Acta Crystallographica Section E
**Structure Reports Online**

ISSN 1600-5368

# Ethyl 3′-cyano-1′-methyl-2-oxo-4′-phenylspiro[acenaphthene-1,2′-pyrrolidine]-3′-carboxylate

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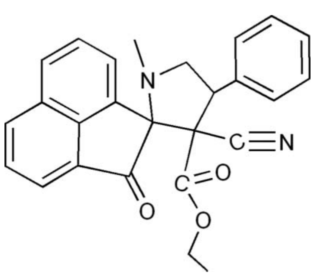
Received 16 November 2007; accepted 18 December 2007

Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.041; $wR$ factor = 0.101; data-to-parameter ratio = 8.9.

In the title compound, $C_{26}H_{22}N_2O_3$, the acenaphthen-1-one ring system is nearly planar and the pyrrolidine ring adopts a distorted envelope conformation. An intermolecular C—H···O hydrogen bond stabilizes the crystal structure.

## Related literature

For related literature, see: Ma & Hecht (2004); Usui *et al.* (1998); Raghunathan & Suresh Babu (2004).



## Experimental

*Crystal data*

$C_{26}H_{22}N_2O_3$
$M_r$ = 410.46
Orthorhombic, $P2_12_12_1$
$a$ = 7.564 (3) Å
$b$ = 14.549 (6) Å
$c$ = 19.397 (8) Å
$V$ = 2134.7 (15) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 294 (2) K
0.22 × 0.18 × 0.14 mm

*Data collection*

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.982, $T_{max}$ = 0.988
12376 measured reflections
2513 independent reflections
1924 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.049

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.101
$S$ = 1.13
2513 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C26—H26$B$···O1$^{i}$ | 0.96 | 2.53 | 3.276 (5) | 135 |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

This project was supported by the National Science Foundation of China (No. 20572057) and the Natural Science Foundation of Shandong Province (Y2006B11) and the Doctoral Foundation of Qingdao University of Science and Technology.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2186).

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# supporting information

*Acta Cryst.* (2008). E**64**, o440 [doi:10.1107/S1600536807067530]

## Ethyl 3′-cyano-1′-methyl-2-oxo-4′-phenylspiro[acenaphthene-1,2′-pyrrolidine]-3′-carboxylate

**Wen-Long Yang, Zheng-Quan Zuo and Ming Li**

### S1. Comment

Spiro compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties. The spirooxindole system is the core structure of many pharmacological agents and natural alkaloids (Ma & Hecht, 2004). Spirotryprostatin A, a natural alkaloid isolated from the fermentation broth of Aspergillus fumigatus, has been identified as a novel inhibitor of microtubule assembly (Usui *et al.*, 1998). Because of their synthetic and biological potential, considerable interest has been focused on the synthesis of spirooxindole derivatives *via* 1,3-dipolar cycloaddition reactions (Raghunathan & Suresh Babu, 2006). In order to develop new biological activities, we synthesized the title compound, the structure of which is reported here.

In the molecule of the title compound (Fig. 1) there is one spiro junction at atom C12. The 2*H*-acenaphthylen-1-one ring system (C1—C12) is nearly planar, with a maximum displacement of 0.081 (3) Å for atom C1. The pyrrolidine ring adopts a distorted envelope conformation, with atom C12 forming the flap of the envelope displaced by 0.288 (2) A%. The mean plane through the pyrrolidine ring is almost perpendicular to the 2*H*-acenaphthylen-1-one ring (dihedral angle 86.62 (7)°). The crystal structure (Fig. 2) is stabilized by an intermolecular C—H···O hydrogen bond (Table 1).

### S2. Experimental

A mixture of acenaphthylene-1,2-dione (1 mmol, 0.182 g), sarcosine (1 mmol, 0.089 g), benzaldehyde (1 mmol, 0.106 g), cyanoacetic acid ethyl ester (1 mmol, 0.113 g), and acetonitrile (15 ml) in a 25 ml flask was stirred for 3 h under reflux and monitored by TLC). After cooling to room temperature, the solid product was filtered off. Single crystals of the title compound were obtained by slow evaporation of an ethanol solution (m.p. 458 K).

### S3. Refinement

All H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for methyl H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged in the final refinement.

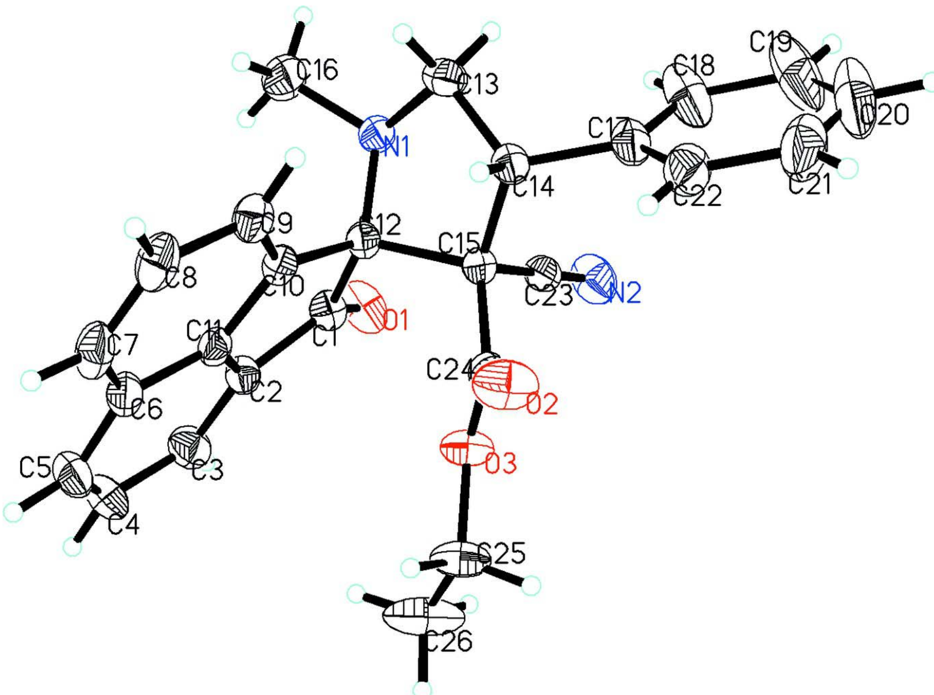


**Figure 1**

The molecular structure of the title compound with 35% probability ellipsoids.

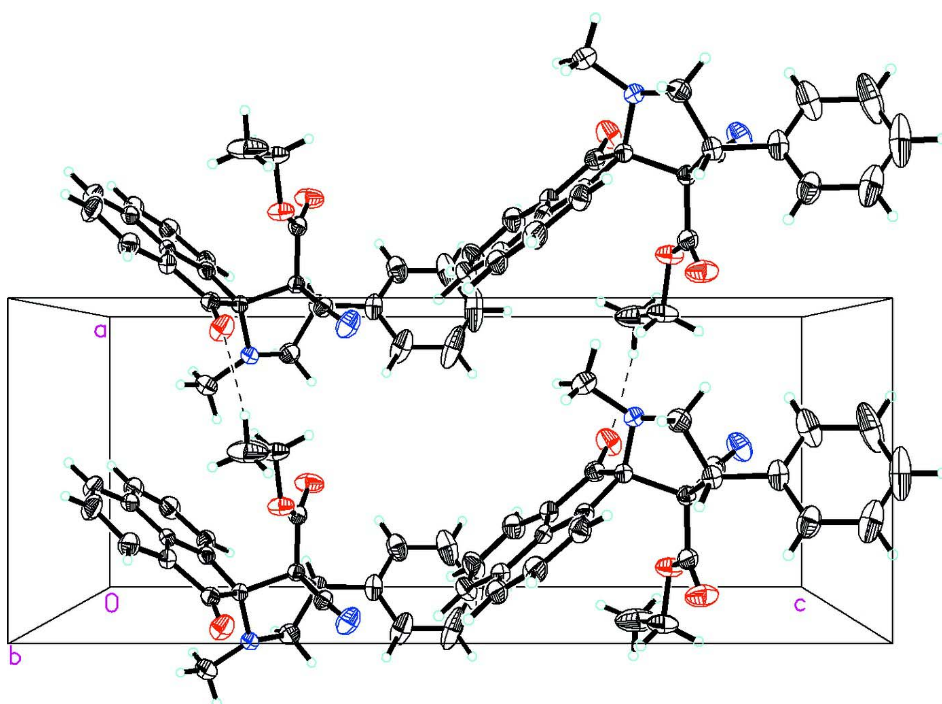


**Figure 2**

Packing diagram of the title compound viewed along the *b* axis. Intermolecular H bonds are shown as dashed lines.

**Ethyl 3′-cyano-1′-methyl-2-oxo-4′-phenyl-spiro[acenaphthene-1,2′- pyrrolidine]-3′-carboxylate**

*Crystal data*

$C_{26}H_{22}N_2O_3$
$M_r = 410.46$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.564$ (3) Å
$b = 14.549$ (6) Å
$c = 19.397$ (8) Å
$V = 2134.7$ (15) Å$^3$
$Z = 4$
$F(000) = 864$
$D_x = 1.277$ Mg m$^{-3}$
Melting point: 458 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3377 reflections
$\theta = 2.5–22.3°$
$\mu = 0.08$ mm$^{-1}$
$T = 294$ K
Prism, brown
0.22 × 0.18 × 0.14 mm

*Data collection*

Bruker SMART 100 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.982$, $T_{max} = 0.988$
12376 measured reflections
2513 independent reflections
1924 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 26.4°$, $\theta_{min} = 1.8°$
$h = -9 \rightarrow 7$
$k = -17 \rightarrow 18$
$l = -23 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.101$
$S = 1.13$
2513 reflections
282 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.2087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ 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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 1.0623 (3) | 0.94458 (13) | 0.30244 (11) | 0.0575 (6) |
| O2 | 0.5975 (3) | 0.73612 (15) | 0.19934 (13) | 0.0628 (6) |
| O3 | 0.6641 (2) | 0.88028 (13) | 0.22923 (11) | 0.0481 (5) |
| N1 | 1.1489 (3) | 0.74780 (15) | 0.28010 (11) | 0.0376 (5) |

| | | | | |
|---|---|---|---|---|
| N2 | 1.0479 (4) | 0.90974 (17) | 0.13933 (13) | 0.0585 (8) |
| C1 | 0.9708 (4) | 0.88274 (18) | 0.32249 (14) | 0.0374 (6) |
| C2 | 0.8422 (4) | 0.88321 (19) | 0.37975 (13) | 0.0376 (6) |
| C3 | 0.7857 (4) | 0.9520 (2) | 0.42294 (16) | 0.0505 (8) |
| H3 | 0.8282 | 1.0117 | 0.4183 | 0.061* |
| C4 | 0.6623 (5) | 0.9298 (3) | 0.47409 (16) | 0.0612 (9) |
| H4 | 0.6204 | 0.9760 | 0.5029 | 0.073* |
| C5 | 0.6013 (4) | 0.8416 (3) | 0.48296 (15) | 0.0557 (9) |
| H5 | 0.5186 | 0.8295 | 0.5173 | 0.067* |
| C6 | 0.6615 (4) | 0.7694 (2) | 0.44122 (14) | 0.0423 (7) |
| C7 | 0.6195 (4) | 0.6742 (2) | 0.44642 (15) | 0.0524 (8) |
| H7 | 0.5406 | 0.6539 | 0.4799 | 0.063* |
| C8 | 0.6951 (4) | 0.6131 (2) | 0.40222 (15) | 0.0525 (8) |
| H8 | 0.6690 | 0.5510 | 0.4075 | 0.063* |
| C9 | 0.8115 (4) | 0.63939 (19) | 0.34877 (14) | 0.0433 (7) |
| H9 | 0.8600 | 0.5955 | 0.3195 | 0.052* |
| C10 | 0.8515 (3) | 0.73065 (18) | 0.34083 (13) | 0.0341 (6) |
| C11 | 0.7806 (3) | 0.79340 (18) | 0.38829 (12) | 0.0338 (6) |
| C12 | 0.9724 (3) | 0.78274 (17) | 0.29125 (12) | 0.0320 (6) |
| C13 | 1.1399 (4) | 0.6722 (2) | 0.23060 (15) | 0.0463 (7) |
| H13A | 1.1395 | 0.6136 | 0.2543 | 0.056* |
| H13B | 1.2406 | 0.6740 | 0.1996 | 0.056* |
| C14 | 0.9672 (4) | 0.68563 (16) | 0.19040 (13) | 0.0357 (6) |
| H14 | 0.8830 | 0.6407 | 0.2088 | 0.043* |
| C15 | 0.9028 (3) | 0.78135 (17) | 0.21481 (12) | 0.0314 (6) |
| C16 | 1.2531 (4) | 0.7268 (3) | 0.34135 (17) | 0.0578 (9) |
| H16A | 1.2516 | 0.7786 | 0.3720 | 0.087* |
| H16B | 1.3727 | 0.7136 | 0.3281 | 0.087* |
| H16C | 1.2035 | 0.6743 | 0.3643 | 0.087* |
| C17 | 0.9744 (5) | 0.67010 (18) | 0.11349 (14) | 0.0460 (8) |
| C18 | 1.1162 (6) | 0.6991 (2) | 0.07389 (17) | 0.0716 (12) |
| H18 | 1.2091 | 0.7311 | 0.0941 | 0.086* |
| C19 | 1.1185 (9) | 0.6799 (3) | 0.0032 (2) | 0.1023 (19) |
| H19 | 1.2134 | 0.6990 | −0.0238 | 0.123* |
| C20 | 0.9814 (10) | 0.6332 (3) | −0.0262 (2) | 0.107 (2) |
| H20 | 0.9847 | 0.6201 | −0.0731 | 0.128* |
| C21 | 0.8409 (8) | 0.6057 (3) | 0.0116 (2) | 0.0861 (14) |
| H21 | 0.7473 | 0.5750 | −0.0094 | 0.103* |
| C22 | 0.8374 (5) | 0.6235 (2) | 0.08185 (16) | 0.0591 (9) |
| H22 | 0.7416 | 0.6037 | 0.1080 | 0.071* |
| C23 | 0.9873 (4) | 0.85531 (18) | 0.17424 (14) | 0.0365 (6) |
| C24 | 0.7021 (4) | 0.79504 (19) | 0.21206 (14) | 0.0370 (6) |
| C25 | 0.4781 (4) | 0.9068 (2) | 0.2361 (2) | 0.0607 (9) |
| H25A | 0.4209 | 0.9062 | 0.1914 | 0.073* |
| H25B | 0.4168 | 0.8639 | 0.2660 | 0.073* |
| C26 | 0.4721 (5) | 0.9978 (3) | 0.2651 (3) | 0.0941 (14) |
| H26A | 0.5226 | 0.9969 | 0.3105 | 0.141* |
| H26B | 0.3516 | 1.0181 | 0.2677 | 0.141* |

| | | | | |
|---|---|---|---|---|
| H26C | 0.5383 | 1.0391 | 0.2364 | 0.141* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0665 (15) | 0.0462 (12) | 0.0598 (13) | −0.0230 (12) | 0.0173 (12) | −0.0112 (10) |
| O2 | 0.0395 (12) | 0.0543 (13) | 0.0945 (18) | −0.0096 (11) | −0.0045 (12) | −0.0145 (13) |
| O3 | 0.0324 (10) | 0.0414 (11) | 0.0706 (14) | 0.0034 (9) | 0.0027 (10) | −0.0039 (10) |
| N1 | 0.0311 (11) | 0.0483 (13) | 0.0335 (12) | 0.0031 (10) | −0.0026 (10) | −0.0012 (10) |
| N2 | 0.0741 (19) | 0.0453 (15) | 0.0561 (16) | −0.0064 (14) | 0.0201 (15) | 0.0074 (13) |
| C1 | 0.0370 (15) | 0.0385 (14) | 0.0365 (14) | −0.0067 (13) | −0.0005 (12) | −0.0024 (12) |
| C2 | 0.0384 (15) | 0.0433 (15) | 0.0312 (13) | 0.0010 (13) | 0.0001 (12) | −0.0016 (12) |
| C3 | 0.0575 (19) | 0.0464 (17) | 0.0477 (17) | 0.0045 (15) | 0.0032 (16) | −0.0073 (14) |
| C4 | 0.065 (2) | 0.073 (2) | 0.0458 (18) | 0.016 (2) | 0.0149 (17) | −0.0116 (17) |
| C5 | 0.0451 (19) | 0.083 (3) | 0.0386 (17) | 0.0052 (17) | 0.0114 (14) | 0.0015 (16) |
| C6 | 0.0340 (15) | 0.0610 (18) | 0.0320 (14) | −0.0032 (15) | −0.0006 (13) | 0.0070 (13) |
| C7 | 0.0482 (19) | 0.071 (2) | 0.0380 (17) | −0.0146 (16) | −0.0013 (14) | 0.0183 (16) |
| C8 | 0.060 (2) | 0.0519 (18) | 0.0456 (17) | −0.0141 (17) | −0.0057 (16) | 0.0170 (15) |
| C9 | 0.0525 (18) | 0.0398 (16) | 0.0377 (15) | −0.0043 (14) | −0.0028 (14) | 0.0046 (12) |
| C10 | 0.0338 (14) | 0.0377 (14) | 0.0307 (14) | −0.0025 (12) | −0.0028 (11) | 0.0041 (11) |
| C11 | 0.0319 (14) | 0.0426 (15) | 0.0269 (13) | −0.0009 (12) | −0.0054 (11) | 0.0015 (11) |
| C12 | 0.0301 (13) | 0.0373 (14) | 0.0287 (13) | −0.0023 (11) | 0.0002 (11) | −0.0005 (10) |
| C13 | 0.0448 (16) | 0.0455 (16) | 0.0486 (17) | 0.0104 (14) | 0.0008 (14) | −0.0063 (14) |
| C14 | 0.0438 (15) | 0.0297 (13) | 0.0336 (14) | −0.0011 (12) | 0.0025 (12) | −0.0001 (11) |
| C15 | 0.0328 (13) | 0.0319 (13) | 0.0293 (13) | −0.0014 (11) | 0.0007 (11) | 0.0003 (11) |
| C16 | 0.0418 (18) | 0.081 (2) | 0.0503 (19) | 0.0054 (17) | −0.0088 (14) | 0.0014 (17) |
| C17 | 0.074 (2) | 0.0295 (14) | 0.0348 (15) | 0.0050 (15) | 0.0030 (16) | −0.0001 (11) |
| C18 | 0.120 (3) | 0.0406 (17) | 0.055 (2) | −0.018 (2) | 0.033 (2) | −0.0067 (15) |
| C19 | 0.200 (6) | 0.048 (2) | 0.059 (2) | −0.013 (3) | 0.060 (3) | 0.0018 (19) |
| C20 | 0.228 (7) | 0.055 (2) | 0.038 (2) | 0.008 (4) | 0.001 (3) | −0.0052 (18) |
| C21 | 0.141 (4) | 0.065 (2) | 0.052 (2) | 0.016 (3) | −0.029 (3) | −0.012 (2) |
| C22 | 0.081 (2) | 0.0478 (17) | 0.0484 (18) | 0.0094 (19) | −0.0120 (18) | −0.0104 (15) |
| C23 | 0.0376 (16) | 0.0357 (14) | 0.0363 (14) | 0.0031 (12) | 0.0035 (13) | 0.0001 (12) |
| C24 | 0.0373 (15) | 0.0394 (16) | 0.0343 (14) | −0.0020 (13) | 0.0005 (12) | 0.0038 (12) |
| C25 | 0.0289 (16) | 0.065 (2) | 0.089 (2) | 0.0090 (15) | 0.0051 (17) | −0.0028 (18) |
| C26 | 0.045 (2) | 0.068 (2) | 0.169 (4) | 0.0164 (19) | −0.002 (3) | −0.025 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.200 (3) | C13—C14 | 1.534 (4) |
| O2—C24 | 1.193 (3) | C13—H13A | 0.9700 |
| O3—C24 | 1.316 (3) | C13—H13B | 0.9700 |
| O3—C25 | 1.465 (4) | C14—C17 | 1.510 (4) |
| N1—C12 | 1.445 (3) | C14—C15 | 1.550 (3) |
| N1—C16 | 1.458 (4) | C14—H14 | 0.9800 |
| N1—C13 | 1.461 (3) | C15—C23 | 1.479 (4) |
| N2—C23 | 1.138 (3) | C15—C24 | 1.532 (4) |
| C1—C2 | 1.476 (4) | C16—H16A | 0.9600 |

| | | | |
|---|---|---|---|
| C1—C12 | 1.576 (4) | C16—H16B | 0.9600 |
| C2—C3 | 1.374 (4) | C16—H16C | 0.9600 |
| C2—C11 | 1.397 (4) | C17—C22 | 1.382 (4) |
| C3—C4 | 1.401 (4) | C17—C18 | 1.385 (5) |
| C3—H3 | 0.9300 | C18—C19 | 1.400 (5) |
| C4—C5 | 1.374 (5) | C18—H18 | 0.9300 |
| C4—H4 | 0.9300 | C19—C20 | 1.365 (8) |
| C5—C6 | 1.401 (4) | C19—H19 | 0.9300 |
| C5—H5 | 0.9300 | C20—C21 | 1.352 (7) |
| C6—C11 | 1.410 (4) | C20—H20 | 0.9300 |
| C6—C7 | 1.425 (4) | C21—C22 | 1.388 (5) |
| C7—C8 | 1.362 (5) | C21—H21 | 0.9300 |
| C7—H7 | 0.9300 | C22—H22 | 0.9300 |
| C8—C9 | 1.413 (4) | C25—C26 | 1.440 (5) |
| C8—H8 | 0.9300 | C25—H25A | 0.9700 |
| C9—C10 | 1.371 (4) | C25—H25B | 0.9700 |
| C9—H9 | 0.9300 | C26—H26A | 0.9600 |
| C10—C11 | 1.403 (4) | C26—H26B | 0.9600 |
| C10—C12 | 1.528 (3) | C26—H26C | 0.9600 |
| C12—C15 | 1.574 (3) | | |

| | | | |
|---|---|---|---|
| C24—O3—C25 | 118.7 (2) | C13—C14—C15 | 103.1 (2) |
| C12—N1—C16 | 116.8 (2) | C17—C14—H14 | 106.5 |
| C12—N1—C13 | 108.7 (2) | C13—C14—H14 | 106.5 |
| C16—N1—C13 | 113.8 (2) | C15—C14—H14 | 106.5 |
| O1—C1—C2 | 128.3 (2) | C23—C15—C24 | 108.4 (2) |
| O1—C1—C12 | 124.3 (2) | C23—C15—C14 | 110.8 (2) |
| C2—C1—C12 | 107.4 (2) | C24—C15—C14 | 114.7 (2) |
| C3—C2—C11 | 120.4 (3) | C23—C15—C12 | 110.3 (2) |
| C3—C2—C1 | 131.8 (3) | C24—C15—C12 | 111.3 (2) |
| C11—C2—C1 | 107.7 (2) | C14—C15—C12 | 101.19 (19) |
| C2—C3—C4 | 118.1 (3) | N1—C16—H16A | 109.5 |
| C2—C3—H3 | 121.0 | N1—C16—H16B | 109.5 |
| C4—C3—H3 | 121.0 | H16A—C16—H16B | 109.5 |
| C5—C4—C3 | 121.9 (3) | N1—C16—H16C | 109.5 |
| C5—C4—H4 | 119.0 | H16A—C16—H16C | 109.5 |
| C3—C4—H4 | 119.0 | H16B—C16—H16C | 109.5 |
| C4—C5—C6 | 121.2 (3) | C22—C17—C18 | 118.9 (3) |
| C4—C5—H5 | 119.4 | C22—C17—C14 | 119.0 (3) |
| C6—C5—H5 | 119.4 | C18—C17—C14 | 122.0 (3) |
| C5—C6—C11 | 116.3 (3) | C17—C18—C19 | 119.5 (4) |
| C5—C6—C7 | 127.9 (3) | C17—C18—H18 | 120.3 |
| C11—C6—C7 | 115.8 (3) | C19—C18—H18 | 120.3 |
| C8—C7—C6 | 119.8 (3) | C20—C19—C18 | 120.0 (5) |
| C8—C7—H7 | 120.1 | C20—C19—H19 | 120.0 |
| C6—C7—H7 | 120.1 | C18—C19—H19 | 120.0 |
| C7—C8—C9 | 123.2 (3) | C21—C20—C19 | 121.2 (4) |
| C7—C8—H8 | 118.4 | C21—C20—H20 | 119.4 |

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| C9—C8—H8 | 118.4 | C19—C20—H20 | 119.4 |
| C10—C9—C8 | 118.8 (3) | C20—C21—C22 | 119.5 (5) |
| C10—C9—H9 | 120.6 | C20—C21—H21 | 120.2 |
| C8—C9—H9 | 120.6 | C22—C21—H21 | 120.2 |
| C9—C10—C11 | 118.2 (2) | C17—C22—C21 | 120.9 (4) |
| C9—C10—C12 | 133.1 (3) | C17—C22—H22 | 119.6 |
| C11—C10—C12 | 108.6 (2) | C21—C22—H22 | 119.6 |
| C2—C11—C10 | 113.8 (2) | N2—C23—C15 | 175.6 (3) |
| C2—C11—C6 | 122.1 (3) | O2—C24—O3 | 125.8 (3) |
| C10—C11—C6 | 124.1 (3) | O2—C24—C15 | 124.8 (3) |
| N1—C12—C10 | 118.2 (2) | O3—C24—C15 | 109.3 (2) |
| N1—C12—C15 | 99.43 (19) | C26—C25—O3 | 107.9 (3) |
| C10—C12—C15 | 112.7 (2) | C26—C25—H25A | 110.1 |
| N1—C12—C1 | 112.9 (2) | O3—C25—H25A | 110.1 |
| C10—C12—C1 | 102.2 (2) | C26—C25—H25B | 110.1 |
| C15—C12—C1 | 111.8 (2) | O3—C25—H25B | 110.1 |
| N1—C13—C14 | 106.1 (2) | H25A—C25—H25B | 108.4 |
| N1—C13—H13A | 110.5 | C25—C26—H26A | 109.5 |
| C14—C13—H13A | 110.5 | C25—C26—H26B | 109.5 |
| N1—C13—H13B | 110.5 | H26A—C26—H26B | 109.5 |
| C14—C13—H13B | 110.5 | C25—C26—H26C | 109.5 |
| H13A—C13—H13B | 108.7 | H26A—C26—H26C | 109.5 |
| C17—C14—C13 | 116.9 (3) | H26B—C26—H26C | 109.5 |
| C17—C14—C15 | 116.6 (2) | | |

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| O1—C1—C2—C3 | 3.3 (5) | C2—C1—C12—C10 | −5.6 (3) |
| C12—C1—C2—C3 | −178.0 (3) | O1—C1—C12—C15 | −66.0 (3) |
| O1—C1—C2—C11 | −174.2 (3) | C2—C1—C12—C15 | 115.2 (2) |
| C12—C1—C2—C11 | 4.6 (3) | C12—N1—C13—C14 | 21.8 (3) |
| C11—C2—C3—C4 | −1.6 (4) | C16—N1—C13—C14 | 153.8 (2) |
| C1—C2—C3—C4 | −178.8 (3) | N1—C13—C14—C17 | 137.8 (2) |
| C2—C3—C4—C5 | 1.7 (5) | N1—C13—C14—C15 | 8.5 (3) |
| C3—C4—C5—C6 | 0.5 (5) | C17—C14—C15—C23 | −44.9 (3) |
| C4—C5—C6—C11 | −2.8 (4) | C13—C14—C15—C23 | 84.6 (2) |
| C4—C5—C6—C7 | 176.0 (3) | C17—C14—C15—C24 | 78.2 (3) |
| C5—C6—C7—C8 | −178.0 (3) | C13—C14—C15—C24 | −152.3 (2) |
| C11—C6—C7—C8 | 0.8 (4) | C17—C14—C15—C12 | −161.9 (2) |
| C6—C7—C8—C9 | −2.1 (5) | C13—C14—C15—C12 | −32.4 (2) |
| C7—C8—C9—C10 | 0.3 (4) | N1—C12—C15—C23 | −72.4 (2) |
| C8—C9—C10—C11 | 2.7 (4) | C10—C12—C15—C23 | 161.5 (2) |
| C8—C9—C10—C12 | 178.2 (3) | C1—C12—C15—C23 | 47.1 (3) |
| C3—C2—C11—C10 | −179.3 (3) | N1—C12—C15—C24 | 167.3 (2) |
| C1—C2—C11—C10 | −1.5 (3) | C10—C12—C15—C24 | 41.2 (3) |
| C3—C2—C11—C6 | −0.8 (4) | C1—C12—C15—C24 | −73.2 (3) |
| C1—C2—C11—C6 | 177.0 (2) | N1—C12—C15—C14 | 45.0 (2) |
| C9—C10—C11—C2 | 174.3 (3) | C10—C12—C15—C14 | −81.1 (2) |
| C12—C10—C11—C2 | −2.3 (3) | C1—C12—C15—C14 | 164.5 (2) |
| C9—C10—C11—C6 | −4.2 (4) | C13—C14—C17—C22 | 138.3 (3) |

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| C12—C10—C11—C6 | 179.2 (2) | C15—C14—C17—C22 | −99.1 (3) |
| C5—C6—C11—C2 | 3.0 (4) | C13—C14—C17—C18 | −40.0 (4) |
| C7—C6—C11—C2 | −176.0 (3) | C15—C14—C17—C18 | 82.5 (4) |
| C5—C6—C11—C10 | −178.7 (3) | C22—C17—C18—C19 | −0.6 (5) |
| C7—C6—C11—C10 | 2.4 (4) | C14—C17—C18—C19 | 177.7 (3) |
| C16—N1—C12—C10 | −49.9 (3) | C17—C18—C19—C20 | 0.2 (6) |
| C13—N1—C12—C10 | 80.4 (3) | C18—C19—C20—C21 | 0.8 (7) |
| C16—N1—C12—C15 | −172.1 (2) | C19—C20—C21—C22 | −1.4 (7) |
| C13—N1—C12—C15 | −41.8 (2) | C18—C17—C22—C21 | 0.0 (5) |
| C16—N1—C12—C1 | 69.2 (3) | C14—C17—C22—C21 | −178.4 (3) |
| C13—N1—C12—C1 | −160.4 (2) | C20—C21—C22—C17 | 1.0 (6) |
| C9—C10—C12—N1 | −46.5 (4) | C25—O3—C24—O2 | 2.2 (5) |
| C11—C10—C12—N1 | 129.4 (2) | C25—O3—C24—C15 | −174.6 (3) |
| C9—C10—C12—C15 | 68.7 (4) | C23—C15—C24—O2 | 131.9 (3) |
| C11—C10—C12—C15 | −115.4 (2) | C14—C15—C24—O2 | 7.5 (4) |
| C9—C10—C12—C1 | −171.1 (3) | C12—C15—C24—O2 | −106.6 (3) |
| C11—C10—C12—C1 | 4.7 (3) | C23—C15—C24—O3 | −51.2 (3) |
| O1—C1—C12—N1 | 45.1 (4) | C14—C15—C24—O3 | −175.7 (2) |
| C2—C1—C12—N1 | −133.6 (2) | C12—C15—C24—O3 | 70.2 (3) |
| O1—C1—C12—C10 | 173.2 (3) | C24—O3—C25—C26 | 171.4 (3) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C26—H26*B*···O1$^{i}$ | 0.96 | 2.53 | 3.276 (5) | 135 |

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