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(*Z*)-5-Amino-2-dibenzylamino-1-phenyl-6-(2-pyridyl)hex-4-en-3-one

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Key indicators

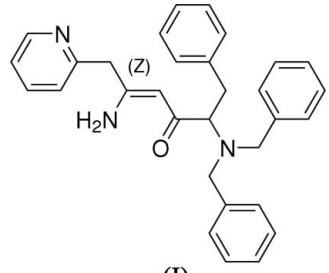
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.064
wR factor = 0.178
Data-to-parameter ratio = 13.7

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

The title compound, $C_{31}H_{31}N_3O$, was obtained by the reaction of 4-dibenzylamino-3-oxo-5-phenylpentanenitrile with 2-methylpyridinylmagnesium chloride. The crystal structure involves intermolecular N—H···O hydrogen bonds.

Comment

In the process of the synthesis of hydroxyethylene dipeptide isosteres used as key structures for the preparation of biologically active candidates with anti-HIV properties, a very important intermediate, (*Z*)-5-amino-2-dibenzylamino-1-phenyl-6-(2-pyridyl)hex-4-en-3-one, (I), was prepared by the reaction of 4-dibenzylamino-3-oxo-5-phenylpentanenitrile with 2-methylpyridinylmagnesium chloride.



(I)

There is an N3—H3*B*···O1^{i} hydrogen bond between two molecules (see Table 1), which links the molecules into a centrosymmetric dimer.

Experimental

To a 268 K solution of 4-dibenzylamino-3-oxo-5-phenylpentanenitrile (1.0 g, 2.2 mmol) in tetrahydrofuran (THF, 10 ml) was added 2-methylpyridinylmagnesium chloride (9.0 ml, 1 *M* in THF, 9.0 mmol). The solution was warmed to ambient temperature and stirred until no starting material was detected by thin-layer chromatography. The solution was then cooled to 278 K and added slowly to a solution of 15% citric acid (20 ml). The organic layer was separated and washed with 10% sodium chloride (10 ml). After concentration *in vacuo*, the residue was crystallized to give a yellow solid (0.9 g, yield 72%) (Stuk *et al.*, 1994). Single crystals were obtained by crystallization from ethyl acetate and petroleum ether (1:2 *v*/*v*).

Crystal data

$C_{31}H_{31}N_3O$
M_r = 461.59
Triclinic, $P\overline{1}$
a = 9.003 (3) Å
b = 10.892 (4) Å
c = 13.802 (5) Å
α = 76.427 (6)°
β = 71.484 (6)°
γ = 84.656 (7)°
V = 1247.2 (8) Å^3
Z = 2
D_x = 1.229 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2165 reflections
θ = 2.4–25.6°
μ = 0.08 mm^{-1}
T = 273 (2) K
Block, yellow
0.56 × 0.34 × 0.21 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.959, T_{max} = 0.984
6380 measured reflections
4319 independent reflections
3386 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.178
S = 1.06
4319 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2 + 0.1595P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···O1^{i}	0.86	2.35	2.987 (3)	131

Symmetry code: (i) $-x, -y+1, -z$.

The H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and N—H = 0.86 Å. H-atom displacement parameters were set equal to $1.5U_{eq}$ of their parent atoms.

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: *ORTEP-3* (Farrugia, 1997) and *ViewerPro* (Accelrys, 2001); software used to prepare material for publication: *SHELXL97*.

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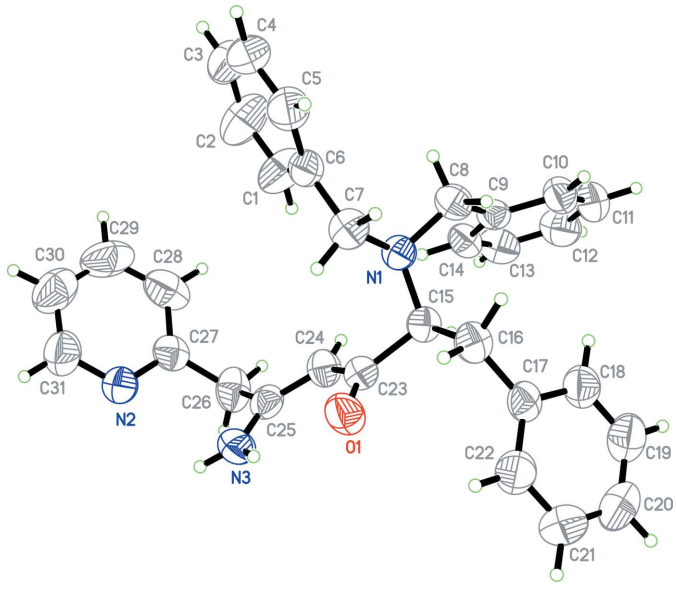

Figure 1
ORTEP-3 (Farrugia, 1997) plot of the title compound. Displacement ellipsoids are drawn at the 50% probability level and the H atoms are shown as spheres of arbitrary radii.

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