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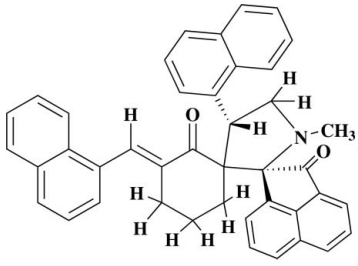
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.050; $wR$ factor = 0.163; data-to-parameter ratio = 13.5.

In the title compound, $C_{42}H_{33}NO_2$, 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 C—H···O interactions and an intramolecular C—H···$\pi$ interaction. Furthermore, the crystal packing is stabilized by an intermolecular C—H···O and three intermolecular C—H···$\pi$ 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

$C_{42}H_{33}NO_2$
$M_r$ = 583.69
Monoclinic, $P2_1/n$
$a$ = 12.4398 (8) Å
$b$ = 17.3501 (11) Å
$c$ = 14.4685 (9) Å
$\beta$ = 90.728 (17)°
$V$ = 3122.5 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 293 (2) K
0.22 × 0.18 × 0.16 mm

### Data collection

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

### Refinement

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

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

*Cg*1, *Cg*2, *Cg*3 and *Cg*4 are the centroids of the C7/C70–72/C80, C95–C100, C26–C31 and C72–76/80 rings.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5*B*···O1 | 0.97 | 2.37 | 3.084 (3) | 130 |
| C8—H8*A*···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—H4*A*···*Cg*1 | 0.97 | 2.64 | 3.337 (3) | 129 |
| C75—H75···*Cg*2$^{ii}$ | 0.93 | 2.74 | 3.646 (3) | 164 |
| C78—H78···*Cg*3$^{iii}$ | 0.93 | 2.82 | 3.622 (4) | 145 |
| C96—H96···*Cg*4$^{iv}$ | 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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# supporting information

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

**S. Athimoolam, V. Anu Radha, S. Asath Bahadur, R. Ranjith Kumar and S. Perumal**

### S1. 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 $\alpha$-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···O and a C—H···$\pi$ intramolecular interactions (Desiraju & Steiner, 1999) and the crystal packing is further stabilized by a C—H···O and three C—H···$\pi$ intermolecular interactions (Fig 2; Table 1). The centroids in detailed in Table 1 are identified as follows: *Cg*1 - ring C7/C70–72/C80; *Cg*2 - ring C95–100; *Cg*3 - ring C26–31; *Cg*4 - ring C72–76/80.

### S2. 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)methylidenecyclo-hexanone as yellow solid. The compound was recrystallized from a 1:1 mixture of methanol:ethyl acetate and a yellow solid is obtained, Yield 98%

### S3. 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 $U_{iso}$(H) = 1.2–1.5 $U_{eq}$ (parent atom).

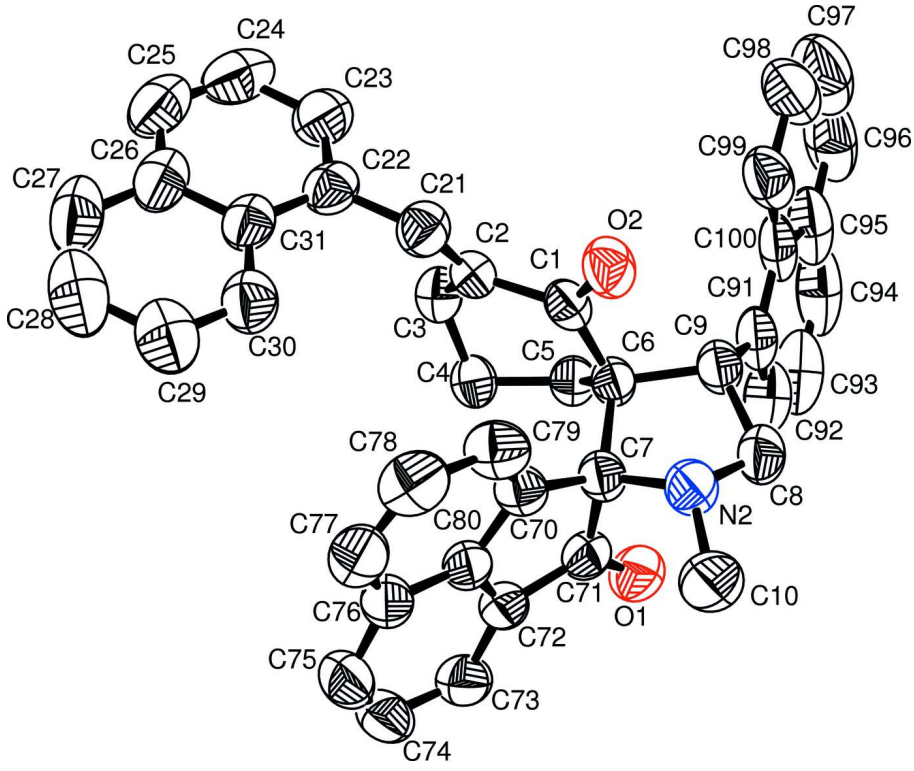


**Figure 1**

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.

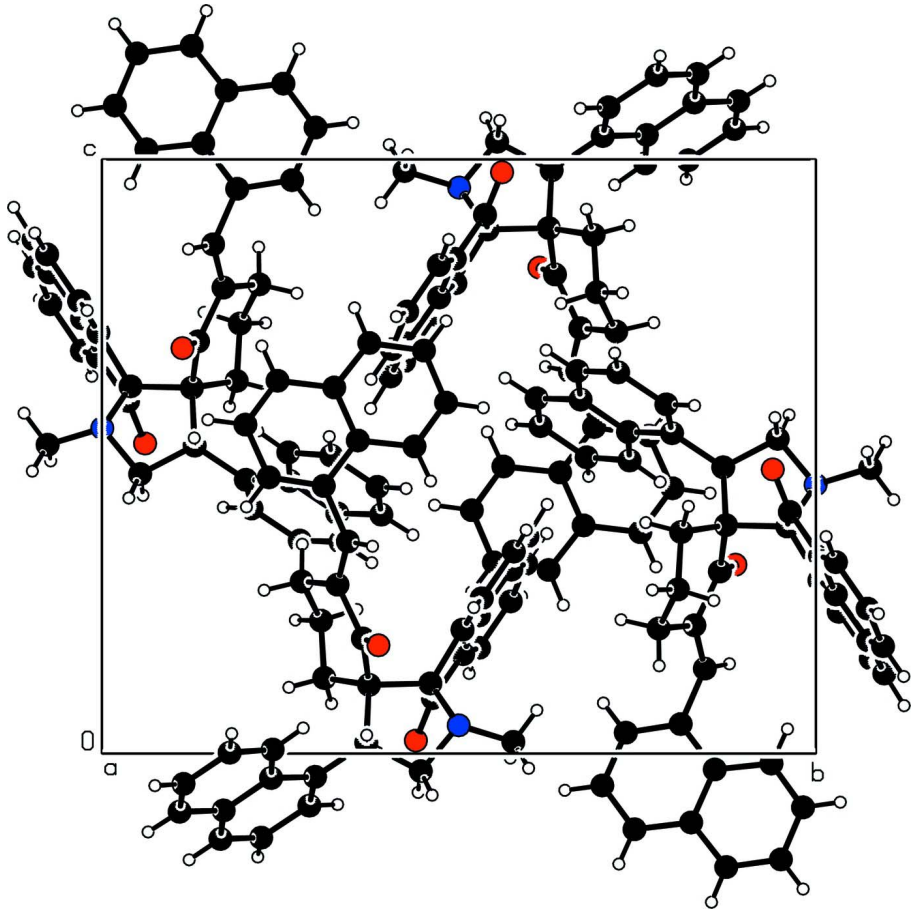


**Figure 2**

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

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*Crystal data*

$C_{42}H_{33}NO_2$
$M_r = 583.69$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.4398\ (8)$ Å
$b = 17.3501\ (11)$ Å
$c = 14.4685\ (9)$ Å
$\beta = 90.728\ (17)°$
$V = 3122.5\ (3)$ Å$^3$
$Z = 4$
$F(000) = 1232$
$D_x = 1.242$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.4–13.6°$
$\mu = 0.08$ mm$^{-1}$
$T = 293$ K
Block, pale yellow
0.22 × 0.18 × 0.16 mm

*Data collection*

Nonius MACH3 sealed tube diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$–$2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min} = 0.943$, $T_{max} = 0.986$
6169 measured reflections
5489 independent reflections

3098 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = 0 \rightarrow 14$
$k = -1 \rightarrow 20$
$l = -17 \rightarrow 17$
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
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
$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.7574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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) |
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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) |
| 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, º)*

| | | | |
|---|---|---|---|
| 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) |
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*B*···O1 | 0.97 | 2.37 | 3.084 (3) | 130 |
| C8—H8*A*···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—H4*A*···*Cg*1 | 0.97 | 2.64 | 3.337 (3) | 129 |

| | | | | |
|---|---|---|---|---|
| C75—H75···*Cg*2$^{ii}$ | 0.93 | 2.74 | 3.646 (3) | 164 |
| C78—H78···*Cg*3$^{iii}$ | 0.93 | 2.82 | 3.622 (4) | 145 |
| C96—H96···*Cg*4$^{iv}$ | 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.