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# 2-(4-Methylphenyl)-3-oxo-4-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]-isoindole-6-carbonitrile

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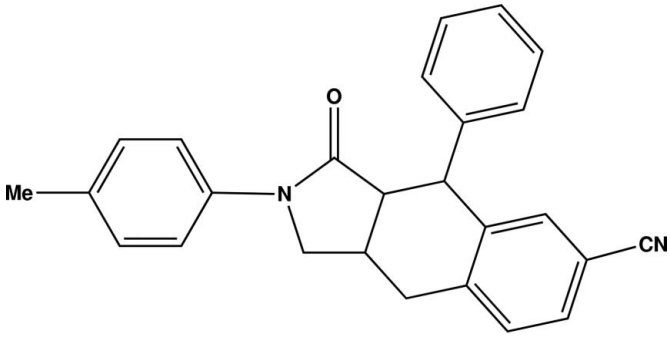
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.052; $wR$ factor = 0.095; data-to-parameter ratio = 15.2.

In the title compound, $C_{26}H_{22}N_2O$, one phenyl ring, one five-membered N-heterocyclic ring and one six-membered carbocyclic ring make up the hexahydrobenzo[*f*]isoindole core. Another phenyl group is attached to the heterocyclic N atom as a substituent. The non-aromatic five- and six-membered rings both exhibit boat conformations. In the crystal, weak C—H···O and C—H···N interactions establish the observed three-dimensional structure. The crystal studied was refined as an inversion twin.

## Related literature

For background to domino reactions, see Zhao *et al.* (2012) and for palladium-catalyzed domino reactions, see Hu *et al.* (2009, 2010). For the wide variety of active pharmaceutical ingredients, natural products and other complex organic molecules economically accessible, see: Yu & Hu (2012); Wang & Hu (2011). For benzo[*f*]isoindol-1-one derivatives as effective intermediates, see: Rixson *et al.* (2012).




## Experimental

### Crystal data

$C_{26}H_{22}N_2O$
$M_r$ = 378.45
Orthorhombic, $Pca2_1$
$a$ = 25.005 (6) Å
$b$ = 5.5023 (14) Å
$c$ = 14.790 (4) Å
$V$ = 2034.9 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 291 K
0.28 × 0.24 × 0.22 mm

### Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.972, $T_{max}$ = 0.983
14617 measured reflections
4001 independent reflections
2187 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.068

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.095
$S$ = 1.03
4001 reflections
264 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C25—H25···O1$^{i}$ | 0.93 | 2.69 | 3.577 (6) | 159 |
| C4—H4···N2$^{ii}$ | 0.93 | 2.62 | 3.329 (6) | 134 |
| C21—H21···O1$^{iii}$ | 0.93 | 2.58 | 3.498 (5) | 169 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2425).

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# supplementary materials

*Acta Cryst.* (2013). E**69**, o652 [doi:10.1107/S1600536813008568]

## 2-(4-Methylphenyl)-3-oxo-4-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindole-6-carbonitrile

**Lei Wen and Yimin Hu**

### Comment

Domino reactions have become an important tool of modern organic synthetic chemistry (Zhao *et al.*, 2012). They have made a wide variety of active pharmaceutical ingredients, natural products and other complex organic molecules economically accessible (Yu *et al.*, 2012; Wang *et al.*, 2011). Benzo[*f*]isoindol-1-one derivatives, which have physiological activities themselves, are effective intermediates in the synthesis of many complex natural products (Rixson *et al.*, 2012). We have reported some novel palladium-catalyzed domino reactions of aryl halides with olefins and diynes (Hu *et al.*, 2010; Hu *et al.*, 2009). The reaction of *N*-allyl-3-phenyl-*N*-(*p*-tolyl)acrylamide with 4-bromobenzonitrile, in the presence of palladium(II) acetate and triphenylphosphine in DMF at 413 K for 26 h unexpectedly generated title product.

The crystal structural data of molecule (I), $C_{26}H_{22}N_2O$, reveals that all the bond lengths and angles have normal values. An asymmetric unit is composed of one title compound molecule. The title compound molecule contains one phenyl ring, one five-membered N-heterocyclic ring and one six-membered carbocyclic ring to make up the hexahydro-benzo[*f*]isoindole core. Another phenyl group is attached to nitrogen as a substituent. The non-aromatic five-membered and six-membered ring both show a boat conformation. All the rings are not coplanar (figure 1). In the crystal structure there are weak intermolecular C—H···O and C—H···N interactions (C25—H25 ···$O_1^{i}$ (i: 1 - *x*,2 - *y*,-0.5 + *z*), C4—H4···$N_2^{ii}$ (ii: -0.5 + *x*,2 - *y*,*z*) and C21—H21···$O_1^{iii}$ (iii: *x*,1 + *y*,*z*)) that establish the observed 3D-structure (Figure 2).

### Experimental

An oven-dried Schlenk flask was evacuated, filled with nitrogen, and then charged with *N*-allyl-3-phenyl-*N*-(*p*-tolyl)-acrylamide (2.77 g, 10 mmol), 4-bromobenzonitrile (1.82 g, 10 mmol), tributylamine (3 ml), $PPh_3$ (52.5 mg, 0.2 mmol), $Pd(OAc)_2$ (24 mg, 0.1 mol), and DMF (10 ml) to give a yellow solution. The reaction mixture was heated to 413 K while stirring for 26 hours. The reaction mixture was then cooled to room temperature and the resulting yellow-orange mixture was diluted with $Et_2O$ (10 ml). The mixture was washed with $H_2O$ (15 ml) and the aqueous layer was extracted with $Et_2O$ (20 ml). The combined organic layers were dried ($MgSO_4$), filtered, and concentrated *in vacuo*. The crude material was purified by flash column chromatography on silica gel (petroleum ether: EtOAc = 9:1) and recrystallized from EtOAc (yield 3.14 g, 83%). Colorless crystals suitable for X-ray diffraction were obtained by another recrystallization from a solution of the title compound in ethyl acetate over a period of one week.

### Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.98 Å, and with $U_{iso}$(H) = 1.2 (1.5 for methyl groups) times $U_{eq}$(C).

Since the crystal obviously was a racemic twin it makes no sense to give information about Flack parameter. So the Flack parameter was omitted from CIF.

### Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

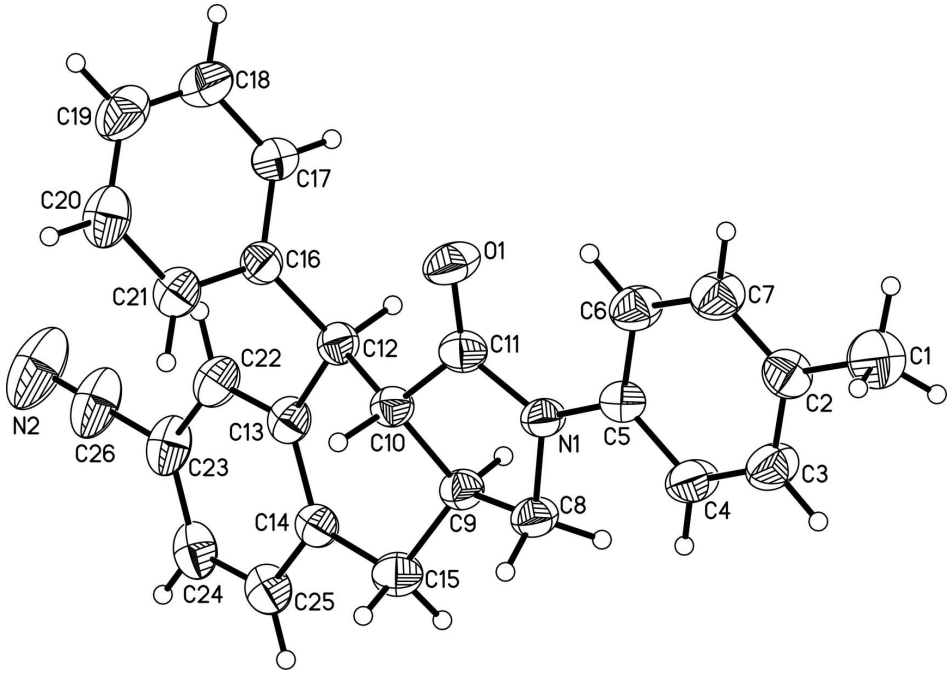


**Figure 1**

A view of the title compound showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

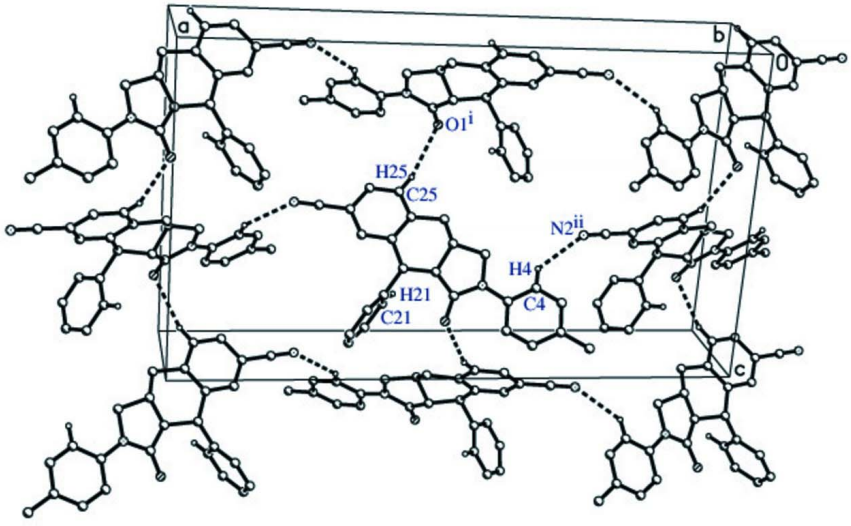


**Figure 2**

A view of a 2-D layer of the title compound (i: 1 - *x*,2 - *y*,-0.5 + *z*; ii: -0.5 + *x*,2 - *y*,*z*). The interaction C21–H21···O1 producing the 3D-structure was omitted for clarity.

**2-(4-Methylphenyl)-3-oxo-4-phenyl-2,3,3a,4,9,9a-hexahydro-1*H*-benzo[*f*]isoindole-6-carbonitrile**

*Crystal data*

$C_{26}H_{22}N_2O$
$M_r = 378.45$
Orthorhombic, $Pca2_1$
$a = 25.005$ (6) Å
$b = 5.5023$ (14) Å
$c = 14.790$ (4) Å
$V = 2034.9$ (9) Å$^3$
$Z = 4$
$F(000) = 800$
$D_x = 1.235$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2537 reflections
$\theta = 2.1$–25.4°
$\mu = 0.08$ mm$^{-1}$
$T = 291$ K
Block, colourless
0.28 × 0.24 × 0.22 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.972$, $T_{max} = 0.983$
14617 measured reflections
4001 independent reflections
2187 reflections with $I > 2\sigma(I)$
$R_{int} = 0.068$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.6°$
$h = -30\rightarrow29$
$k = -6\rightarrow6$
$l = -18\rightarrow18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.095$
$S = 1.03$
4001 reflections
264 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.030P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å$^{-3}$
$\Delta\rho_{min} = -0.15$ 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**. Refined as a 2-component inversion twin.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.27416 (18) | −0.1214 (8) | 0.8866 (4) | 0.0920 (15) |
| H1A | 0.2484 | −0.0456 | 0.9255 | 0.138* |
| H1B | 0.2899 | −0.2577 | 0.9171 | 0.138* |
| H1C | 0.2568 | −0.1761 | 0.8323 | 0.138* |
| C2 | 0.31699 (18) | 0.0587 (8) | 0.8628 (3) | 0.0682 (12) |
| C3 | 0.31038 (18) | 0.2255 (8) | 0.7939 (3) | 0.0762 (13) |
| H3 | 0.2783 | 0.2264 | 0.7621 | 0.091* |
| C4 | 0.34912 (17) | 0.3895 (8) | 0.7706 (3) | 0.0741 (13) |
| H4 | 0.3424 | 0.5008 | 0.7247 | 0.089* |
| C5 | 0.39807 (16) | 0.3928 (7) | 0.8141 (3) | 0.0543 (10) |
| C6 | 0.40556 (18) | 0.2282 (8) | 0.8835 (3) | 0.0690 (13) |
| H6 | 0.4375 | 0.2277 | 0.9157 | 0.083* |
| C7 | 0.36579 (18) | 0.0645 (8) | 0.9050 (3) | 0.0751 (14) |
| H7 | 0.3724 | −0.0479 | 0.9506 | 0.090* |
| C8 | 0.42905 (15) | 0.7232 (7) | 0.7095 (3) | 0.0594 (11) |
| H8A | 0.4059 | 0.8575 | 0.7261 | 0.071* |
| H8B | 0.4135 | 0.6377 | 0.6585 | 0.071* |
| C9 | 0.48494 (15) | 0.8106 (7) | 0.6881 (2) | 0.0525 (10) |
| H9 | 0.5021 | 0.6863 | 0.6506 | 0.063* |
| C10 | 0.51232 (13) | 0.8093 (7) | 0.7792 (3) | 0.0515 (10) |
| H10 | 0.5022 | 0.9586 | 0.8110 | 0.062* |
| C11 | 0.48623 (16) | 0.6001 (7) | 0.8268 (3) | 0.0566 (11) |
| C12 | 0.57264 (14) | 0.8097 (7) | 0.7688 (2) | 0.0515 (10) |
| H12 | 0.5823 | 0.6549 | 0.7404 | 0.062* |
| C13 | 0.58818 (16) | 1.0120 (7) | 0.7015 (3) | 0.0545 (10) |
| C14 | 0.55105 (16) | 1.1225 (7) | 0.6438 (3) | 0.0523 (10) |
| C15 | 0.49270 (16) | 1.0529 (7) | 0.6417 (3) | 0.0640 (11) |
| H15A | 0.4806 | 1.0424 | 0.5795 | 0.077* |
| H15B | 0.4717 | 1.1763 | 0.6722 | 0.077* |
| C16 | 0.60352 (16) | 0.8242 (7) | 0.8567 (3) | 0.0520 (10) |
| C17 | 0.63873 (18) | 0.6434 (8) | 0.8796 (3) | 0.0755 (14) |
| H17 | 0.6434 | 0.5123 | 0.8407 | 0.091* |
| C18 | 0.6674 (2) | 0.6525 (11) | 0.9596 (4) | 0.0998 (18) |
| H18 | 0.6907 | 0.5270 | 0.9747 | 0.120* |
| C19 | 0.6615 (2) | 0.8440 (12) | 1.0156 (4) | 0.0884 (16) |
| H19 | 0.6810 | 0.8514 | 1.0690 | 0.106* |
| C20 | 0.62664 (19) | 1.0285 (10) | 0.9938 (3) | 0.0782 (14) |
| H20 | 0.6226 | 1.1602 | 1.0326 | 0.094* |
| C21 | 0.59746 (17) | 1.0182 (8) | 0.9137 (3) | 0.0625 (11) |

| | | | | |
|---|---|---|---|---|
| H21 | 0.5739 | 1.1429 | 0.8989 | 0.075* |
| C22 | 0.64117 (16) | 1.0829 (8) | 0.6969 (3) | 0.0624 (11) |
| H22 | 0.6661 | 1.0069 | 0.7341 | 0.075* |
| C23 | 0.65808 (19) | 1.2639 (9) | 0.6386 (3) | 0.0681 (12) |
| C24 | 0.6207 (2) | 1.3755 (8) | 0.5835 (3) | 0.0665 (12) |
| H24 | 0.6314 | 1.4998 | 0.5448 | 0.080* |
| C25 | 0.56853 (19) | 1.3048 (7) | 0.5855 (3) | 0.0625 (12) |
| H25 | 0.5441 | 1.3796 | 0.5472 | 0.075* |
| C26 | 0.7132 (2) | 1.3289 (10) | 0.6345 (3) | 0.0920 (17) |
| N1 | 0.43779 (13) | 0.5579 (5) | 0.7866 (2) | 0.0536 (8) |
| N2 | 0.7571 (2) | 1.3807 (9) | 0.6321 (3) | 0.1284 (19) |
| O1 | 0.50390 (11) | 0.4871 (5) | 0.8918 (2) | 0.0770 (8) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.078 (3) | 0.098 (4) | 0.101 (4) | −0.015 (3) | 0.014 (3) | 0.018 (4) |
| C2 | 0.070 (3) | 0.066 (3) | 0.069 (3) | −0.002 (2) | 0.008 (3) | 0.006 (3) |
| C3 | 0.058 (3) | 0.088 (3) | 0.083 (3) | −0.009 (3) | −0.010 (3) | 0.022 (3) |
| C4 | 0.056 (3) | 0.079 (3) | 0.087 (3) | −0.005 (3) | −0.012 (3) | 0.027 (3) |
| C5 | 0.055 (3) | 0.055 (3) | 0.053 (2) | 0.005 (2) | 0.000 (2) | 0.004 (2) |
| C6 | 0.066 (3) | 0.079 (3) | 0.062 (3) | −0.001 (3) | −0.004 (2) | 0.016 (3) |
| C7 | 0.079 (3) | 0.083 (4) | 0.063 (3) | −0.007 (3) | −0.003 (3) | 0.022 (3) |
| C8 | 0.057 (3) | 0.063 (3) | 0.058 (3) | −0.001 (2) | −0.009 (2) | 0.014 (2) |
| C9 | 0.055 (3) | 0.056 (2) | 0.046 (2) | 0.003 (2) | −0.003 (2) | 0.003 (2) |
| C10 | 0.050 (2) | 0.052 (2) | 0.052 (3) | 0.005 (2) | −0.002 (2) | 0.000 (2) |
| C11 | 0.054 (3) | 0.060 (3) | 0.055 (3) | 0.009 (2) | 0.002 (2) | 0.003 (2) |
| C12 | 0.054 (2) | 0.052 (2) | 0.048 (2) | 0.003 (2) | −0.002 (2) | −0.006 (2) |
| C13 | 0.060 (3) | 0.061 (3) | 0.042 (2) | 0.002 (2) | 0.004 (2) | −0.010 (2) |
| C14 | 0.060 (3) | 0.050 (2) | 0.046 (2) | 0.002 (2) | 0.002 (2) | −0.001 (2) |
| C15 | 0.070 (3) | 0.067 (3) | 0.055 (2) | 0.004 (2) | −0.001 (2) | 0.010 (2) |
| C16 | 0.053 (3) | 0.051 (3) | 0.052 (3) | −0.003 (2) | 0.000 (2) | 0.003 (2) |
| C17 | 0.081 (3) | 0.062 (3) | 0.083 (4) | 0.010 (2) | −0.028 (3) | −0.003 (3) |
| C18 | 0.101 (4) | 0.088 (4) | 0.111 (5) | −0.005 (3) | −0.050 (4) | 0.022 (4) |
| C19 | 0.091 (4) | 0.112 (5) | 0.063 (3) | −0.022 (4) | −0.018 (3) | 0.018 (4) |
| C20 | 0.086 (4) | 0.098 (4) | 0.051 (3) | −0.017 (3) | 0.009 (3) | −0.016 (3) |
| C21 | 0.067 (3) | 0.074 (3) | 0.047 (3) | 0.000 (2) | 0.004 (2) | −0.007 (2) |
| C22 | 0.059 (3) | 0.082 (3) | 0.046 (2) | −0.005 (2) | 0.001 (2) | −0.007 (2) |
| C23 | 0.073 (3) | 0.092 (3) | 0.039 (2) | −0.023 (3) | 0.011 (2) | −0.011 (3) |
| C24 | 0.092 (4) | 0.066 (3) | 0.042 (2) | −0.018 (3) | 0.014 (3) | −0.008 (2) |
| C25 | 0.079 (3) | 0.062 (3) | 0.046 (3) | −0.004 (3) | 0.008 (2) | −0.001 (2) |
| C26 | 0.094 (4) | 0.140 (5) | 0.042 (3) | −0.050 (4) | 0.004 (3) | −0.001 (3) |
| N1 | 0.051 (2) | 0.055 (2) | 0.055 (2) | 0.0007 (17) | −0.0069 (18) | 0.0107 (17) |
| N2 | 0.110 (4) | 0.214 (5) | 0.061 (3) | −0.072 (4) | −0.001 (3) | 0.005 (4) |
| O1 | 0.0645 (17) | 0.096 (2) | 0.0705 (19) | −0.0025 (16) | −0.0153 (17) | 0.0313 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.501 (5) | C12—C13 | 1.543 (5) |
| C1—H1A | 0.9600 | C12—H12 | 0.9800 |

| | | | |
|---|---|---|---|
| C1—H1B | 0.9600 | C13—C22 | 1.383 (5) |
| C1—H1C | 0.9600 | C13—C14 | 1.400 (5) |
| C2—C7 | 1.372 (5) | C14—C25 | 1.393 (5) |
| C2—C3 | 1.380 (6) | C14—C15 | 1.509 (5) |
| C3—C4 | 1.368 (6) | C15—H15A | 0.9700 |
| C3—H3 | 0.9300 | C15—H15B | 0.9700 |
| C4—C5 | 1.383 (5) | C16—C21 | 1.369 (5) |
| C4—H4 | 0.9300 | C16—C17 | 1.371 (5) |
| C5—C6 | 1.381 (5) | C17—C18 | 1.385 (6) |
| C5—N1 | 1.406 (5) | C17—H17 | 0.9300 |
| C6—C7 | 1.379 (5) | C18—C19 | 1.348 (7) |
| C6—H6 | 0.9300 | C18—H18 | 0.9300 |
| C7—H7 | 0.9300 | C19—C20 | 1.376 (6) |
| C8—N1 | 1.475 (5) | C19—H19 | 0.9300 |
| C8—C9 | 1.512 (5) | C20—C21 | 1.393 (6) |
| C8—H8A | 0.9700 | C20—H20 | 0.9300 |
| C8—H8B | 0.9700 | C21—H21 | 0.9300 |
| C9—C15 | 1.512 (5) | C22—C23 | 1.384 (6) |
| C9—C10 | 1.512 (5) | C22—H22 | 0.9300 |
| C9—H9 | 0.9800 | C23—C24 | 1.383 (6) |
| C10—C11 | 1.499 (5) | C23—C26 | 1.424 (6) |
| C10—C12 | 1.516 (5) | C24—C25 | 1.363 (5) |
| C10—H10 | 0.9800 | C24—H24 | 0.9300 |
| C11—O1 | 1.227 (5) | C25—H25 | 0.9300 |
| C11—N1 | 1.369 (5) | C26—N2 | 1.136 (6) |
| C12—C16 | 1.514 (5) | | |

| | | | |
|---|---|---|---|
| C2—C1—H1A | 109.5 | C10—C12—H12 | 106.8 |
| C2—C1—H1B | 109.5 | C13—C12—H12 | 106.8 |
| H1A—C1—H1B | 109.5 | C22—C13—C14 | 118.9 (4) |
| C2—C1—H1C | 109.5 | C22—C13—C12 | 118.4 (4) |
| H1A—C1—H1C | 109.5 | C14—C13—C12 | 122.6 (3) |
| H1B—C1—H1C | 109.5 | C25—C14—C13 | 118.8 (4) |
| C7—C2—C3 | 115.3 (4) | C25—C14—C15 | 118.3 (4) |
| C7—C2—C1 | 122.9 (4) | C13—C14—C15 | 122.9 (3) |
| C3—C2—C1 | 121.8 (4) | C14—C15—C9 | 109.8 (3) |
| C4—C3—C2 | 122.7 (4) | C14—C15—H15A | 109.7 |
| C4—C3—H3 | 118.7 | C9—C15—H15A | 109.7 |
| C2—C3—H3 | 118.7 | C14—C15—H15B | 109.7 |
| C3—C4—C5 | 121.2 (4) | C9—C15—H15B | 109.7 |
| C3—C4—H4 | 119.4 | H15A—C15—H15B | 108.2 |
| C5—C4—H4 | 119.4 | C21—C16—C17 | 119.0 (4) |
| C6—C5—C4 | 117.2 (4) | C21—C16—C12 | 120.9 (4) |
| C6—C5—N1 | 122.9 (4) | C17—C16—C12 | 120.1 (4) |
| C4—C5—N1 | 119.9 (4) | C16—C17—C18 | 121.2 (5) |
| C7—C6—C5 | 120.1 (4) | C16—C17—H17 | 119.4 |
| C7—C6—H6 | 119.9 | C18—C17—H17 | 119.4 |
| C5—C6—H6 | 119.9 | C19—C18—C17 | 119.7 (5) |
| C2—C7—C6 | 123.4 (4) | C19—C18—H18 | 120.2 |

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| C2—C7—H7 | 118.3 | C17—C18—H18 | 120.2 |
| C6—C7—H7 | 118.3 | C18—C19—C20 | 120.2 (5) |
| N1—C8—C9 | 102.8 (3) | C18—C19—H19 | 119.9 |
| N1—C8—H8A | 111.2 | C20—C19—H19 | 119.9 |
| C9—C8—H8A | 111.2 | C19—C20—C21 | 120.0 (5) |
| N1—C8—H8B | 111.2 | C19—C20—H20 | 120.0 |
| C9—C8—H8B | 111.2 | C21—C20—H20 | 120.0 |
| H8A—C8—H8B | 109.1 | C16—C21—C20 | 119.9 (4) |
| C8—C9—C15 | 119.6 (3) | C16—C21—H21 | 120.1 |
| C8—C9—C10 | 103.3 (3) | C20—C21—H21 | 120.1 |
| C15—C9—C10 | 110.5 (3) | C13—C22—C23 | 121.7 (4) |
| C8—C9—H9 | 107.6 | C13—C22—H22 | 119.1 |
| C15—C9—H9 | 107.6 | C23—C22—H22 | 119.1 |
| C10—C9—H9 | 107.6 | C24—C23—C22 | 118.7 (4) |
| C11—C10—C9 | 103.0 (3) | C24—C23—C26 | 121.1 (5) |
| C11—C10—C12 | 118.8 (3) | C22—C23—C26 | 120.2 (5) |
| C9—C10—C12 | 111.1 (3) | C25—C24—C23 | 120.4 (4) |
| C11—C10—H10 | 107.8 | C25—C24—H24 | 119.8 |
| C9—C10—H10 | 107.8 | C23—C24—H24 | 119.8 |
| C12—C10—H10 | 107.8 | C24—C25—C14 | 121.3 (4) |
| O1—C11—N1 | 125.0 (4) | C24—C25—H25 | 119.3 |
| O1—C11—C10 | 126.9 (4) | C14—C25—H25 | 119.3 |
| N1—C11—C10 | 108.1 (3) | N2—C26—C23 | 179.3 (5) |
| C16—C12—C10 | 114.8 (3) | C11—N1—C5 | 127.5 (3) |
| C16—C12—C13 | 112.8 (3) | C11—N1—C8 | 111.3 (3) |
| C10—C12—C13 | 108.5 (3) | C5—N1—C8 | 121.2 (3) |
| C16—C12—H12 | 106.8 | | |

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| C7—C2—C3—C4 | −1.5 (7) | C8—C9—C15—C14 | 169.1 (4) |
| C1—C2—C3—C4 | −179.3 (5) | C10—C9—C15—C14 | 49.4 (4) |
| C2—C3—C4—C5 | 1.5 (7) | C10—C12—C16—C21 | −60.2 (5) |
| C3—C4—C5—C6 | −1.7 (6) | C13—C12—C16—C21 | 64.8 (4) |
| C3—C4—C5—N1 | 177.8 (4) | C10—C12—C16—C17 | 120.8 (4) |
| C4—C5—C6—C7 | 2.0 (6) | C13—C12—C16—C17 | −114.2 (4) |
| N1—C5—C6—C7 | −177.5 (4) | C21—C16—C17—C18 | 1.0 (7) |
| C3—C2—C7—C6 | 1.9 (7) | C12—C16—C17—C18 | −180.0 (4) |
| C1—C2—C7—C6 | 179.6 (4) | C16—C17—C18—C19 | −1.1 (8) |
| C5—C6—C7—C2 | −2.2 (7) | C17—C18—C19—C20 | 0.7 (8) |
| N1—C8—C9—C15 | −154.6 (3) | C18—C19—C20—C21 | −0.1 (7) |
| N1—C8—C9—C10 | −31.4 (4) | C17—C16—C21—C20 | −0.4 (6) |
| C8—C9—C10—C11 | 32.8 (4) | C12—C16—C21—C20 | −179.5 (4) |
| C15—C9—C10—C11 | 161.9 (3) | C19—C20—C21—C16 | 0.0 (6) |
| C8—C9—C10—C12 | 161.0 (3) | C14—C13—C22—C23 | 1.7 (6) |
| C15—C9—C10—C12 | −69.8 (4) | C12—C13—C22—C23 | −179.7 (4) |
| C9—C10—C11—O1 | 159.3 (4) | C13—C22—C23—C24 | −0.1 (6) |
| C12—C10—C11—O1 | 36.0 (6) | C13—C22—C23—C26 | −178.8 (4) |
| C9—C10—C11—N1 | −22.2 (4) | C22—C23—C24—C25 | −1.3 (6) |
| C12—C10—C11—N1 | −145.5 (3) | C26—C23—C24—C25 | 177.3 (4) |
| C11—C10—C12—C16 | −64.4 (5) | C23—C24—C25—C14 | 1.2 (6) |

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| C9—C10—C12—C16 | 176.4 (3) | C13—C14—C25—C24 | 0.3 (6) |
| C11—C10—C12—C13 | 168.4 (3) | C15—C14—C25—C24 | −179.6 (4) |
| C9—C10—C12—C13 | 49.2 (4) | O1—C11—N1—C5 | 4.4 (6) |
| C16—C12—C13—C22 | 37.2 (5) | C10—C11—N1—C5 | −174.2 (3) |
| C10—C12—C13—C22 | 165.6 (3) | O1—C11—N1—C8 | −179.2 (4) |
| C16—C12—C13—C14 | −144.2 (4) | C10—C11—N1—C8 | 2.2 (4) |
| C10—C12—C13—C14 | −15.8 (5) | C6—C5—N1—C11 | −6.8 (6) |
| C22—C13—C14—C25 | −1.7 (6) | C4—C5—N1—C11 | 173.8 (4) |
| C12—C13—C14—C25 | 179.7 (3) | C6—C5—N1—C8 | 177.2 (4) |
| C22—C13—C14—C15 | 178.2 (4) | C4—C5—N1—C8 | −2.3 (5) |
| C12—C13—C14—C15 | −0.4 (6) | C9—C8—N1—C11 | 18.7 (4) |
| C25—C14—C15—C9 | 163.7 (3) | C9—C8—N1—C5 | −164.7 (3) |
| C13—C14—C15—C9 | −16.2 (5) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C25—H25···O1$^{i}$ | 0.93 | 2.69 | 3.577 (6) | 159 |
| C4—H4···N2$^{ii}$ | 0.93 | 2.62 | 3.329 (6) | 134 |
| C21—H21···O1$^{iii}$ | 0.93 | 2.58 | 3.498 (5) | 169 |

Symmetry codes: (i) −*x*+1, −*y*+2, *z*−1/2; (ii) *x*−1/2, −*y*+2, *z*; (iii) *x*, *y*+1, *z*.