

1'-Methyl-4'-(1-naphthyl)-3''-(1-naphthyl-methylene)acenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-1''-cyclohexane-2,2''-dione

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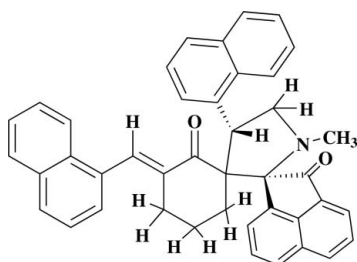
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.050; wR factor = 0.163; data-to-parameter ratio = 13.5.

In the title compound, $\text{C}_{42}\text{H}_{33}\text{NO}_2$, the six-membered cyclohexanone ring adopts a slightly distorted chair conformation and the five-membered pyrrolidine ring is in an envelope conformation. The molecular structure features four intramolecular $\text{C}-\text{H}\cdots\text{O}$ interactions and an intramolecular $\text{C}-\text{H}\cdots\pi$ interaction. Furthermore, the crystal packing is stabilized by an intermolecular $\text{C}-\text{H}\cdots\text{O}$ and three intermolecular $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the biological importance of pyran derivatives, see: Babu & Raghunathan (2007); Chande *et al.* (2005); De March *et al.* (2002); Escolano & Jones (2000); Fejes *et al.* (2001); Poornachandran & Raghunathan (2006); Raj & Raghunathan (2001); Raj *et al.* (2003); Pinna *et al.* (2002). For ring puckering analysis, see: Cremer & Pople (1975). For hydrogen-bonding interactions, see: Desiraju & Steiner (1999).



Experimental

Crystal data

$\text{C}_{42}\text{H}_{33}\text{NO}_2$	$V = 3122.5$ (3) Å ³
$M_r = 583.69$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 12.4398$ (8) Å	$\mu = 0.08$ mm ⁻¹
$b = 17.3501$ (11) Å	$T = 293$ (2) K
$c = 14.4685$ (9) Å	$0.22 \times 0.18 \times 0.16$ mm
$\beta = 90.728$ (17)°	

Data collection

Nonius MACH3 diffractometer	3098 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$R_{\text{int}} = 0.022$
$T_{\text{min}} = 0.943$, $T_{\text{max}} = 0.986$	3 standard reflections
6169 measured reflections	frequency: 60 min
5489 independent reflections	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	407 parameters
$wR(F^2) = 0.164$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.31$ e Å ⁻³
5489 reflections	$\Delta\rho_{\text{min}} = -0.24$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$\text{Cg}1$, $\text{Cg}2$, $\text{Cg}3$ and $\text{Cg}4$ are the centroids of the $\text{C}7/\text{C}70-72/\text{C}80$, $\text{C}95-\text{C}100$, $\text{C}26-\text{C}31$ and $\text{C}72-76/80$ rings.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}5-\text{H}5\text{B}\cdots\text{O}1$	0.97	2.37	3.084 (3)	130
$\text{C}8-\text{H}8\text{A}\cdots\text{O}1$	0.97	2.51	3.078 (4)	117
$\text{C}9-\text{H}9\cdots\text{O}2$	0.98	2.26	2.803 (3)	114
$\text{C}21-\text{H}21\cdots\text{O}2$	0.93	2.42	2.750 (3)	101
$\text{C}73-\text{H}73\cdots\text{O}1^{\text{i}}$	0.93	2.50	3.231 (4)	136
$\text{C}4-\text{H}4\text{A}\cdots\text{Cg}1$	0.97	2.64	3.337 (3)	129
$\text{C}75-\text{H}75\cdots\text{Cg}2^{\text{ii}}$	0.93	2.74	3.646 (3)	164
$\text{C}78-\text{H}78\cdots\text{Cg}3^{\text{iii}}$	0.93	2.82	3.622 (4)	145
$\text{C}96-\text{H}96\cdots\text{Cg}4^{\text{iv}}$	0.93	2.96	3.742 (4)	142

Symmetry codes: (i) $-x, -y, -z + 1$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x + 1, -y, -z + 2$; (iv) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: *CAD-4 EXPRESS* (Enraf-Nonius, 1994); cell refinement: *CAD-4 Express*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2436).

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supplementary materials

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1'-Methyl-4'-(1-naphthyl)-3''-(1-naphthylmethylene)acenaphthene-1-spiro-2'-pyrrolidine-3'-spiro-1''-cyclohexane-2,2''-dione

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Comment

1,3-Dipolar cycloaddition of azomethine ylides to alkenes affords pyrrolidines with high selectivities. Azomethine ylides are reactive and versatile 1,3-dipoles, which react readily with diverse dipolarophiles affording pyrrolizines, pyrrolidines and pyrazolidines (Fejes *et al.*, 2001; De March *et al.*, 2002). Pyrrolidine derivatives are widely used as organic catalysts and also serve as important structural units in biologically active molecules. Pyrrolidine derivatives, apart from displaying important biological activities (Pinna *et al.*, 2002; Escolano & Jones, 2000), are present in natural products such as cephalotoxin, kainic acid, domoic acid and quinocarcin. The cycloaddition of azomethine ylides to dipolarophiles with exocyclic double bonds affords spiro-pyrrolidines (Raj & Raghunathan, 2001; Poornachandran & Raghunathan, 2006), which display important biological activities (Raj *et al.*, 2003). Synthesis of spiro compounds has drawn considerable attention from chemists, in view of their very good antimycobacterial activity (Chande *et al.*, 2005). Acenaphthenequinone is a versatile precursor for azomethine ylide cycloaddition as it reacts with various α -amino acids generating reactive 1,3-dipoles (Babu & Raghunathan, 2007).

In the title compound (I), Fig. 1, the six-membered cyclohexanone ring adopts a slightly distorted chair conformation [$q_2=0.283$ (3) Å, $\pi_2=202.3$ (5)° and $q_3=0.419$ (3) Å; Cremer & Pople, 1975] and the five-membered pyrrolidine ring is in envelope conformation [$q_2=0.401$ (3) Å and $\pi_2=351.5$ (4)°; Cremer & Pople, 1975] (Fig. 1). The dihedral angles between the acenaphthene group and the planes through the naphthyl rings are observed to be 78.7 (1) and 33.2 (1)°. Planes through the naphthyl units themselves are oriented at a dihedral angle of 68.3 (1)°.

The molecular structure features four C—H \cdots O and a C—H \cdots π intramolecular interactions (Desiraju & Steiner, 1999) and the crystal packing is further stabilized by a C—H \cdots O and three C—H \cdots π intermolecular interactions (Fig 2; Table 1). The centroids in detailed in Table 1 are identified as follows: Cg1 - ring C7/C70–72/C80; Cg2 - ring C95–100; Cg3 - ring C26–31; Cg4 - ring C72–76/80.

Experimental

A mixture of 2,6-bis[(*E*)-1-naphthylmethylidene] cyclohexanone (1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was dissolved in methanol (10 ml) and refluxed for 1 h. After completion of the reaction as evident from TLC, the mixture was poured into water (50 ml), the precipitated solid was filtered and washed with water (100 ml) to obtain pure 1-methyl-4-(1-naphthyl)-pyrrolo-(spiro-[2.2'']-acenaphthene-1'-one) -spiro[3.3']-6'-(1-naphthyl)methylidencyclohexanone as yellow solid. The compound was recrystallized from a 1:1 mixture of methanol:ethyl acetate and a yellow solid is obtained, Yield 98%

Refinement

All the H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å and N—H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{parent atom})$.

Figures

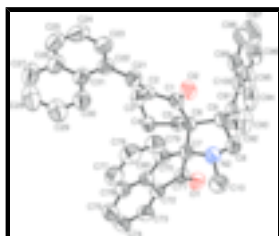


Fig. 1. The molecular structure of the title compound (I) with the numbering scheme for the atoms and 50% probability displacement ellipsoids. H atoms are omitted for clarity.

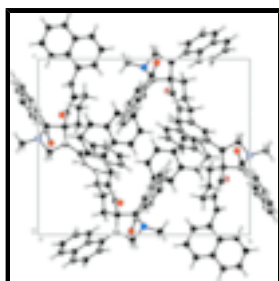


Fig. 2. Packing diagram of the molecules, viewed down the *a*-axis.

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Crystal data

$\text{C}_{42}\text{H}_{33}\text{NO}_2$

$M_r = 583.69$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 12.4398\ (8)\ \text{\AA}$

$b = 17.3501\ (11)\ \text{\AA}$

$c = 14.4685\ (9)\ \text{\AA}$

$\beta = 90.728\ (17)^\circ$

$V = 3122.5\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 1232$

$D_x = 1.242\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 25 reflections

$\theta = 9.4\text{--}13.6^\circ$

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

$T = 293\ (2)\ \text{K}$

Block, pale yellow

$0.22 \times 0.18 \times 0.16\ \text{mm}$

Data collection

Nonius MACH3 sealed tube
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$R_{\text{int}} = 0.022$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.0^\circ$

$T = 293(2)$ K
 ω -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.943$, $T_{\max} = 0.986$
 6169 measured reflections
 5489 independent reflections
 3098 reflections with $I > 2\sigma(I)$

$h = 0 \rightarrow 14$
 $k = -1 \rightarrow 20$
 $l = -17 \rightarrow 17$
 3 standard reflections
 every 60 min
 intensity decay: none

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.164$
 $S = 1.02$
 5489 reflections
 407 parameters
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.7574P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = <0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$
 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C31	0.40070 (19)	0.14164 (18)	1.02686 (18)	0.0648 (7)
C23	0.4443 (2)	0.26622 (19)	0.9649 (2)	0.0809 (8)
H23	0.4609	0.2977	0.9150	0.097*
C96	0.4902 (5)	0.3913 (2)	0.4021 (3)	0.1277 (18)
H96	0.4749	0.4354	0.3680	0.153*
C26	0.3922 (2)	0.1747 (2)	1.1160 (2)	0.0769 (8)
C1	0.4091 (2)	0.13447 (13)	0.69369 (16)	0.0542 (6)
C76	0.1547 (2)	-0.07341 (14)	0.80960 (19)	0.0681 (7)
C80	0.1785 (2)	-0.02788 (13)	0.73238 (17)	0.0573 (6)
C22	0.4280 (2)	0.18940 (17)	0.95041 (18)	0.0653 (7)

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C95	0.4139 (4)	0.3296 (2)	0.4043 (2)	0.0971 (11)
C27	0.3649 (3)	0.1256 (3)	1.1910 (2)	0.1050 (12)
H27	0.3605	0.1462	1.2502	0.126*
O1	0.11519 (16)	0.06038 (12)	0.52182 (13)	0.0769 (5)
C98	0.6076 (3)	0.3212 (2)	0.5014 (3)	0.1121 (13)
H98	0.6728	0.3183	0.5334	0.135*
C99	0.5353 (3)	0.26097 (18)	0.5068 (2)	0.0833 (9)
H99	0.5523	0.2182	0.5429	0.100*
C7	0.27886 (19)	0.04002 (13)	0.61796 (16)	0.0553 (6)
C2	0.37832 (18)	0.17015 (14)	0.78418 (16)	0.0539 (6)
N2	0.34277 (17)	0.00032 (12)	0.54755 (15)	0.0670 (6)
C29	0.3521 (3)	0.0180 (2)	1.0906 (2)	0.0988 (10)
H29	0.3383	-0.0342	1.0818	0.119*
C21	0.4406 (2)	0.15553 (16)	0.85697 (18)	0.0654 (7)
H21	0.4971	0.1211	0.8489	0.079*
C25	0.4114 (2)	0.2541 (3)	1.1274 (2)	0.0935 (11)
H25	0.4069	0.2760	1.1859	0.112*
C28	0.3450 (3)	0.0490 (3)	1.1782 (3)	0.1136 (13)
H28	0.3269	0.0179	1.2280	0.136*
C5	0.2369 (2)	0.18867 (14)	0.62718 (16)	0.0554 (6)
H5A	0.2676	0.2383	0.6114	0.067*
H5B	0.1783	0.1785	0.5840	0.067*
C8	0.3543 (2)	0.05347 (16)	0.47035 (19)	0.0742 (8)
H8A	0.2881	0.0573	0.4345	0.089*
H8B	0.4119	0.0375	0.4300	0.089*
C3	0.2801 (2)	0.22026 (16)	0.78963 (17)	0.0640 (7)
H3A	0.2994	0.2730	0.7748	0.077*
H3B	0.2533	0.2195	0.8523	0.077*
C92	0.2646 (3)	0.2095 (2)	0.4123 (2)	0.0912 (10)
H92	0.2142	0.1699	0.4143	0.109*
C30	0.3787 (2)	0.06225 (19)	1.0169 (2)	0.0760 (8)
H30	0.3826	0.0398	0.9586	0.091*
C75	0.0458 (3)	-0.09408 (18)	0.8194 (2)	0.0869 (9)
H75	0.0254	-0.1231	0.8702	0.104*
C74	-0.0301 (3)	-0.07252 (19)	0.7561 (3)	0.0910 (10)
H74	-0.1010	-0.0877	0.7647	0.109*
C72	0.1000 (2)	-0.00583 (15)	0.66737 (17)	0.0603 (6)
C71	0.1555 (2)	0.03648 (14)	0.59335 (18)	0.0589 (6)
C94	0.3165 (5)	0.3341 (3)	0.3563 (3)	0.1228 (17)
H94	0.3022	0.3779	0.3212	0.147*
C93	0.2409 (4)	0.2770 (3)	0.3584 (2)	0.1118 (13)
H93	0.1762	0.2818	0.3260	0.134*
C9	0.3810 (2)	0.12982 (14)	0.51825 (17)	0.0609 (7)
H9	0.4585	0.1292	0.5311	0.073*
C24	0.4365 (2)	0.2991 (2)	1.0536 (3)	0.0944 (11)
H24	0.4485	0.3516	1.0618	0.113*
C97	0.5839 (5)	0.3858 (3)	0.4489 (4)	0.143 (2)
H97	0.6334	0.4259	0.4459	0.171*
C100	0.4371 (3)	0.26315 (16)	0.45885 (19)	0.0763 (9)

C6	0.32354 (19)	0.12593 (13)	0.61563 (15)	0.0528 (6)
C73	-0.0050 (2)	-0.02810 (17)	0.6779 (2)	0.0769 (8)
H73	-0.0578	-0.0143	0.6349	0.092*
C91	0.3588 (2)	0.20153 (16)	0.46075 (18)	0.0699 (8)
C4	0.19211 (19)	0.19324 (15)	0.72368 (16)	0.0580 (6)
H4A	0.1663	0.1430	0.7425	0.070*
H4B	0.1322	0.2290	0.7247	0.070*
C77	0.2434 (3)	-0.09586 (15)	0.8653 (2)	0.0808 (9)
H77	0.2324	-0.1236	0.9194	0.097*
C78	0.3445 (3)	-0.07686 (16)	0.8400 (2)	0.0829 (9)
H78	0.4019	-0.0943	0.8761	0.100*
C79	0.3667 (2)	-0.03199 (15)	0.7614 (2)	0.0736 (8)
H79	0.4372	-0.0209	0.7456	0.088*
C70	0.2830 (2)	-0.00505 (13)	0.70856 (17)	0.0572 (6)
C10	0.3049 (3)	-0.07619 (17)	0.5196 (2)	0.0916 (10)
H10A	0.2343	-0.0720	0.4925	0.137*
H10B	0.3023	-0.1092	0.5728	0.137*
H10C	0.3532	-0.0976	0.4753	0.137*
O2	0.50114 (14)	0.11274 (11)	0.68220 (12)	0.0725 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C31	0.0473 (14)	0.090 (2)	0.0573 (16)	0.0081 (13)	-0.0047 (11)	-0.0089 (15)
C23	0.081 (2)	0.082 (2)	0.079 (2)	-0.0038 (16)	-0.0039 (16)	-0.0090 (17)
C96	0.202 (5)	0.071 (3)	0.113 (4)	0.006 (3)	0.079 (4)	0.021 (2)
C26	0.0566 (16)	0.111 (3)	0.0627 (18)	0.0165 (16)	-0.0038 (13)	-0.0151 (18)
C1	0.0554 (15)	0.0486 (14)	0.0588 (15)	-0.0017 (11)	0.0139 (11)	0.0046 (11)
C76	0.095 (2)	0.0458 (14)	0.0639 (17)	-0.0112 (14)	0.0151 (15)	-0.0039 (13)
C80	0.0722 (17)	0.0434 (13)	0.0566 (15)	-0.0049 (12)	0.0085 (13)	-0.0059 (12)
C22	0.0543 (15)	0.0771 (19)	0.0642 (17)	0.0040 (13)	-0.0078 (12)	-0.0079 (15)
C95	0.145 (3)	0.072 (2)	0.076 (2)	0.024 (2)	0.050 (2)	0.0145 (18)
C27	0.091 (2)	0.165 (4)	0.060 (2)	0.020 (3)	0.0118 (17)	-0.009 (2)
O1	0.0827 (13)	0.0851 (13)	0.0625 (12)	-0.0054 (10)	-0.0116 (10)	0.0023 (10)
C98	0.121 (3)	0.087 (3)	0.130 (3)	-0.026 (2)	0.062 (2)	-0.022 (2)
C99	0.090 (2)	0.0696 (19)	0.092 (2)	-0.0005 (17)	0.0401 (19)	-0.0078 (16)
C7	0.0601 (15)	0.0514 (14)	0.0547 (14)	-0.0004 (11)	0.0092 (11)	-0.0004 (11)
C2	0.0542 (14)	0.0565 (15)	0.0513 (14)	-0.0078 (12)	0.0064 (11)	0.0007 (11)
N2	0.0743 (14)	0.0531 (12)	0.0740 (14)	0.0009 (11)	0.0165 (11)	-0.0073 (11)
C29	0.099 (2)	0.110 (3)	0.088 (2)	0.001 (2)	0.0126 (19)	0.013 (2)
C21	0.0592 (15)	0.0738 (18)	0.0633 (16)	0.0051 (13)	0.0011 (13)	-0.0059 (14)
C25	0.071 (2)	0.129 (3)	0.080 (2)	0.012 (2)	-0.0083 (17)	-0.039 (2)
C28	0.109 (3)	0.148 (4)	0.084 (3)	0.015 (3)	0.023 (2)	0.025 (3)
C5	0.0639 (15)	0.0500 (14)	0.0525 (14)	0.0019 (11)	0.0053 (11)	0.0007 (11)
C8	0.086 (2)	0.0732 (19)	0.0637 (17)	-0.0031 (15)	0.0229 (15)	-0.0104 (14)
C3	0.0688 (16)	0.0680 (17)	0.0553 (15)	0.0050 (13)	0.0086 (12)	-0.0020 (13)
C92	0.110 (3)	0.106 (3)	0.0574 (17)	0.025 (2)	0.0134 (17)	0.0077 (17)
C30	0.0715 (18)	0.090 (2)	0.0669 (18)	0.0040 (16)	0.0056 (14)	-0.0005 (16)

supplementary materials

C75	0.110 (3)	0.0688 (19)	0.082 (2)	-0.0224 (19)	0.030 (2)	-0.0024 (17)
C74	0.082 (2)	0.090 (2)	0.102 (3)	-0.0288 (19)	0.036 (2)	-0.019 (2)
C72	0.0638 (16)	0.0558 (15)	0.0615 (15)	-0.0080 (12)	0.0086 (12)	-0.0101 (12)
C71	0.0660 (16)	0.0539 (15)	0.0568 (15)	-0.0013 (12)	0.0014 (12)	-0.0084 (13)
C94	0.190 (5)	0.104 (3)	0.076 (2)	0.057 (3)	0.057 (3)	0.029 (2)
C93	0.134 (3)	0.134 (4)	0.068 (2)	0.052 (3)	0.020 (2)	0.011 (2)
C9	0.0692 (16)	0.0583 (16)	0.0556 (15)	-0.0022 (12)	0.0161 (12)	-0.0016 (12)
C24	0.076 (2)	0.092 (2)	0.115 (3)	0.0027 (18)	-0.014 (2)	-0.038 (2)
C97	0.194 (6)	0.089 (3)	0.147 (5)	-0.026 (4)	0.082 (4)	-0.004 (3)
C100	0.110 (2)	0.0607 (18)	0.0590 (17)	0.0103 (17)	0.0449 (17)	0.0025 (14)
C6	0.0600 (14)	0.0499 (14)	0.0486 (13)	-0.0029 (11)	0.0101 (11)	0.0006 (11)
C73	0.0668 (18)	0.083 (2)	0.082 (2)	-0.0135 (15)	0.0081 (15)	-0.0172 (17)
C91	0.088 (2)	0.0700 (18)	0.0520 (15)	0.0099 (16)	0.0259 (15)	0.0033 (13)
C4	0.0556 (14)	0.0620 (15)	0.0566 (14)	0.0066 (12)	0.0081 (11)	0.0004 (12)
C77	0.130 (3)	0.0462 (16)	0.0663 (18)	-0.0080 (17)	0.0000 (19)	0.0075 (13)
C78	0.109 (3)	0.0551 (17)	0.084 (2)	0.0058 (17)	-0.0210 (19)	0.0105 (16)
C79	0.0805 (19)	0.0548 (16)	0.085 (2)	0.0013 (14)	-0.0076 (16)	0.0097 (15)
C70	0.0646 (16)	0.0441 (13)	0.0628 (15)	-0.0005 (12)	0.0034 (12)	0.0024 (12)
C10	0.105 (2)	0.0616 (19)	0.109 (3)	-0.0059 (17)	0.0251 (19)	-0.0234 (17)
O2	0.0570 (11)	0.0907 (14)	0.0700 (12)	0.0072 (10)	0.0124 (9)	-0.0036 (10)

Geometric parameters (Å, °)

C31—C30	1.411 (4)	C25—H25	0.9300
C31—C26	1.417 (4)	C28—H28	0.9300
C31—C22	1.427 (4)	C5—C4	1.512 (3)
C23—C22	1.364 (4)	C5—C6	1.543 (3)
C23—C24	1.409 (4)	C5—H5A	0.9700
C23—H23	0.9300	C5—H5B	0.9700
C96—C97	1.344 (7)	C8—C9	1.530 (4)
C96—C95	1.431 (6)	C8—H8A	0.9700
C96—H96	0.9300	C8—H8B	0.9700
C26—C25	1.407 (5)	C3—C4	1.517 (3)
C26—C27	1.425 (5)	C3—H3A	0.9700
C1—O2	1.218 (3)	C3—H3B	0.9700
C1—C2	1.502 (3)	C92—C91	1.365 (4)
C1—C6	1.549 (3)	C92—C93	1.436 (5)
C76—C80	1.403 (3)	C92—H92	0.9300
C76—C75	1.411 (4)	C30—H30	0.9300
C76—C77	1.412 (4)	C75—C74	1.359 (4)
C80—C72	1.401 (3)	C75—H75	0.9300
C80—C70	1.405 (3)	C74—C73	1.407 (4)
C22—C21	1.484 (4)	C74—H74	0.9300
C95—C94	1.391 (6)	C72—C73	1.373 (4)
C95—C100	1.425 (4)	C72—C71	1.477 (4)
C27—C28	1.363 (5)	C94—C93	1.366 (6)
C27—H27	0.9300	C94—H94	0.9300
O1—C71	1.217 (3)	C93—H93	0.9300
C98—C99	1.381 (4)	C9—C91	1.520 (4)

C98—C97	1.384 (6)	C9—C6	1.589 (3)
C98—H98	0.9300	C9—H9	0.9800
C99—C100	1.398 (4)	C24—H24	0.9300
C99—H99	0.9300	C97—H97	0.9300
C7—N2	1.471 (3)	C100—C91	1.447 (4)
C7—C70	1.527 (3)	C73—H73	0.9300
C7—C71	1.572 (3)	C4—H4A	0.9700
C7—C6	1.591 (3)	C4—H4B	0.9700
C2—C21	1.324 (3)	C77—C78	1.355 (4)
C2—C3	1.503 (3)	C77—H77	0.9300
N2—C8	1.457 (3)	C78—C79	1.408 (4)
N2—C10	1.464 (3)	C78—H78	0.9300
C29—C30	1.358 (4)	C79—C70	1.366 (3)
C29—C28	1.381 (5)	C79—H79	0.9300
C29—H29	0.9300	C10—H10A	0.9600
C21—H21	0.9300	C10—H10B	0.9600
C25—C24	1.363 (5)	C10—H10C	0.9600
C30—C31—C26	118.2 (3)	H3A—C3—H3B	108.0
C30—C31—C22	122.3 (3)	C91—C92—C93	122.0 (4)
C26—C31—C22	119.5 (3)	C91—C92—H92	119.0
C22—C23—C24	121.6 (3)	C93—C92—H92	119.0
C22—C23—H23	119.2	C29—C30—C31	121.3 (3)
C24—C23—H23	119.2	C29—C30—H30	119.3
C97—C96—C95	120.5 (5)	C31—C30—H30	119.3
C97—C96—H96	119.7	C74—C75—C76	121.5 (3)
C95—C96—H96	119.7	C74—C75—H75	119.2
C25—C26—C31	119.2 (3)	C76—C75—H75	119.2
C25—C26—C27	122.6 (3)	C75—C74—C73	122.2 (3)
C31—C26—C27	118.2 (3)	C75—C74—H74	118.9
O2—C1—C2	119.8 (2)	C73—C74—H74	118.9
O2—C1—C6	120.6 (2)	C73—C72—C80	120.4 (3)
C2—C1—C6	119.6 (2)	C73—C72—C71	132.4 (3)
C80—C76—C75	115.8 (3)	C80—C72—C71	107.1 (2)
C80—C76—C77	116.0 (3)	O1—C71—C72	126.5 (2)
C75—C76—C77	128.1 (3)	O1—C71—C7	124.7 (2)
C72—C80—C76	122.4 (2)	C72—C71—C7	108.6 (2)
C72—C80—C70	113.4 (2)	C93—C94—C95	122.9 (4)
C76—C80—C70	124.0 (2)	C93—C94—H94	118.5
C23—C22—C31	119.0 (3)	C95—C94—H94	118.5
C23—C22—C21	120.7 (3)	C94—C93—C92	117.9 (4)
C31—C22—C21	120.3 (3)	C94—C93—H93	121.1
C94—C95—C100	119.4 (4)	C92—C93—H93	121.1
C94—C95—C96	121.4 (4)	C91—C9—C8	115.1 (2)
C100—C95—C96	119.2 (4)	C91—C9—C6	116.1 (2)
C28—C27—C26	121.6 (3)	C8—C9—C6	105.51 (19)
C28—C27—H27	119.2	C91—C9—H9	106.5
C26—C27—H27	119.2	C8—C9—H9	106.5
C99—C98—C97	120.7 (5)	C6—C9—H9	106.5
C99—C98—H98	119.7	C25—C24—C23	120.0 (3)

supplementary materials

C97—C98—H98	119.7	C25—C24—H24	120.0
C98—C99—C100	121.1 (4)	C23—C24—H24	120.0
C98—C99—H99	119.4	C96—C97—C98	120.7 (5)
C100—C99—H99	119.4	C96—C97—H97	119.7
N2—C7—C70	110.0 (2)	C98—C97—H97	119.7
N2—C7—C71	111.06 (19)	C99—C100—C95	117.8 (3)
C70—C7—C71	101.33 (19)	C99—C100—C91	123.7 (3)
N2—C7—C6	103.41 (18)	C95—C100—C91	118.5 (3)
C70—C7—C6	119.34 (19)	C5—C6—C1	109.17 (19)
C71—C7—C6	111.84 (19)	C5—C6—C9	112.88 (19)
C21—C2—C1	117.4 (2)	C1—C6—C9	109.24 (19)
C21—C2—C3	122.5 (2)	C5—C6—C7	114.45 (19)
C1—C2—C3	120.1 (2)	C1—C6—C7	108.13 (18)
C8—N2—C10	113.4 (2)	C9—C6—C7	102.69 (18)
C8—N2—C7	107.08 (19)	C72—C73—C74	117.7 (3)
C10—N2—C7	116.2 (2)	C72—C73—H73	121.2
C30—C29—C28	121.3 (4)	C74—C73—H73	121.2
C30—C29—H29	119.3	C92—C91—C100	119.3 (3)
C28—C29—H29	119.3	C92—C91—C9	120.8 (3)
C2—C21—C22	125.5 (2)	C100—C91—C9	119.8 (3)
C2—C21—H21	117.2	C5—C4—C3	109.0 (2)
C22—C21—H21	117.2	C5—C4—H4A	109.9
C24—C25—C26	120.7 (3)	C3—C4—H4A	109.9
C24—C25—H25	119.7	C5—C4—H4B	109.9
C26—C25—H25	119.7	C3—C4—H4B	109.9
C27—C28—C29	119.4 (4)	H4A—C4—H4B	108.3
C27—C28—H28	120.3	C78—C77—C76	119.9 (3)
C29—C28—H28	120.3	C78—C77—H77	120.0
C4—C5—C6	113.8 (2)	C76—C77—H77	120.0
C4—C5—H5A	108.8	C77—C78—C79	123.0 (3)
C6—C5—H5A	108.8	C77—C78—H78	118.5
C4—C5—H5B	108.8	C79—C78—H78	118.5
C6—C5—H5B	108.8	C70—C79—C78	119.0 (3)
H5A—C5—H5B	107.7	C70—C79—H79	120.5
N2—C8—C9	102.9 (2)	C78—C79—H79	120.5
N2—C8—H8A	111.2	C79—C70—C80	117.8 (2)
C9—C8—H8A	111.2	C79—C70—C7	132.3 (2)
N2—C8—H8B	111.2	C80—C70—C7	109.5 (2)
C9—C8—H8B	111.2	N2—C10—H10A	109.5
H8A—C8—H8B	109.1	N2—C10—H10B	109.5
C2—C3—C4	111.6 (2)	H10A—C10—H10B	109.5
C2—C3—H3A	109.3	N2—C10—H10C	109.5
C4—C3—H3A	109.3	H10A—C10—H10C	109.5
C2—C3—H3B	109.3	H10B—C10—H10C	109.5
C4—C3—H3B	109.3		

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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C5—H5B…O1	0.97	2.37	3.084 (3)	130
C8—H8A…O1	0.97	2.51	3.078 (4)	117
C9—H9…O2	0.98	2.26	2.803 (3)	114
C21—H21…O2	0.93	2.42	2.750 (3)	101
C73—H73…O1 ⁱ	0.93	2.50	3.231 (4)	136
C4—H4A…Cg1	0.97	2.64	3.337 (3)	129
C75—H75…Cg2 ⁱⁱ	0.93	2.74	3.646 (3)	164
C78—H78…Cg3 ⁱⁱⁱ	0.93	2.82	3.622 (4)	145
C96—H96…Cg4 ^{iv}	0.93	2.96	3.742 (4)	142

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $-x+1/2, y-1/2, -z+3/2$; (iii) $-x+1, -y, -z+2$; (iv) $x+1/2, -y+1/2, z-1/2$.

Fig. 1

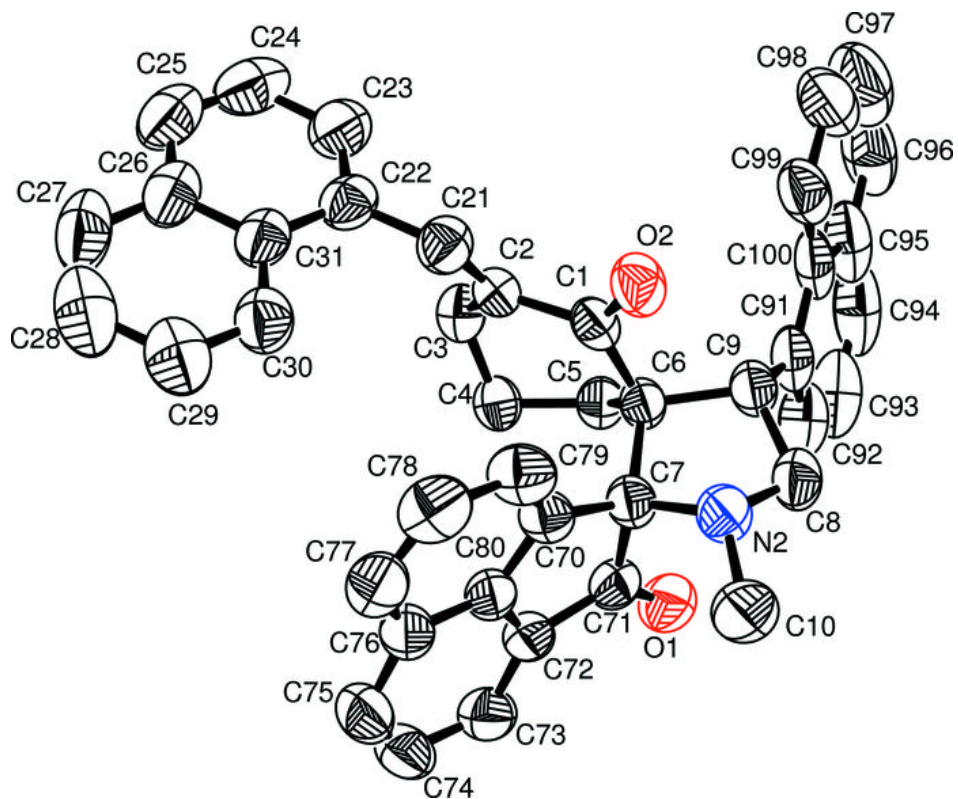


Fig. 2

