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2,3-Dihydro-3-phenyl-1*H*-isoindol-1-one at 220 K

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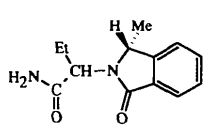
Abstract

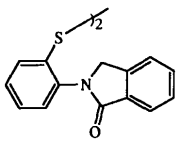
The title compound, $C_{14}H_{11}NO$, forms hydrogen-bonded dimers in the solid state; its geometry correlates well with those of related molecules.

Comment

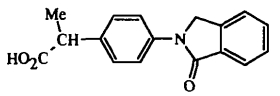
In the structural literature of the 2,3-dihydroisoindol-1-one (phthalimidine) system the majority of simple examples are either *N*-substituted [(1)–(3); Carlström, Hacksell, Jönsson & Söderholm, 1983; Fawcett, Kemmitt, Russell, Serindag & Gok, 1993; Ravikumar, 1994], or have a hydroxy substituent at the 3-position [(4) and (5); Ohrt, Tsoucaris-Kupfer & Lechat, 1978; Rodier, Martin, Miocque, Mettey & Vierfond, 1988]. We report here the crystal structure of the 3-phenyl derivative, (6), to serve as a model for the family of *N*-unsubstituted 3-aryl derivatives.



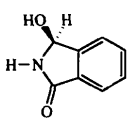
(1)



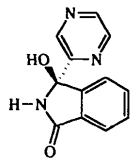
(2)



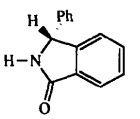
(3)



(4)



(5)



(6)

Bond lengths and angles of the five-membered ring of (6) are effectively identical with those of the other *N*-unsubstituted derivatives (4) and (5), and so substitution at C3 apparently has little influence. *N*-alkyl, (1), and particularly *N*-aryl, (2) and (3), derivatives are also very similar to (6), except for C1—N2 and N2—C3, which are generally longer [for example, in (3), C1—N2 is 1.486 (7) and N2—C3 is 1.391 (7) Å]. There appears to be little variation in the bond angles in the five-membered rings of all six structures, while in the fused six-membered ring the angles at C4 and C7 are both less than 118°, an effect previously noted for (1). The maximum deviations from planarity in the five- and six-membered rings of the isoindole system are 0.007 (1) and 0.001 (1) Å, respectively, and the angle between these planes is 1.59 (11)° [*cf.* 2.1 (4)° in (1)].

Packing in (6) is dominated by the formation of hydrogen-bonded dimers about a crystallographic inversion centre (Fig. 1). The N···O distance is 2.861 (2) Å.

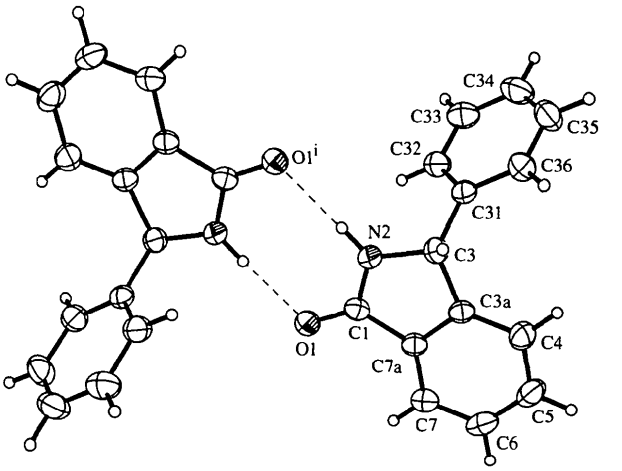


Fig. 1. Formation of hydrogen-bonded dimers of (6). O1^i is related to O1 by the symmetry operation ($\frac{1}{2}-x$, $\frac{1}{2}-y$, $1-z$). Displacement ellipsoids enclose 50% probability surfaces. H atoms are represented by spheres of arbitrary radii.

Experimental

Compound (6) was prepared by the condensation of 2-benzoylbenzoic acid with formamide in the presence of formic acid (Vollmann, Bredereck & Bredereck, 1972).

Crystal data

$C_{14}H_{11}NO$
M_r = 209.24
Monoclinic
$C2/c$
a = 16.3820 (8) Å
b = 6.1648 (6) Å
c = 21.6676 (12) Å
β = 104.835 (3)°
V = 2115.3 (3) Å^3
Z = 8
D_x = 1.314 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 40 reflections
θ = 20–22°
μ = 0.659 mm^{-1}
T = 220 (2) K
Lath developed in (101)
0.43 × 0.23 × 0.04 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer equipped with an Oxford Cryosystems variable temperature device (Cosier & Glazer, 1986)	1238 reflections with $I > 2\sigma(I)$
ω–θ scans	$R_{int} = 0.026$
Absorption correction: none	$\theta_{max} = 60.05°$
3393 measured reflections	$h = -18 \rightarrow 16$
1535 independent reflections	$k = -6 \rightarrow 6$
	$l = -24 \rightarrow 15$
	3 standard reflections
	frequency: 120 min
	intensity decay: 5%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{max} = 0.135$ e Å^{-3}
$wR(F^2) = 0.094$	$\Delta\rho_{min} = -0.132$ e Å^{-3}
$S = 1.048$	Extinction correction: *SHELXTL*
1533 reflections	Extinction coefficient: 0.0012 (2)
146 parameters	Scattering factors from *International Tables for Crystallography* (Vol. C)
H atoms not refined	
$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.058P]$ where $P = (F_o^2 + 2F_c^2)/3$	

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

C1—O1	1.238 (2)	C3a—C7a	1.384 (2)
C1—N2	1.346 (2)	C3a—C4	1.385 (2)
C1—C7a	1.479 (2)	C4—C5	1.385 (2)
N2—C3	1.456 (2)	C5—C6	1.386 (3)
C3—C3a	1.513 (2)	C6—C7	1.380 (2)
C3—C31	1.516 (2)	C7—C7a	1.388 (2)
O1—C1—N2	125.8 (2)	C4—C3a—C3	129.8 (2)
O1—C1—C7a	127.56 (15)	C3a—C4—C5	117.7 (2)
N2—C1—C7a	106.60 (14)	C4—C5—C6	121.5 (2)
C1—N2—C3	114.19 (13)	C7—C6—C5	120.9 (2)
N2—C3—C3a	101.40 (12)	C6—C7—C7a	117.6 (2)
N2—C3—C31	113.16 (13)	C3a—C7a—C7	121.6 (2)
C3a—C3—C31	113.71 (13)	C3a—C7a—C1	108.30 (14)
C7a—C3a—C4	120.7 (2)	C7—C7a—C1	130.1 (2)
C7a—C3a—C3	109.50 (14)		

The presence of the low-temperature device limited $2\theta_{max}$ to 120°.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1321). Services for accessing these data are described at the back of the journal.

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3,4-Etheno-5-methoxymethyl-2′-deoxycytidine†

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Abstract

In the title compound, $C_{13}H_{17}N_3O_5$, the deoxyribose sugar ring adopts a C2′-*exo*–C3′-*endo* symmetrical half-chair conformation (^3_2T), with pseudorotational parameters of $P = 2.54(1)°$ and $\tau_m = 27.82(7)°$. The deoxyribose sugar ring is in the anticlinal (–*ac*) conformation with respect to the base [$\chi = -93.2(4)°$]. The exocyclic side chain at C5′ is in the *gg* conformation [$\gamma = 57.2(3)°$]. The methoxymethyl side chain at C5 is oriented towards the exocyclic side chain at C5′.

Comment

The title compound (3,4-etheno-MMdCyd), (I), is a structural analogue of 5-methoxymethyl-2′-deoxy-

† Alternative name: 6-(4-hydroxy-5-hydroxymethyltetrahydrofuran-2-yl)-8-(methoxymethyl)imidazo[1,2-*c*][1,3]diazin-5(6*H*)-one.