0.0735 (5)
C12	0.6314 (3)	0.8332 (3)	0.3783 (2)	0.0702 (6)
H12A	0.7511	0.8612	0.3274	0.084*
H12B	0.6198	0.8374	0.4582	0.084*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.0503 (10)	0.0393 (9)	0.0514 (10)	−0.0052 (8)	−0.0077 (8)	−0.0076 (8)
C6	0.0462 (10)	0.0416 (10)	0.0507 (10)	−0.0090 (8)	−0.0008 (8)	−0.0063 (8)

C1	0.0592 (12)	0.0547 (12)	0.0595 (12)	−0.0115 (9)	0.0063 (10)	−0.0126 (9)
C2	0.0907 (17)	0.0596 (13)	0.0482 (12)	−0.0221 (12)	0.0054 (11)	−0.0126 (9)
C4	0.0660 (13)	0.0545 (12)	0.0644 (13)	−0.0011 (10)	−0.0197 (10)	−0.0114 (10)
C3	0.0923 (17)	0.0585 (13)	0.0547 (12)	−0.0146 (12)	−0.0202 (11)	−0.0083 (10)
C7	0.0414 (10)	0.0476 (10)	0.0584 (11)	−0.0055 (8)	−0.0031 (8)	−0.0081 (8)
C8	0.0426 (10)	0.0459 (10)	0.0554 (11)	−0.0051 (8)	−0.0056 (8)	−0.0103 (8)
N1	0.0416 (8)	0.0536 (9)	0.0472 (9)	−0.0035 (7)	−0.0052 (7)	−0.0102 (7)
C9	0.0530 (11)	0.0514 (11)	0.0500 (11)	−0.0085 (8)	−0.0091 (8)	−0.0102 (8)
C10	0.0640 (12)	0.0582 (12)	0.0486 (11)	−0.0144 (10)	−0.0094 (9)	−0.0116 (9)
C11	0.0720 (14)	0.1010 (19)	0.0505 (12)	−0.0271 (13)	0.0016 (10)	−0.0143 (11)
O2	0.0649 (9)	0.0775 (10)	0.0517 (8)	−0.0216 (7)	−0.0003 (6)	−0.0216 (7)
O1	0.1201 (14)	0.1023 (13)	0.0672 (10)	−0.0674 (12)	0.0164 (9)	−0.0346 (9)
O4	0.0460 (8)	0.0774 (10)	0.0729 (9)	0.0052 (7)	−0.0061 (7)	−0.0287 (7)
O3	0.0454 (8)	0.0910 (11)	0.0790 (10)	0.0080 (7)	−0.0149 (7)	−0.0214 (8)
C12	0.0627 (13)	0.0895 (17)	0.0636 (13)	−0.0207 (12)	−0.0133 (10)	−0.0135 (11)

Geometric parameters (Å, °)

C5—C4	1.375 (3)	C8—O4	1.204 (2)
C5—C6	1.381 (2)	C8—N1	1.408 (2)
C5—C8	1.483 (3)	N1—C9	1.421 (2)
C6—C1	1.379 (3)	C9—C12	1.317 (3)
C6—C7	1.477 (3)	C9—C10	1.485 (3)
C1—C2	1.385 (3)	C10—O1	1.196 (2)
C1—H1	0.9300	C10—O2	1.326 (2)
C2—C3	1.375 (3)	C11—O2	1.449 (2)
C2—H2	0.9300	C11—H11A	0.9600
C4—C3	1.384 (3)	C11—H11B	0.9600
C4—H4	0.9300	C11—H11C	0.9600
C3—H3	0.9300	C12—H12A	0.9300
C7—O3	1.203 (2)	C12—H12B	0.9300
C7—N1	1.408 (2)		
C4—C5—C6	120.94 (17)	O4—C8—C5	129.91 (17)
C4—C5—C8	130.64 (17)	N1—C8—C5	105.68 (14)
C6—C5—C8	108.42 (15)	C8—N1—C7	111.36 (15)
C1—C6—C5	121.66 (18)	C8—N1—C9	123.22 (15)
C1—C6—C7	129.53 (17)	C7—N1—C9	125.39 (15)
C5—C6—C7	108.79 (15)	C12—C9—N1	122.78 (18)
C6—C1—C2	117.30 (19)	C12—C9—C10	122.85 (18)
C6—C1—H1	121.3	N1—C9—C10	113.85 (15)
C2—C1—H1	121.3	O1—C10—O2	123.94 (19)
C3—C2—C1	120.99 (19)	O1—C10—C9	123.63 (18)
C3—C2—H2	119.5	O2—C10—C9	112.41 (16)
C1—C2—H2	119.5	O2—C11—H11A	109.5
C5—C4—C3	117.59 (19)	O2—C11—H11B	109.5
C5—C4—H4	121.2	H11A—C11—H11B	109.5
C3—C4—H4	121.2	O2—C11—H11C	109.5
C2—C3—C4	121.5 (2)	H11A—C11—H11C	109.5
C2—C3—H3	119.2	H11B—C11—H11C	109.5

C4—C3—H3	119.2	C10—O2—C11	116.00 (16)
O3—C7—N1	124.79 (18)	C9—C12—H12A	120.0
O3—C7—C6	129.47 (18)	C9—C12—H12B	120.0
N1—C7—C6	105.73 (15)	H12A—C12—H12B	120.0
O4—C8—N1	124.39 (17)		

C4—C5—C6—C1	0.3 (3)	O4—C8—N1—C7	−176.95 (18)
C8—C5—C6—C1	179.59 (16)	C5—C8—N1—C7	1.5 (2)
C4—C5—C6—C7	−178.18 (16)	O4—C8—N1—C9	5.0 (3)
C8—C5—C6—C7	1.11 (19)	C5—C8—N1—C9	−176.60 (15)
C5—C6—C1—C2	−0.3 (3)	O3—C7—N1—C8	−179.88 (18)
C7—C6—C1—C2	177.86 (18)	C6—C7—N1—C8	−0.8 (2)
C6—C1—C2—C3	0.3 (3)	O3—C7—N1—C9	−1.9 (3)
C6—C5—C4—C3	−0.3 (3)	C6—C7—N1—C9	177.20 (15)
C8—C5—C4—C3	−179.41 (19)	C8—N1—C9—C12	−137.6 (2)
C1—C2—C3—C4	−0.3 (3)	C7—N1—C9—C12	44.7 (3)
C5—C4—C3—C2	0.3 (3)	C8—N1—C9—C10	50.6 (2)
C1—C6—C7—O3	0.5 (3)	C7—N1—C9—C10	−127.22 (19)
C5—C6—C7—O3	178.8 (2)	C12—C9—C10—O1	−142.1 (2)
C1—C6—C7—N1	−178.55 (18)	N1—C9—C10—O1	29.7 (3)
C5—C6—C7—N1	−0.22 (19)	C12—C9—C10—O2	36.7 (3)
C4—C5—C8—O4	−4.1 (3)	N1—C9—C10—O2	−151.46 (16)
C6—C5—C8—O4	176.72 (19)	O1—C10—O2—C11	0.8 (3)
C4—C5—C8—N1	177.62 (18)	C9—C10—O2—C11	−178.04 (17)
C6—C5—C8—N1	−1.57 (19)		

Fig. 1