Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(*Z*)-3-Acetonylidene-2,3-dihydro-1*H*-isoindolin-1-one

Krishna Chowdhury,[a] Rupa Mukhopadhyay,[b] Monika Mukherjee,[a]* Charlotte K. Broder[c] and Nitya G. Kundu[b]

[a]Department of Solid State Physics, Indian Association for the Cultivation of Science, Jadavpur, Calcutta 700 032, India, [b]Department of Organic Chemistry, Indian Association for the Cultivation of Science, Jadavpur, Calcutta 700 032, India, and [c]Department of Chemistry, University of Durham, Durham DH1 3LE, England

Correspondence e-mail:
sspmm@mahendra.iacs.res.in

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.121
Data-to-parameter ratio = 19.1

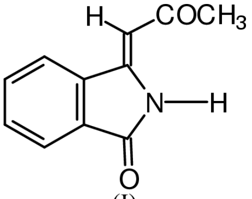
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 March 2001
Accepted 4 April 2001
Online 12 April 2001

The asymmetric unit of the title compound, $C_{11}H_9NO_2$, contains two molecules which are related by a pseudo-inversion centre and form an N—H···O hydrogen-bonded dimer. The least-squares planes through the non-H atoms of the molecules illustrate the overall molecular planarity. The molecules assume *Z* configurations about the exocyclic C═C bond. In the crystal, intermolecular hydrogen bonds link the molecules into infinite chains.

Comment

The isoindolinone skeleton is present in a number of natural products (Tamaoki *et al.*, 1986; Valencia *et al.*, 1985). Many isoindolinone derivatives exhibit biological activity as potential antiviral, antileukemic, anti-inflammatory, antipsychotic and antiulcer agents (Zhuang *et al.*, 1998; Taylor *et al.*, 1997; De Clercq, 1995; Pendrak *et al.*, 1994). As part of our ongoing studies on the synthesis and characterization of new heterocyclic systems containing an isoindolinone moiety (Mukherjee *et al.*, 2000; Kundu, Khan & Mukhopadhyay, 1999), the structure determination of (*Z*)-3-acetonylidene-2,3-dihydro-1*H*-isoindolin-1-one, (I), was undertaken.



(I)

The two approximately parallel molecules (*A* and *B*) in the asymmetric unit related by a pseudo-inversion centre at (0.50, 0.38, 0.25), can be described as an N—H···O bonded dimer (Fig. 1). The dihedral angle between the nearly planar molecules *A* and *B* is 2.45 (2)°. The Z configuration of the molecules is established by the N1—C8—C9—C10 torsion angles of −4.6 (2)° in *A* and 4.3 (2)° in *B*. The angles between the five- and six-membered rings of the isoindolinone system in *A* and *B* are 1.92 (6) and 1.54 (6)°, respectively. The bond distances and angles in (I) are similar to those reported for substituted isoindoline structures (Kundu, Khan, Guha & Mukherjee, 1999; Khan *et al.*, 1998; McNab *et al.*, 1997). In the solid state, weak intermolecular C9—H9···O1 hydrogen bonds link the dimers to form infinite chains which are cross-linked in the *c* direction by weak intermolecular C4*B*—H4*B*···O2*A* hydrogen bonds (Table 2), to form a network.

DOI: 101107/S1600536801005864

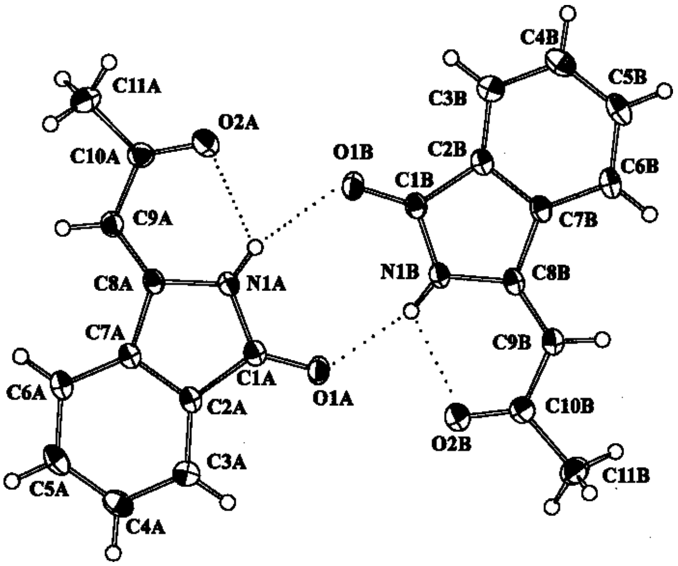


Figure 1

ZORTEP view (50% probability level) of the two hydrogen-bonded molecules of (I).

Experimental

2-[(Trimethylsilyl)ethynyl]benzamide (TEB) was prepared by stirring a mixture of *o*-iodobenzamide (1 mmol), (trimethylsilyl)acetylene (2 mmol), $(PPh_3)_2PdCl_2$ (3.5 mol%), CuI (8 mol%) and triethylamine (4 mmol) in dimethylformamide at room temperature under a nitrogen atmosphere for 24 h. The reaction of TEB (1 mmol) with CH_3COCl (acetyl chloride, 1.2 mmol) in the presence of anhydrous $AlCl_3$ (4 mmol) and tetrachloroethane (10 ml) at 273 K yielded the title compound, (I) (m.p. 381 K). Single crystals suitable for X-ray analysis were obtained by slow crystallization from a dilute solution of (I) in ethanol. Elemental analysis, calculated for $C_{11}H_9NO_2$: C 70.58, H 4.81, N 7.48%; found: C 70.53, H 4.89, N 7.24%.

Crystal data

$C_{11}H_9NO_2$
$M_r = 187.19$
Monoclinic, $P2_1/n$
$a = 6.8673$ (2) Å
$b = 12.8243$ (3) Å
$c = 20.6130$ (5) Å
$\beta = 94.520$ (1)°
$V = 1809.71$ (8) Å^3
$Z = 8$
$D_x = 1.374$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 922 reflections
$\theta = 5.4$–31.7°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Hexagonal block, colourless
0.50 × 0.45 × 0.30 mm

Data collection

Bruker SMART–CCD diffractometer
ω scans
14 180 measured reflections
4883 independent reflections
4172 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\rm max} = 29.5°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.121$
$S = 1.05$
4883 reflections
255 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 0.6557P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

N1*A*—C1*A*	1.3842 (15)	N1*B*—C1*B*	1.3863 (15)
N1*A*—C8*A*	1.3931 (14)	N1*B*—C8*B*	1.3923 (13)
C7*A*—C8*A*	1.4768 (15)	C7*B*—C8*B*	1.4757 (15)
C8*A*—C9*A*	1.3513 (16)	C8*B*—C9*B*	1.3512 (16)
C9*A*—C10*A*	1.4682 (16)	C9*B*—C10*B*	1.4682 (16)
C1*A*—N1*A*—C8*A*	112.57 (9)	C1*B*—N1*B*—C8*B*	112.68 (9)
C9*A*—C8*A*—N1*A*	126.67 (10)	C9*B*—C8*B*—N1*B*	126.87 (11)
C9*A*—C8*A*—C7*A*	127.24 (10)	C9*B*—C8*B*—C7*B*	127.19 (10)
N1*A*—C8*A*—C7*A*	105.98 (9)	N1*B*—C8*B*—C7*B*	105.90 (9)
C8*A*—C9*A*—C10*A*	123.37 (11)	C8*B*—C9*B*—C10*B*	123.66 (11)
N1*A*—C8*A*—C9*A*—C10*A*	−4.56 (18)	N1*B*—C8*B*—C9*B*—C10*B*	4.26 (18)
C7*A*—C8*A*—C9*A*—C10*A*	171.14 (10)	C7*B*—C8*B*—C9*B*—C10*B*	−172.90 (10)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1*A*—H1*A*···O2*A*	0.86	2.25	2.776 (1)	120
N1*A*—H1*A*···O1*B*	0.86	2.38	3.065 (1)	137
N1*B*—H1*B*···O1*A*	0.86	2.30	2.977 (1)	135
N1*B*—H1*B*···O2*B*	0.86	2.26	2.787 (1)	120
C9*A*—H9*A*···O1*A*$^{\rm i}$	0.93	2.49	3.208 (1)	135
C4*B*—H4*B*···O2*A*$^{\rm ii}$	0.93	2.58	3.488 (2)	167
C9*B*—H9*B*···O1*B*$^{\rm iii}$	0.93	2.57	3.283 (1)	134

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

The H atoms were refined using a riding model and their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameters of their parent atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1995); software used to prepare material for publication: *SHELXL*97).

The authors wish to thank Professor J. A. K. Howard, University of Durham, for XRD data collection. Grateful acknowledgement is made for a CSIR fellowship to Rupa Mukhopadhyay.

References

Debaerdemaeker, T., Germain, G., Main, P., Refaat, L. S., Tate, C. & Woolfson, M. M. (1988). *MULTAN*88. Universities of York, England, and Louvain, Belgium.

De Clercq, E. (1995). *J. Med. Chem.* **38**, 2491–2517.

Khan, M. W., Guha, S., Mukherjee, A. K. & Kundu, N. G. (1998). *Acta Cryst.* C**54**, 119–120.

Kundu, N. G., Khan, M. W., Guha, S. & Mukherjee, A. K. (1999). *Acta Cryst.* C**55**, 239–241.

Kundu, N. G., Khan, M. W. & Mukhopadhyay, R. (1999). *Tetrahedron*, **55**, 12361–12376.

McNab, H., Parsons, S. & Shannon, D. A. (1997). *Acta Cryst.* C**53**, 1098–1099.

Mukherjee, A. K., Guha, S., Khan, M. W., Kundu, N. G. & Helliwell, M. (2000). *Acta Cryst.* C**56**, 85–87.

Pendrak, I., Barney, S., Wittrock, R., Lambert, D. M., Kingsbury, W. D. (1994). *J. Org. Chem.* **59**, 2623–2625.

Siemens (1996). *SMART* and *SAINT*. Siemens AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Tamaoki, T., Nomoto, H., Takahashi, I., Kato, Y., Morimoto, M. & Tomita, F. (1986). *Biochem. Biophys. Res. Commun.* **135**, 397–398.

Taylor, E. C., Zhou, P., Jennings, L. D., Mao, Z., Hu, B. & Jun. J.-G. (1997). *Tetrahedron Lett.* **38**, 521–524.

Valencia, E., Fajardo, V., Freyer, A. J. & Shamma, M. (1985). *Tetrahedron Lett.* **26**, 993–996.

Zhuang, Z.-P., Kung, M.-P., Mu, M. & Kung, H. F. (1998). *J. Med. Chem.* **41**, 157–166.

Zsolnai, L. (1995). *ZORTEP*. University of Heidelberg, Germany.