

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

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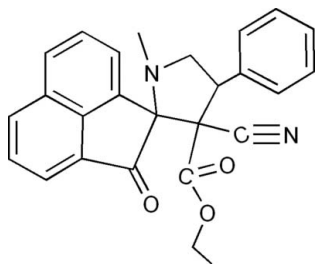
Received 16 November 2007; accepted 18 December 2007

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

In the title compound, $\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_3$, the acenaphthen-1-one ring system is nearly planar and the pyrrolidine ring adopts a distorted envelope conformation. An intermolecular $\text{C}-\text{H}\cdots\text{O}$ 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

$\text{C}_{26}\text{H}_{22}\text{N}_2\text{O}_3$

$M_r = 410.46$

Orthorhombic, $P2_12_1$

$a = 7.564$ (3) Å

$b = 14.549$ (6) Å

$c = 19.397$ (8) Å

$V = 2134.7$ (15) Å³

$Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹

$T = 294$ (2) K
 $0.22 \times 0.18 \times 0.14$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$

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
 $\Delta\rho_{\text{max}} = 0.15$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C}26-\text{H}26B\cdots\text{O}1^i$	0.96	2.53	3.276 (5)	135

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *S SAINT* (Bruker, 1999); data reduction: *S 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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supplementary materials

Acta Cryst. (2008). E64, o440 [doi:10.1107/S1600536807067530]

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

W.-L. Yang, Z.-Q. Zuo and M. Li

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) Å. 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).

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).

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 $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged in the final refinement.

Figures

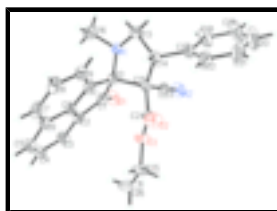


Fig. 1. The molecular structure of the title compound with 35% probability ellipsoids.

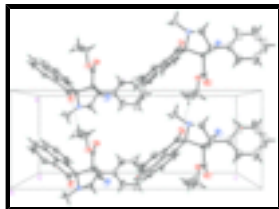


Fig. 2. Packing diagram of the title compound viewed along the *b* axis. Intermolecular H bonds are shown as dashed lines.

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

$a = 7.564 (3) \text{ \AA}$

$b = 14.549 (6) \text{ \AA}$

$c = 19.397 (8) \text{ \AA}$

$V = 2134.7 (15) \text{ \AA}^3$

$Z = 4$

$F_{000} = 864$

$D_x = 1.277 \text{ Mg m}^{-3}$

Melting point: 458 K

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3377 reflections

$\theta = 2.5\text{--}22.3^\circ$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 294 (2) \text{ K}$

Prism, brown

$0.22 \times 0.18 \times 0.14 \text{ mm}$

Data collection

Bruker SMART 100 CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294(2) \text{ K}$

φ and ω 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.049$

$\theta_{\text{max}} = 26.4^\circ$

$\theta_{\text{min}} = 1.8^\circ$

$h = -9 \rightarrow 7$

$k = -17 \rightarrow 18$

$l = -23 \rightarrow 24$

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

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 $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

Primary atom site location: structure-invariant direct methods Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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*

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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 (\AA^2)

	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, °)

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)

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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
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)		
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)
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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C26—H26B \cdots O1 ⁱ	0.96	2.53	3.276 (5)	135

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

Fig. 1

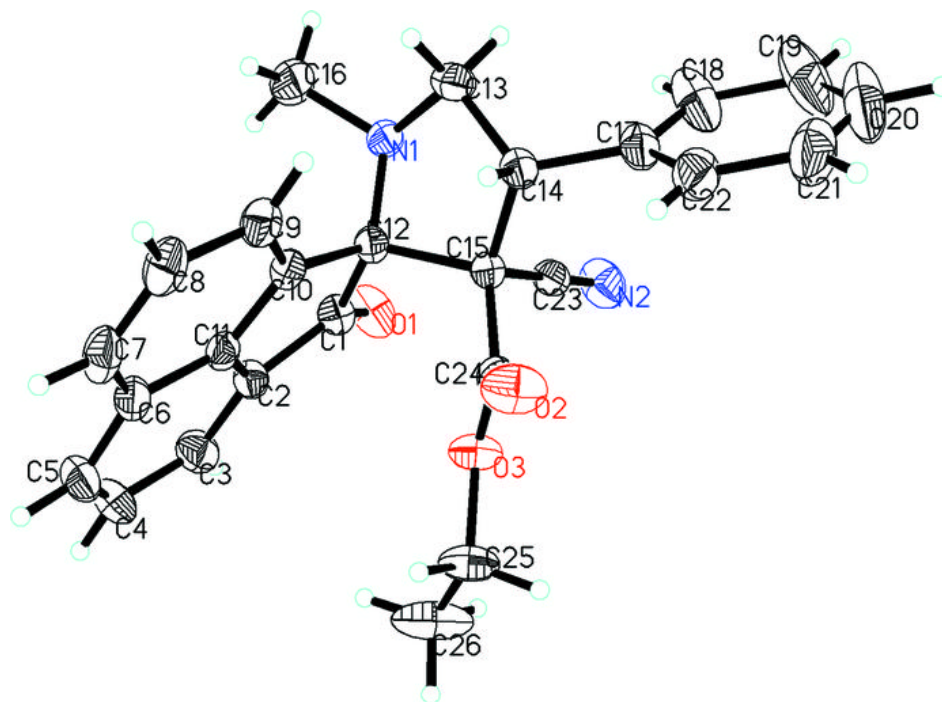


Fig. 2

