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

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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.

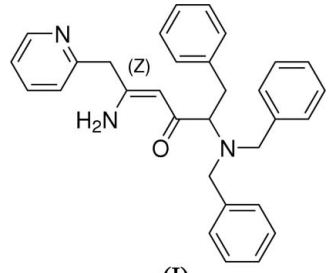
Key indicators

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.

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 $K\alpha$ 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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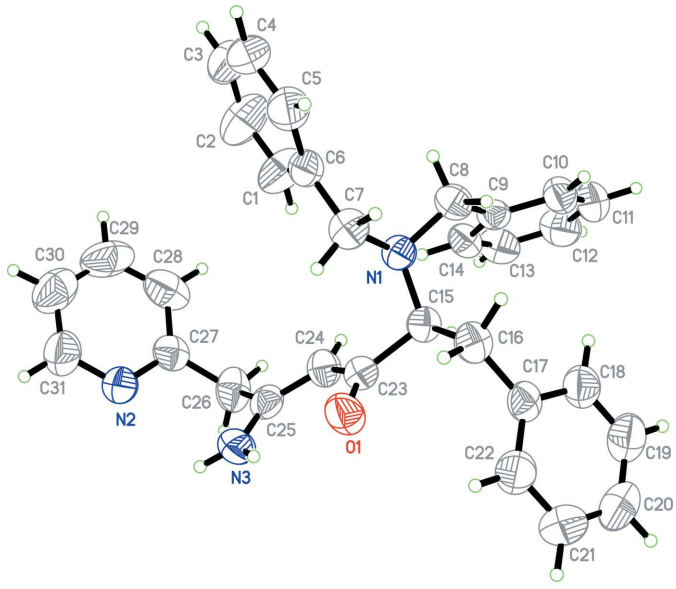

Figure 1
ORTEP-3 (Farrugia, 1997) plot of the title compound. Displacement ellipsoids are drawn at the 50% probability level and the H atoms are shown as spheres of arbitrary radii.

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supporting information

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

Jie Meng, Jian-Feng Zheng, Hua Fang and Li-Ren Jin

S1. 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. X-ray single-crystal analysis of (I) facilitated the characterization of the compound.

S2. Experimental

To a 268 K solution of 4-dibenzylamino-3-oxo-5-phenylpentanenitrile (1.0 g, 2.2 mmol) in tetrahydrofuran (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 transferred 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*).

S3. Refinement

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.

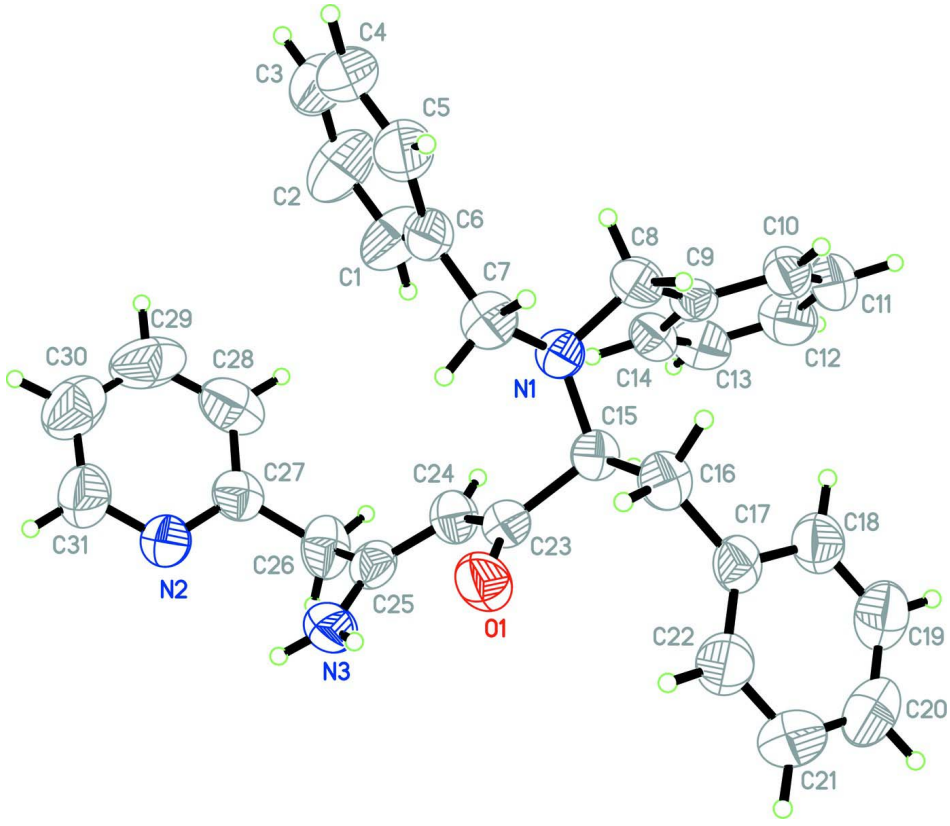

Figure 1

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.

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

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$\gamma = 84.656\ (7)°$
$V = 1247.2\ (8)$ Å^3
$Z = 2$
$F(000) = 492$
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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2165 reflections
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$T = 273$ K
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Radiation source: fine-focus sealed tube
Graphite monochromator
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Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
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$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.178$
$S = 1.06$
4319 reflections
316 parameters
19 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0706 (2)	0.52032 (16)	0.09477 (12)	0.0670 (5)
C15	0.1609 (2)	0.59698 (19)	0.21393 (15)	0.0444 (5)
H15A	0.2546	0.5601	0.2322	0.053*
C23	0.1775 (3)	0.57049 (19)	0.10723 (16)	0.0470 (5)
C9	0.3266 (2)	0.72021 (18)	0.32215 (16)	0.0437 (5)
C25	0.3456 (3)	0.60131 (19)	−0.07408 (17)	0.0505 (6)
C14	0.4661 (3)	0.7042 (2)	0.24900 (17)	0.0516 (6)
H14A	0.4709	0.7188	0.1789	0.062*
C16	0.0216 (3)	0.5303 (2)	0.29939 (17)	0.0528 (6)
H16A	−0.0124	0.5756	0.3556	0.063*
H16B	−0.0644	0.5319	0.2710	0.063*
C24	0.3161 (3)	0.6087 (2)	0.02686 (16)	0.0515 (5)
H24A	0.3937	0.6415	0.0442	0.062*
C17	0.0601 (3)	0.3968 (2)	0.34238 (16)	0.0498 (5)
C8	0.1823 (3)	0.7699 (2)	0.29289 (17)	0.0523 (6)
H8A	0.0912	0.7406	0.3520	0.063*
H8B	0.1811	0.8614	0.2797	0.063*
C10	0.3230 (3)	0.6955 (2)	0.42514 (18)	0.0565 (6)
H10A	0.2289	0.7038	0.4766	0.068*
C27	0.4636 (3)	0.7703 (2)	−0.22811 (17)	0.0539 (6)
C7	0.0466 (3)	0.8063 (2)	0.16528 (19)	0.0543 (6)
H7A	−0.0488	0.8039	0.2236	0.065*
H7B	0.0253	0.7688	0.1140	0.065*
C6	0.0924 (2)	0.9405 (2)	0.11711 (17)	0.0525 (6)
C26	0.4931 (3)	0.6522 (3)	−0.15612 (18)	0.0669 (7)

H26A	0.5662	0.6691	−0.1224	0.080*
H26B	0.5409	0.5890	−0.1964	0.080*
C13	0.5996 (3)	0.6670 (2)	0.2771 (2)	0.0632 (7)
H13A	0.6940	0.6577	0.2261	0.076*
C11	0.4562 (4)	0.6586 (2)	0.4530 (2)	0.0690 (7)
H11A	0.4523	0.6436	0.5229	0.083*
C28	0.5033 (4)	0.8864 (3)	−0.2235 (2)	0.0822 (9)
H28A	0.5512	0.8947	−0.1749	0.099*
C12	0.5939 (3)	0.6438 (3)	0.3786 (2)	0.0716 (7)
H12A	0.6840	0.6178	0.3977	0.086*
C18	0.1452 (3)	0.3697 (2)	0.41209 (18)	0.0632 (7)
H18A	0.1774	0.4355	0.4326	0.076*
C22	0.0161 (3)	0.2981 (3)	0.3145 (2)	0.0685 (7)
H22A	−0.0406	0.3135	0.2670	0.082*
C31	0.3648 (4)	0.8605 (3)	−0.3601 (2)	0.0797 (8)
H31A	0.3157	0.8512	−0.4079	0.096*
C5	−0.0107 (4)	1.0382 (2)	0.1347 (2)	0.0770 (8)
H5A	−0.1125	1.0214	0.1784	0.092*
C19	0.1829 (4)	0.2491 (3)	0.4514 (2)	0.0761 (8)
H19A	0.2413	0.2334	0.4980	0.091*
C20	0.1373 (4)	0.1517 (3)	0.4240 (2)	0.0786 (8)
H20A	0.1626	0.0691	0.4519	0.094*
C1	0.2398 (3)	0.9687 (2)	0.0506 (2)	0.0803 (8)
H1A	0.3121	0.9035	0.0363	0.096*
C30	0.4009 (4)	0.9762 (3)	−0.3611 (3)	0.0900 (10)
H30A	0.3780	1.0455	−0.4084	0.108*
C21	0.0533 (4)	0.1758 (3)	0.3547 (2)	0.0824 (9)
H21A	0.0212	0.1094	0.3348	0.099*
C29	0.4705 (4)	0.9910 (3)	−0.2929 (3)	0.0958 (11)
H29A	0.4969	1.0712	−0.2920	0.115*
C3	0.1789 (5)	1.1864 (3)	0.0249 (3)	0.0985 (11)
H3A	0.2087	1.2696	−0.0056	0.118*
C4	0.0320 (5)	1.1605 (3)	0.0893 (3)	0.0996 (11)
H4A	−0.0401	1.2261	0.1029	0.120*
C2	0.2824 (4)	1.0903 (3)	0.0051 (3)	0.1005 (11)
H2A	0.3833	1.1075	−0.0398	0.121*
N1	0.1686 (2)	0.73285 (15)	0.20208 (13)	0.0447 (4)
N3	0.2474 (3)	0.55393 (18)	−0.10781 (15)	0.0649 (6)
H3B	0.1591	0.5255	−0.0649	0.078*
H3C	0.2715	0.5513	−0.1728	0.078*
N2	0.3943 (3)	0.75754 (18)	−0.29557 (15)	0.0634 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0780 (12)	0.0731 (11)	0.0525 (9)	−0.0330 (10)	−0.0177 (8)	−0.0098 (8)
C15	0.0395 (12)	0.0474 (12)	0.0437 (11)	−0.0029 (9)	−0.0098 (9)	−0.0083 (9)
C23	0.0517 (13)	0.0428 (11)	0.0460 (12)	−0.0020 (10)	−0.0145 (10)	−0.0089 (9)

C9	0.0462 (12)	0.0400 (11)	0.0457 (11)	−0.0070 (9)	−0.0118 (9)	−0.0117 (9)
C25	0.0566 (14)	0.0418 (11)	0.0473 (12)	0.0104 (10)	−0.0125 (11)	−0.0079 (9)
C14	0.0516 (14)	0.0589 (13)	0.0470 (12)	−0.0067 (11)	−0.0136 (10)	−0.0164 (10)
C16	0.0433 (13)	0.0623 (14)	0.0475 (12)	−0.0031 (10)	−0.0061 (10)	−0.0117 (10)
C24	0.0484 (13)	0.0581 (13)	0.0461 (12)	−0.0005 (10)	−0.0128 (10)	−0.0102 (10)
C17	0.0443 (12)	0.0590 (13)	0.0374 (11)	−0.0118 (10)	−0.0012 (9)	−0.0052 (10)
C8	0.0486 (13)	0.0572 (13)	0.0519 (13)	0.0016 (10)	−0.0121 (10)	−0.0185 (10)
C10	0.0625 (15)	0.0598 (14)	0.0458 (12)	−0.0076 (12)	−0.0124 (11)	−0.0122 (10)
C27	0.0494 (14)	0.0619 (14)	0.0422 (12)	−0.0055 (11)	0.0012 (10)	−0.0149 (10)
C7	0.0427 (13)	0.0596 (14)	0.0613 (14)	0.0035 (11)	−0.0181 (11)	−0.0133 (11)
C6	0.0568 (14)	0.0532 (13)	0.0537 (13)	0.0100 (11)	−0.0266 (11)	−0.0143 (10)
C26	0.0566 (15)	0.0835 (17)	0.0477 (13)	0.0139 (13)	−0.0063 (11)	−0.0086 (12)
C13	0.0483 (14)	0.0719 (16)	0.0766 (17)	−0.0010 (12)	−0.0194 (12)	−0.0296 (13)
C11	0.087 (2)	0.0709 (16)	0.0561 (15)	0.0021 (15)	−0.0345 (15)	−0.0122 (12)
C28	0.083 (2)	0.094 (2)	0.0697 (17)	−0.0281 (17)	−0.0064 (15)	−0.0322 (16)
C12	0.0696 (18)	0.0759 (17)	0.085 (2)	0.0133 (14)	−0.0411 (16)	−0.0285 (15)
C18	0.0674 (16)	0.0690 (16)	0.0530 (14)	−0.0143 (13)	−0.0192 (12)	−0.0064 (12)
C22	0.0756 (18)	0.0680 (16)	0.0655 (15)	−0.0158 (14)	−0.0292 (14)	−0.0051 (13)
C31	0.095 (2)	0.078 (2)	0.0594 (16)	−0.0004 (16)	−0.0231 (15)	−0.0035 (14)
C5	0.088 (2)	0.0756 (18)	0.0656 (16)	0.0309 (15)	−0.0254 (15)	−0.0228 (14)
C19	0.0790 (19)	0.0802 (19)	0.0657 (17)	−0.0097 (16)	−0.0280 (15)	0.0022 (14)
C20	0.081 (2)	0.0641 (17)	0.0729 (18)	−0.0003 (15)	−0.0140 (15)	0.0065 (14)
C1	0.0677 (18)	0.0584 (16)	0.096 (2)	0.0054 (13)	−0.0142 (16)	0.0009 (14)
C30	0.100 (3)	0.067 (2)	0.076 (2)	−0.0014 (17)	−0.0024 (18)	0.0023 (15)
C21	0.099 (2)	0.0604 (17)	0.088 (2)	−0.0148 (16)	−0.0272 (18)	−0.0151 (15)
C29	0.109 (3)	0.0549 (17)	0.103 (3)	−0.0278 (17)	0.003 (2)	−0.0165 (17)
C3	0.162 (4)	0.0564 (17)	0.091 (2)	−0.006 (2)	−0.067 (2)	−0.0018 (16)
C4	0.159 (3)	0.0649 (19)	0.085 (2)	0.047 (2)	−0.056 (2)	−0.0288 (17)
C2	0.103 (2)	0.067 (2)	0.113 (3)	−0.0160 (18)	−0.029 (2)	0.0178 (17)
N1	0.0423 (10)	0.0462 (10)	0.0465 (10)	0.0013 (8)	−0.0152 (8)	−0.0107 (8)
N3	0.0899 (16)	0.0575 (12)	0.0466 (11)	−0.0094 (11)	−0.0143 (11)	−0.0163 (9)
N2	0.0796 (15)	0.0560 (12)	0.0527 (11)	−0.0049 (10)	−0.0190 (11)	−0.0086 (9)

Geometric parameters (Å, º)

O1—C23	1.225 (3)	C26—H26B	0.9700
C15—N1	1.456 (3)	C13—C12	1.349 (4)
C15—C16	1.523 (3)	C13—H13A	0.9300
C15—C23	1.525 (3)	C11—C12	1.360 (4)
C15—H15A	0.9800	C11—H11A	0.9300
C23—C24	1.401 (3)	C28—C29	1.382 (5)
C9—C14	1.365 (3)	C28—H28A	0.9300
C9—C10	1.373 (3)	C12—H12A	0.9300
C9—C8	1.498 (3)	C18—C19	1.355 (4)
C25—N3	1.313 (3)	C18—H18A	0.9300
C25—C24	1.353 (3)	C22—C21	1.372 (4)
C25—C26	1.497 (3)	C22—H22A	0.9300
C14—C13	1.376 (3)	C31—N2	1.322 (3)

C14—H14A	0.9300	C31—C30	1.327 (4)
C16—C17	1.489 (3)	C31—H31A	0.9300
C16—H16A	0.9700	C5—C4	1.366 (3)
C16—H16B	0.9700	C5—H5A	0.9300
C24—H24A	0.9300	C19—C20	1.343 (4)
C17—C22	1.353 (3)	C19—H19A	0.9300
C17—C18	1.376 (3)	C20—C21	1.364 (4)
C8—N1	1.446 (3)	C20—H20A	0.9300
C8—H8A	0.9700	C1—C2	1.359 (3)
C8—H8B	0.9700	C1—H1A	0.9300
C10—C11	1.370 (4)	C30—C29	1.329 (5)
C10—H10A	0.9300	C30—H30A	0.9300
C27—N2	1.312 (3)	C21—H21A	0.9300
C27—C28	1.366 (4)	C29—H29A	0.9300
C27—C26	1.489 (3)	C3—C4	1.351 (4)
C7—N1	1.451 (3)	C3—C2	1.352 (4)
C7—C6	1.493 (3)	C3—H3A	0.9300
C7—H7A	0.9700	C4—H4A	0.9300
C7—H7B	0.9700	C2—H2A	0.9300
C6—C5	1.359 (3)	N3—H3B	0.8600
C6—C1	1.366 (3)	N3—H3C	0.8600
C26—H26A	0.9700		

N1—C15—C16	116.91 (18)	C12—C13—H13A	120.0
N1—C15—C23	108.31 (16)	C14—C13—H13A	120.0
C16—C15—C23	112.17 (17)	C12—C11—C10	120.1 (2)
N1—C15—H15A	106.3	C12—C11—H11A	119.9
C16—C15—H15A	106.3	C10—C11—H11A	119.9
C23—C15—H15A	106.3	C27—C28—C29	118.3 (3)
O1—C23—C24	123.3 (2)	C27—C28—H28A	120.8
O1—C23—C15	119.79 (19)	C29—C28—H28A	120.8
C24—C23—C15	116.90 (19)	C13—C12—C11	119.9 (2)
C14—C9—C10	117.9 (2)	C13—C12—H12A	120.1
C14—C9—C8	122.21 (19)	C11—C12—H12A	120.1
C10—C9—C8	119.8 (2)	C19—C18—C17	121.3 (2)
N3—C25—C24	123.4 (2)	C19—C18—H18A	119.3
N3—C25—C26	115.4 (2)	C17—C18—H18A	119.3
C24—C25—C26	121.1 (2)	C17—C22—C21	121.6 (3)
C9—C14—C13	121.2 (2)	C17—C22—H22A	119.2
C9—C14—H14A	119.4	C21—C22—H22A	119.2
C13—C14—H14A	119.4	N2—C31—C30	124.1 (3)
C17—C16—C15	112.24 (18)	N2—C31—H31A	118.0
C17—C16—H16A	109.2	C30—C31—H31A	118.0
C15—C16—H16A	109.2	C6—C5—C4	121.4 (3)
C17—C16—H16B	109.2	C6—C5—H5A	119.3
C15—C16—H16B	109.2	C4—C5—H5A	119.3
H16A—C16—H16B	107.9	C20—C19—C18	120.9 (3)
C25—C24—C23	124.9 (2)	C20—C19—H19A	119.5

C25—C24—H24A	117.6	C18—C19—H19A	119.5
C23—C24—H24A	117.6	C19—C20—C21	119.0 (3)
C22—C17—C18	117.2 (2)	C19—C20—H20A	120.5
C22—C17—C16	122.5 (2)	C21—C20—H20A	120.5
C18—C17—C16	120.3 (2)	C2—C1—C6	121.1 (3)
N1—C8—C9	114.62 (18)	C2—C1—H1A	119.4
N1—C8—H8A	108.6	C6—C1—H1A	119.4
C9—C8—H8A	108.6	C31—C30—C29	118.7 (3)
N1—C8—H8B	108.6	C31—C30—H30A	120.7
C9—C8—H8B	108.6	C29—C30—H30A	120.7
H8A—C8—H8B	107.6	C20—C21—C22	119.9 (3)
C11—C10—C9	120.9 (2)	C20—C21—H21A	120.0
C11—C10—H10A	119.6	C22—C21—H21A	120.0
C9—C10—H10A	119.6	C30—C29—C28	119.5 (3)
N2—C27—C28	121.4 (2)	C30—C29—H29A	120.3
N2—C27—C26	116.4 (2)	C28—C29—H29A	120.3
C28—C27—C26	122.3 (3)	C4—C3—C2	119.4 (3)
N1—C7—C6	111.69 (17)	C4—C3—H3A	120.3
N1—C7—H7A	109.3	C2—C3—H3A	120.3
C6—C7—H7A	109.3	C3—C4—C5	120.0 (3)
N1—C7—H7B	109.3	C3—C4—H4A	120.0
C6—C7—H7B	109.3	C5—C4—H4A	120.0
H7A—C7—H7B	107.9	C3—C2—C1	120.4 (3)
C5—C6—C1	117.6 (2)	C3—C2—H2A	119.8
C5—C6—C7	121.8 (2)	C1—C2—H2A	119.8
C1—C6—C7	120.5 (2)	C8—N1—C7	110.99 (17)
C27—C26—C25	112.0 (2)	C8—N1—C15	114.11 (16)
C27—C26—H26A	109.2	C7—N1—C15	114.71 (16)
C25—C26—H26A	109.2	C25—N3—H3B	120.0
C27—C26—H26B	109.2	C25—N3—H3C	120.0
C25—C26—H26B	109.2	H3B—N3—H3C	120.0
H26A—C26—H26B	107.9	C27—N2—C31	118.1 (2)
C12—C13—C14	120.1 (2)		

N1—C15—C23—O1	−118.3 (2)	C22—C17—C18—C19	−0.2 (4)
C16—C15—C23—O1	12.2 (3)	C16—C17—C18—C19	−179.3 (2)
N1—C15—C23—C24	60.2 (2)	C18—C17—C22—C21	0.6 (4)
C16—C15—C23—C24	−169.25 (19)	C16—C17—C22—C21	179.7 (2)
C10—C9—C14—C13	−1.3 (3)	C1—C6—C5—C4	−1.4 (4)
C8—C9—C14—C13	175.4 (2)	C7—C6—C5—C4	−179.3 (2)
N1—C15—C16—C17	−150.55 (18)	C17—C18—C19—C20	−0.5 (4)
C23—C15—C16—C17	83.5 (2)	C18—C19—C20—C21	0.7 (5)
N3—C25—C24—C23	−1.8 (4)	C5—C6—C1—C2	1.1 (4)
C26—C25—C24—C23	176.0 (2)	C7—C6—C1—C2	179.1 (3)
O1—C23—C24—C25	3.7 (4)	N2—C31—C30—C29	−0.3 (5)
C15—C23—C24—C25	−174.84 (19)	C19—C20—C21—C22	−0.3 (5)
C15—C16—C17—C22	−101.8 (3)	C17—C22—C21—C20	−0.4 (5)
C15—C16—C17—C18	77.2 (3)	C31—C30—C29—C28	0.1 (5)

C14—C9—C8—N1	33.9 (3)	C27—C28—C29—C30	0.5 (5)
C10—C9—C8—N1	−149.52 (19)	C2—C3—C4—C5	0.5 (5)
C14—C9—C10—C11	1.4 (3)	C6—C5—C4—C3	0.6 (5)
C8—C9—C10—C11	−175.3 (2)	C4—C3—C2—C1	−0.8 (5)
N1—C7—C6—C5	−138.7 (2)	C6—C1—C2—C3	−0.1 (5)
N1—C7—C6—C1	43.4 (3)	C9—C8—N1—C7	−167.25 (17)
N2—C27—C26—C25	−72.9 (3)	C9—C8—N1—C15	61.3 (2)
C28—C27—C26—C25	105.9 (3)	C6—C7—N1—C8	70.4 (2)
N3—C25—C26—C27	70.2 (3)	C6—C7—N1—C15	−158.48 (17)
C24—C25—C26—C27	−107.7 (3)	C16—C15—N1—C8	62.5 (2)
C9—C14—C13—C12	0.9 (4)	C23—C15—N1—C8	−169.63 (17)
C9—C10—C11—C12	−1.2 (4)	C16—C15—N1—C7	−67.1 (2)
N2—C27—C28—C29	−0.8 (4)	C23—C15—N1—C7	60.7 (2)
C26—C27—C28—C29	−179.6 (2)	C28—C27—N2—C31	0.5 (4)
C14—C13—C12—C11	−0.7 (4)	C26—C27—N2—C31	179.4 (2)
C10—C11—C12—C13	0.8 (4)	C30—C31—N2—C27	0.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*B*···O1^{i}	0.86	2.35	2.987 (3)	131

Symmetry code: (i) −*x*, −*y*+1, −*z*.