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(*E*)-3-(Benzoylmethylene)-*N*-methylisoindolin-1-one

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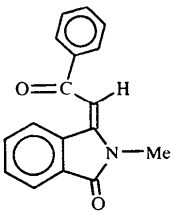
Abstract

In the title compound, $C_{17}H_{13}NO_2$, the molecules contain planar isoindolinone and phenyl moieties, and have the *E* configuration. The phenyl ring is twisted out of the isoindolinone plane by 44.6 (1)°. The structure is stabilized by hydrogen bonding and van der Waals interactions.

Comment

The isoindoline system is present in a number of natural products, many of which are found to possess biological activity (Zhuang *et al.*, 1998; Belliotti *et al.*, 1998). As part of our ongoing studies on the synthesis and characterization of new heterocyclic systems containing an isoindolinone moiety (Khan & Kundu, 1997; Khan *et al.*, 1998), and to build up a hierarchy for such systems, the structure determination of (*E*)-3-(benzoylmethylene)-*N*-methylisoindolin-1-one, (I), was undertaken.



(I)

The results of the present X-ray analysis are in agreement with those reported for other substituted isoindolinone structures (Khan *et al.*, 1998; Kundu *et al.*, 1999; McNab *et al.*, 1997; Feeder & Jones, 1996; Barrett *et al.*, 1996). The *E* configuration of the molecule is established by the torsion angle N—C9—C10—C11 of 177.3 (3)°. The maximum deviation from planarity in the isoindolinone system is 0.034 (4) Å. Intermolecular C—H···O hydrogen bonds (Table 2) stabilize the crystal packing.

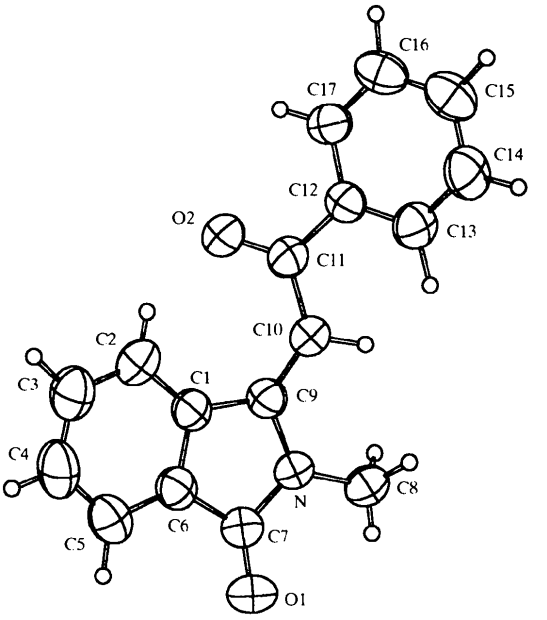


Fig. 1. *ZORTEP* (Zsolnai, 1995) view (50% probability level) of the title compound.

Experimental

2-(Trimethylsilyl)ethynyl-*N*-methylbenzamide was prepared by stirring a mixture of *o*-iodo-*N*-methylbenzamide (1 mmol), (trimethylsilyl)acetylene (2 mmol), $(PPh_3)_2PdCl_2$ (3.5 mol%), cuprous iodide (8 mol%) and trimethylamine (4 mmol) in dimethylformamide at room temperature under a nitrogen atmosphere for 24 h. Reaction with anhydrous aluminium chloride (1.2 equivalents) in tetrachloroethane at 273 K yielded the title compound [m.p. 388 (1) K]. Single crystals suitable for X-ray analysis were obtained by slow crystallization from a dilute solution in ethanol.

Crystal data

$C_{17}H_{13}NO_2$
M_r = 263.28
Orthorhombic
$P2_12_12_1$
a = 9.033 (1) Å
b = 11.081 (1) Å
c = 13.447 (1) Å
V = 1346.0 (2) Å^3
Z = 4
D_x = 1.299 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 12–18°
μ = 0.086 mm^{-1}
T = 293 (2) K
Prism
0.40 × 0.30 × 0.25 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (North *et al.*, 1968)
T_{min} = 0.969, T_{max} = 0.978
1342 measured reflections
1342 independent reflections
965 reflections with $I > 2\sigma(I)$
θ_{max} = 24.97°
h = 0 → 10
k = 0 → 13
l = 0 → 15
3 standard reflections
every 100 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.096$
$S = 0.968$
1342 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0786P)^2 + 0.0649P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.139$ e Å^{-3}
$\Delta\rho_{min} = -0.153$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N—C7	1.377 (4)	C1—C9	1.468 (4)
N—C9	1.401 (4)	C6—C7	1.471 (4)
N—C8	1.452 (4)	C9—C10	1.343 (4)
O1—C7	1.212 (4)	C10—C11	1.475 (4)
O2—C11	1.224 (4)	C11—C12	1.481 (4)
C7—N—C9	112.5 (2)	N—C7—C6	105.7 (2)
C9—N—C8	124.9 (2)	N—C9—C1	105.5 (2)
C6—C1—C9	108.0 (2)	C9—C10—C11	126.3 (3)
C1—C6—C7	108.4 (3)	C10—C11—C12	118.6 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O2^{i}	3.441 (4)	164.2 (2)
C8—H8*B*···O2^{i}	3.301 (4)	117.9 (2)
C8—H8*C*···O1^{ii}	3.453 (4)	126.4 (2)
C16—H16···O1^{iii}	3.364 (4)	122.6 (2)
C17—H17···O1^{iii}	3.222 (4)	126.5 (2)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *MULTAN*88 (Debaerdemaeker *et al.*, 1988). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1246). Services for accessing these data are described at the back of the journal.

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2*H*-Dibenzo[*b*,*f*]azepin-2-one

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Abstract

It has been postulated that the title compound, $C_{14}H_9NO$, (1*a*), may play a role in idiosyncratic reactions induced by carbamazepine, one of the most widely used anticonvulsants in North America. Compound (1*a*) was found to be nearly planar, unlike its parent compound iminostilbene. The possibility of the azepinone having a resonance contribution from the novel nitrenium ion was investigated. The slightly elongated C═N and C═O bond lengths suggest that there is some contribution from the nitrenium structure. However, the alternating bond lengths show that the major resonance contributor is the iminoquinone.

Comment

Nitrenium ions, (1*b*), are of considerable biological importance. Reactions proceeding *via* such intermediates have been investigated extensively, especially as models for processes involved in carcinogenesis by aromatic amines (McClelland, 1996). The actual cellular target is often DNA and indeed covalent adducts of guanine residues have been observed *in vivo* (Schut & Castongauy, 1984), as well as in model systems (Underwood *et al.*, 1988). The molecule under investigation in this work, (1*a*), is believed to be an active metabolite as-