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4-Benzylidene-2-diethylamino-1-phenyl-1*H*-imidazol-5(4*H*)-one

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.169
Data-to-parameter ratio = 16.8

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

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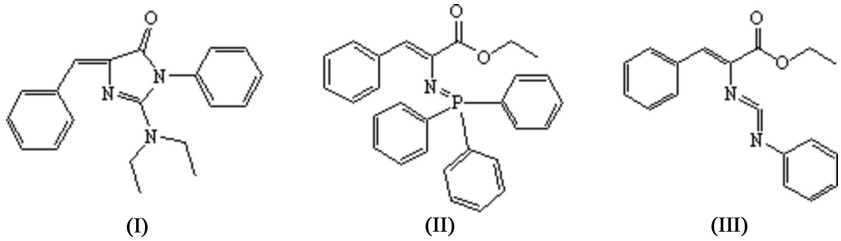
In the crystal structure of the title compound, $C_{20}H_{21}N_3O$, intramolecular C—H···N hydrogen bonds stabilize the conformation of the molecule.

Comment

Derivatives of 4*H*-imidazol-4-ones have shown biological activity (Lacroix *et al.*, 2000). Some 2-alkylaminoimidazolones exhibit good antibacterial and antifungal activities (Trivedi *et al.*, 2002). Recently, in our work on the synthesis of biologically active imidazolinones, we have developed a facile synthesis of 2-dialkylamino-4*H*-imidazolin-4-ones (Hu *et al.*, 2004; Xin & Hu, 2006). The title compound, (I), may be used as a new precursor for obtaining bioactive molecules and its structure is reported here (Fig. 1 and Table 1).



(I) (II) (III)

The five-membered imidazolone ring is planar, with a maximum deviation of 0.012 (2) Å for atoms N2. The C1–C6 phenyl ring is only slightly twisted with respect to the imidazolone ring [dihedral angle = 72.1 (1)°]. The diethylamino substituent (C18/C17/N3/C19/C20) is disordered over two sites, with refined occupancies of 0.829 (4) and 0.171 (4) (Fig. 1). Two intramolecular C—H···N hydrogen-bonding interactions are present which stabilize the conformation of the molecule (Table 2). There are no hydrogen-bonding or π–π interactions (Fig. 2).

Experimental

To a solution of *N*-(1-ethoxycarbonyl-2-phenylethen-1-yl)iminotriphenylphosphorane, (II) (3 mmol), in dry dichloromethane (15 ml) was added phenyl isocyanate (3 mmol) under nitrogen at room temperature. After being left to stand for 8 h, the solvent was removed under reduced pressure, and diethyl ether/petroleum ether (1:2, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration, the solvent was removed to give the carbodiimide (III), which was used directly without further purification. Diethylamine (3 mmol) was added to a solution of the carbodiimide in acetonitrile (15 ml). The mixture was stirred for 6 h, concentrated under reduced pressure and the residue recrystallized from dichloromethane/petroleum ether (1:4) to give the title compound, (I) (yield 92%; m.p.

397 K). Suitable crystals were obtained by vapour diffusion of ethanol into dichloromethane at room temperature.

Crystal data

$C_{20}H_{21}N_3O$
M_r = 319.40
Orthorhombic, *Pbca*
a = 18.8275 (10) Å
b = 8.9784 (5) Å
c = 20.9951 (11) Å
V = 3549.0 (3) Å^3
Z = 8
D_x = 1.196 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, yellow
0.30 × 0.30 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.978, T_{max} = 0.985
28769 measured reflections
4072 independent reflections
2667 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.169
S = 1.08
4072 reflections
243 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.4215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C7—O1	1.209 (2)	C16—N3′	1.456 (7)
C7—N1	1.388 (2)	N3—C19	1.474 (3)
C8—N2	1.385 (2)	N3′—C19′	1.521 (8)
C16—N3	1.346 (3)	N3′—C17′	1.525 (9)
C5—C6—N1	119.61 (17)	N1—C16—N3′	114.5 (5)
O1—C7—N1	125.29 (16)	C16—N3—C17	116.59 (17)
N1—C7—C8	104.07 (14)	C16—N3—C19	123.36 (18)
N2—C8—C7	109.03 (14)	C16—N3′—C19′	123.5 (8)
N2—C16—N3	123.82 (16)	C20′—C19′—N3′	119.1 (17)
N3—C16—N1	121.76 (16)	C7—N1—C6	121.44 (14)
N3—C16—N3′	35.2 (5)	C16—N2—C8	106.04 (14)
C4—C5—C6—N1	−179.7 (2)	N2—C16—N3′—C19′	163.9 (9)
C2—C1—C6—N1	179.28 (19)	N1—C16—N3′—C19′	−54.2 (13)
O1—C7—C8—C9	3.0 (3)	N3—C16—N3′—C17′	−93.7 (16)
N1—C7—C8—C9	−178.30 (17)	C16—N3′—C19′—C20′	−41 (2)
N1—C7—C8—N2	1.30 (19)	C17′—N3′—C19′—C20′	114.5 (19)
N3′—C16—N3—C17	84.7 (7)	O1—C7—N1—C6	19.0 (3)
N1—C16—N3—C19	−31.7 (3)	N3′—C16—N1—C7	−145.4 (6)
N3′—C16—N3—C19	−119.8 (7)	C9—C8—N2—C16	177.51 (19)
C16—N3—C19—C20	127.5 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···N2	0.93	2.44	3.078 (2)	126
C19—H19A···N1	0.97	2.56	2.968 (3)	105

Positional disorder was found in atoms C17–C20/N3 of the diethylamino substituent and atoms of the minor disorder component were refined isotropically. The final site-occupancy factors for the two components were 0.829 (4) and 0.171 (4). All H atoms were positioned geometrically [C—H = 0.93 (CH), 0.97 (CH_2) and 0.96 Å (CH_3)] and constrained to ride on their parent atoms, with U_{iso}(H) values of 1.2 (1.5 for methyl) times U_{eq}(C).

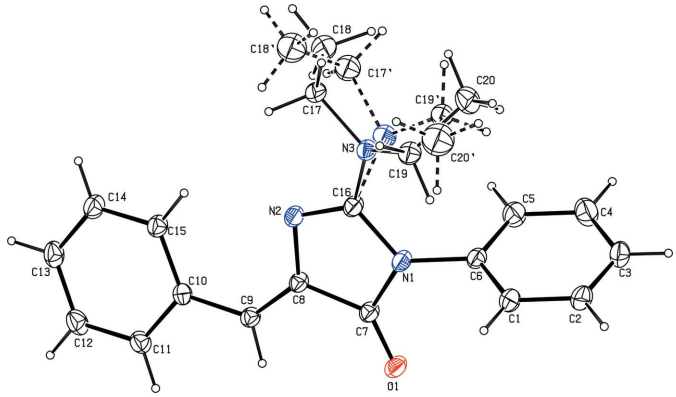


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

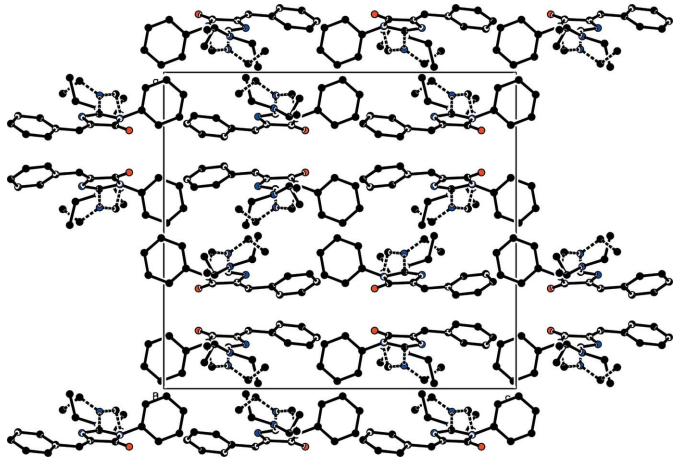


Figure 2
The crystal structure of (I), viewed along the *b* axis. H atoms bonded to C atoms have been omitted for clarity. Only one disorder component is shown.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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