



Crystal structure and molecular Hirshfeld surface analysis of acenaphthene derivatives obeying the chlorine–methyl exchange rule

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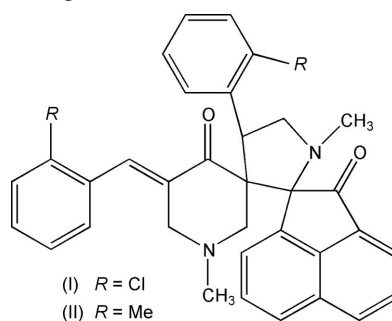
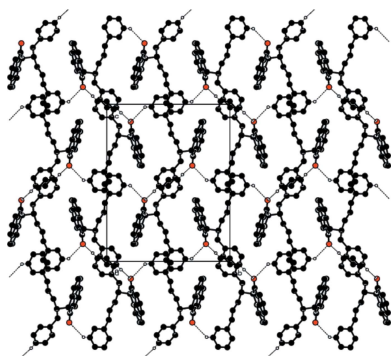
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Instances of crystal structures that remain isomorphous in spite of some minor changes in their respective molecules, such as change in a substituent atom/group, can provide insights into the factors that govern crystal packing. In this context, an accurate description of the crystal structures of an isomorphous pair that differ from each other only by a chlorine–methyl substituent, *viz.* 5''-(2-chlorobenzylidene)-4'-(2-chlorophenyl)-1'-methylspiro[acenaphthene-1,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione, C₃₄H₂₈Cl₂N₂O₂, (I), and its analogue 1'-methyl-5''-(2-methylbenzylidene)-4'-(2-methylphenyl)dispiro[acenaphthene-1,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione, C₃₆H₃₄N₂O₂, (II), is presented. While there are two C–H...O weak intermolecular interactions present in both (I) and (II), the change of substituent from chlorine to methyl has given rise to an additional weak C–H...O intermolecular interaction that is relatively stronger than the other two. However, the presence of the stronger C–H...O interaction in (II) has not disrupted the validity of the chloro–methyl exchange rule. Details of the crystal structures and Hirshfeld analyses of the two compounds are presented.

1. Chemical context

The prediction of crystal structures has emerged as an exciting field involving researchers from diverse fields primarily because of its challenging complexity, which is considered analogous to that of the protein-folding problem. Attempts made in the field of crystal-structure prediction, its present status and the challenges ahead were discussed in detail in a recent article (Oganov, 2018). In this context, instances of crystal structures that remain isomorphous in spite of some minor changes in their respective molecules, such as a change in a substituent atom/group, are worthy of study as they might provide some insights regarding the subtle factors that govern the crystal packing.



The title compounds (I) and (II) are good examples of crystal structures that obey the Cl–Me exchange rule,



Table 1
Selected torsion angles ($^{\circ}$) for (I).

O1–C1–C6–C7	–14.3 (2)	C12–C14–C19–C20	–0.9 (2)
C7–C8–C13–C11	–2.7 (3)	N2–C23–C24–O2	–51.3 (2)

complying with the general conclusions arrived at in earlier studies (Jones *et al.*, 1981; Gnanaguru *et al.*, 1984; Desiraju & Sarma, 1986). In some recent studies carried out in our laboratory on molecules that showcase the validity of the Cl–Me exchange rule, it has been observed that factors such as the presence of disorder and minor conformational differences have not disturbed the tendency of molecules to remain as isomorphous pairs (Rajni Swamy, *et al.*, 2013; Sribala *et al.*, 2018). Interestingly, the validity of the Cl–Me exchange rule has also been observed in some regularly shaped planar molecules (Nath & Nangia, 2012).

From a pharmacological view point, the title compounds (I) and (II) are spiro compounds that consist of a methylpyrrole moiety with its 2- and 3- positions as spiro carbons linked, respectively, to acenaphthene and methyl pyridinone ring systems. Each of these ring systems has a variety of associated biological properties. Studies on some 4-pyridone derivatives have shown them to be potent antimalarial agents (Bueno *et al.*, 2011) and effective in the treatment and prophylaxis of the hepatitis B virus infection (Cheng *et al.*, 2018). Acenaphthene is a pollutant known for its cytotoxicity (Jiang *et al.*, 2019) but is also useful as a dye intermediate. Derivatives of acenaphthene are found to exhibit antitumor (El-Ayaan *et al.*, 2007; Zhu *et al.*, 2008) and fungistatic properties (McDavids & Daniels, 1951). Pyrrole derivatives belong to an important class of heterocycles owing to their potential applications as

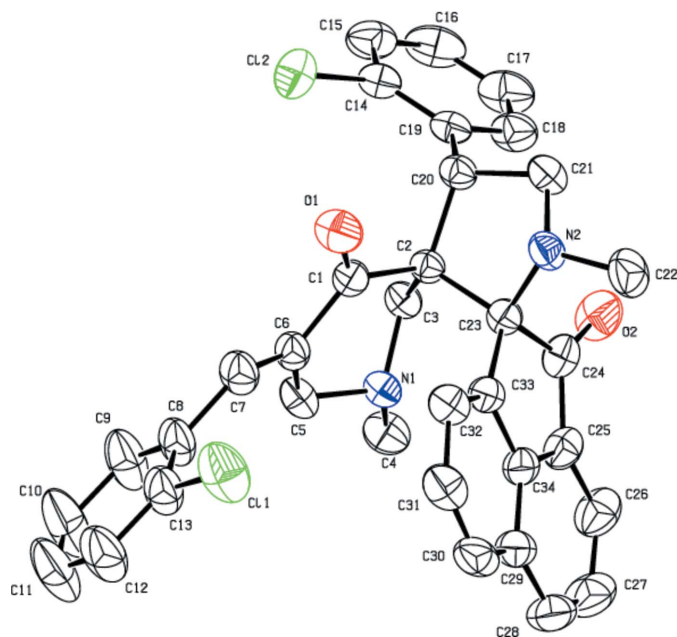


Figure 1
Displacement ellipsoid plot drawn at 50% probability level for (I) showing the atom-labelling scheme. H atoms have been omitted for clarity.

Table 2
Selected torsion angles ($^{\circ}$) for (II).

O1–C1–C6–C7	–12.8 (3)	C36–C14–C19–C20	–0.1 (3)
C7–C8–C13–C35	–3.6 (4)	N2–C23–C24–O2	–51.5 (2)

antimicrobial, antiviral, antimalarial, antitubercular, anti-inflammatory and anticancer agents (Gholap, 2016).

2. Structural commentary

The molecular structures of (I) and (II) (Figs. 1 and 2, respectively) differ from each other only by a chlorine atom in (I) being replaced by a methyl group in (II). This replacement has not induced any significant change in their unit-cell parameters, lattice type or space group. Similarly, there are no substantial changes in the torsion angles of the title compounds (see Tables 1 and 2), as (I) and (II) are isomorphous.

As expected, the conformational features of both compounds are nearly identical, as shown in a overlay diagram (Fig. 3). The five-membered pyrrole ring (N2/C21/C20/C2/C23) adopts an envelope conformation on N2 with puckering parameters $Q(2) = 0.4011(2) \text{ \AA}$ and $\varphi = 180.3733(3)^{\circ}$ for (I), which are comparable with the values of $Q(2) = 0.4047(2) \text{ \AA}$ and $\varphi = 180.3444(3)^{\circ}$ for (II). In both of the structures, the six-membered pyridinone ring (N1/C3/C2/C1/C6/C5) adopts a screw-boat conformation with puckering parameters $Q = 0.5572(16) \text{ \AA}$, $\theta = 138.9(2)^{\circ}$, $\varphi = 219.8(3)^{\circ}$ in (I) and $Q = 0.5603(17) \text{ \AA}$, $\theta = 137.7(2)^{\circ}$, $\varphi = 219.6(3)^{\circ}$ in (II). The acenaphthene ring system is planar in both (I) and (II). However, the O2 atom deviates from the mean plane of the

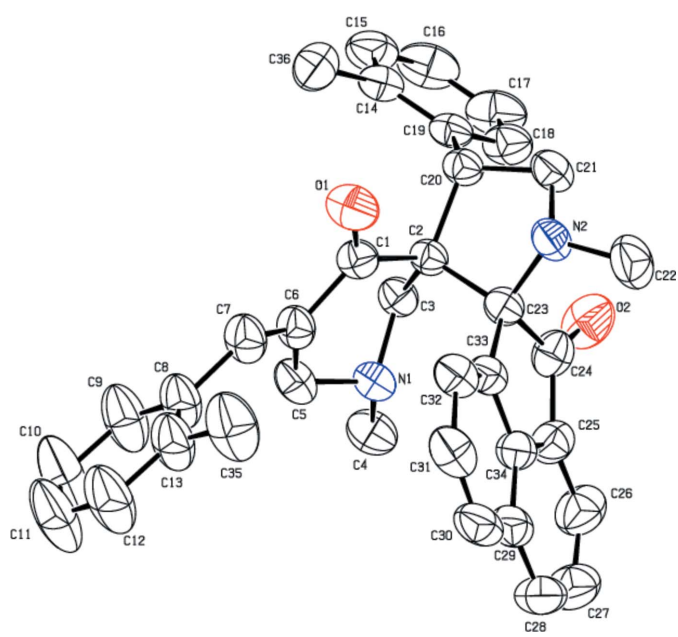


Figure 2
Displacement ellipsoid plot drawn at 50% probability level for (II), showing the atom-labelling scheme. H atoms have been omitted for clarity.

Table 3

Hydrogen-bond geometry (Å, °) for (I).

*Cg*1 is the centroid of the C25–C29/C34 ring.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C10–H10...O2 ⁱ	0.93	2.74	3.492 (3)	139
C16–H16...O2 ⁱⁱ	0.93	2.76	3.481 (3)	135
C5–H5B... <i>Cg</i> 1 ⁱ	0.97	2.99	3.9466 (19)	168

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

acenaphthene ring system by 0.289 (2) Å in (I) and 0.311 (2) Å in (II), with the r.m.s. deviation of the fitted atoms being 0.043 and 0.044, respectively. This deviation is presumably due to the fact that the O2 atom is involved in two weak C–H...O interactions that are characteristic of the molecular interaction patterns of both (I) and (II).

The dihedral angle between the mean planes of the two chlorophenyl groups in (I) is 67.66 (9)°, similarly the corresponding angle between the two methylphenyl groups in (II) is 66.78 (11)°. The dihedral angles between the acenaphthene ring system and the chlorophenyl groups are 69.1 (1) and 49.4 (1)°, respectively. The corresponding angles in the methyl-substituted analogue are 72.3 (1) and 47.8 (1)°, respectively. Thus, it is clear that the minor differences observed in the conformation of the molecules are insufficient to disrupt the tendency of these molecules to remain isomorphous.

3. Supramolecular features

There are no classical hydrogen bonds in the structures of either (I) or (II). However, in both structures two weak C–H...O-type intermolecular interactions, *viz.* C10–H10...O2 and C16–H16...O2, which are identical in nature and char-

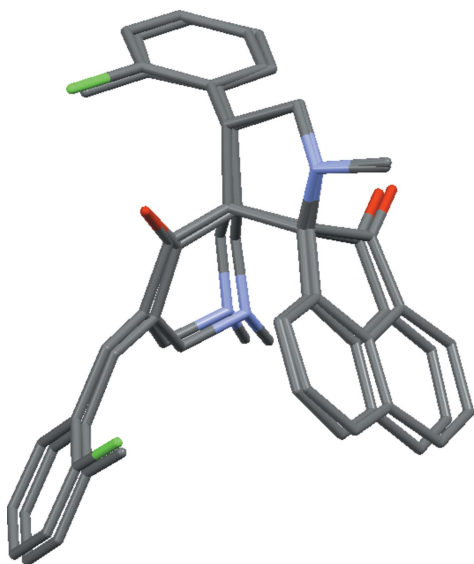


Figure 3

An overlay diagram depicting the superimposition of molecule (I) and (II) showing no differences in the conformations.

Table 4

Hydrogen-bond geometry (Å, °) for (II).

*Cg*2 is the centroid of the C8–C13 ring.

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C36–H36A...O1	0.96	2.66	3.586 (3)	161
C10–H10...O2 ⁱ	0.93	2.77	3.529 (3)	140
C16–H16...O2 ⁱⁱ	0.93	2.79	3.530 (3)	137
C35–H35F... <i>Cg</i> 2 ⁱⁱⁱ	0.96	2.94	3.805 (4)	151

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

acteristic of similar fundamental molecular interaction patterns are present (Tables 3 and 4). The C16–H16...O2 interaction occurs between centrosymmetric pairs (Fig. 4), leading to the formation of $R_2^2(20)$ graph-set motifs along the *b*-axis direction in both (I) and (II). Similarly, in both (I) and (II) the C10–H10...O2 interaction links glide-related molecules along the *b*-axis direction (Fig. 5). The molecular aggregation pattern may be visualized as being composed of these two characteristic weak interactions in such a manner that centrosymmetric dimeric pairs are linked through glide-

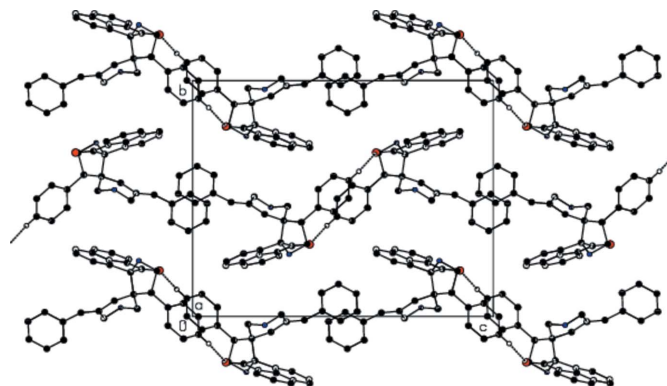


Figure 4

Perspective view along the *a* axis showing the weak C16–H16...O2 intermolecular interactions between centrosymmetric pairs of molecules in (I) and (II). Non-participating H atoms, methyl C atoms and Cl atoms have been omitted for clarity.

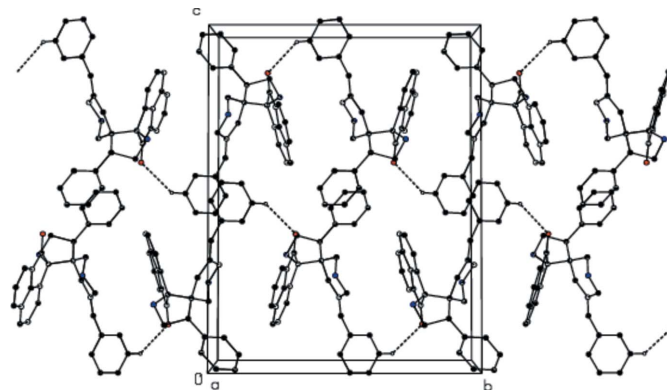


Figure 5

Perspective view along the *a* axis showing the weak C10–H10...O2 intermolecular interactions between glide-related molecules in (I) and (II). Non-participating H atoms, methyl C atoms and Cl atoms have been omitted for clarity.

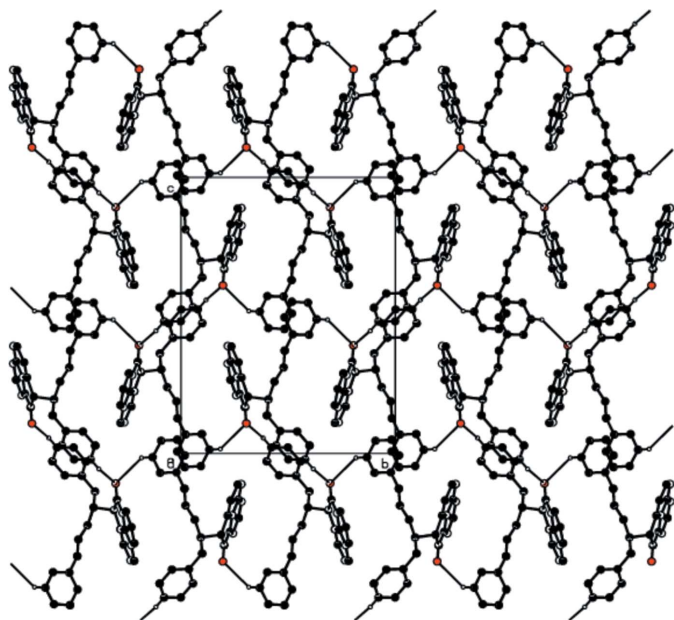


Figure 6

A view of the crystal structure of (I) showing the formation of $R_2^2(20)$ graph-set motifs leading to the formation of layers formed parallel to the bc plane. Dashed lines indicate weak C—H \cdots O intermolecular interactions. H atoms not involved in the interactions have been omitted for clarity.

related chains of molecules, forming a two-dimensional layer parallel to the bc plane in both structures, as shown in Figs. 6 and 7, respectively.

In (II), an additional intermolecular interaction is observed, *viz.* C36—H36A \cdots O1, that is stronger than the two characteristic weak intermolecular interactions and involves the replaced substituent methyl group (C36—H36A) as a donor and the piperidinone O1 atom as an acceptor (see Table 4). It may be concluded that the presence of this additional C—H \cdots O interaction in (II) has not disrupted the validity of the chloro—methyl exchange rule.

In addition, a weak C—H \cdots π interaction involving different donor groups and acceptor π -ring systems is present in both (I) and (II). The C5—H5B \cdots π interaction observed in (I) is between the C5 atom of the methylpiperidinone ring as a donor and the C25—C29/C34 ring of the acenaphthenone system as an acceptor. Interestingly, a geometrically identical weak π — π interaction about an inversion centre is observed with centroid—centroid Cg3 \cdots Cg3(1 - x , 2 - y , - z) distances of 3.7459 (2) Å in (I) and 3.8351 (2) Å in (II) with respective slippages of 1.250 and 1.367 Å where Cg3 is the centroid of the C14—C19 ring. The shortest Cl \cdots Cl distance observed [Cl1 \cdots Cl1(- x + 1, - y + 2, - z + 1)] is 4.088 (1) Å and bears no structural significance.

4. Database survey

A thorough search in the Cambridge Structural Database (CSD Version 5.39, update Nov 2017; Groom *et al.*, 2016) using the main skeleton of the title compounds (having 3D coordi-

nates with no disorder, no ions and no other errors with R factors less than 0.05) gave only three hits: 5''-(4-chlorobenzylidene)-4''-(4-chlorophenyl)-1',1''-dimethyl-2*H*,4''*H*-dispiro[acenaphthylene-1,2''-pyrrolidine-3',3''-piperidine]-2,4''-dione (YIRKUG; Pandiarajan *et al.*, 2008), 5''-benzylidene-1',1''-dimethyl-4''-phenyl-acenaphthene-2-spiro-2''-pyrrolidine-3'-spiro-3''-piperidine-1,4''-dione (MAJHEL; Aravindan *et al.*, 2004) and 1-methyl-4-(4-methylphenyl)pyrrolo-(spiro[2.2'']-acenaphthene-1''-one)-spiro-[3.3']-(spiro-[5'.5''']-3'''-(4-chlorophenyl)-4'''-(4-methylphenyl)-isoxazoline)-1'-methyl-tetrahydro-4'(1*H*)-pyridinone (XUQFOF; Kumar, *et al.*, 2009).

5. Hirshfeld surface analysis

Hirshfeld surface (HS) analysis was used to investigate and visualize the weak intermolecular interactions influential in the packing of the molecules in the crystal. The visual representation of molecular interactions on this isosurface is determined using two parameters, *viz.* d_i and d_e , which represent the distances from a given point on the surface to the nearest atom inside and outside the surface, respectively. The normalized contact distance, d_{norm} is based on the values of d_i and d_e .

In the present work, the Hirshfeld surfaces (Spackman & Jayatilaka, 2009) and the associated two-dimensional fingerprint plots for title compounds (I) and (II) were generated using *CrystalExplorer3.0* (Wolff *et al.*, 2012). The Hirshfeld surfaces mapped over d_{norm} together with decomposed fingerprint plots (McKinnon *et al.*, 2007; Tan *et al.*, 2019) for (I) and (II) are presented in Figs. 8 and 9, respectively. Being isomorphous and isostructural in nature, both (I) and (II)

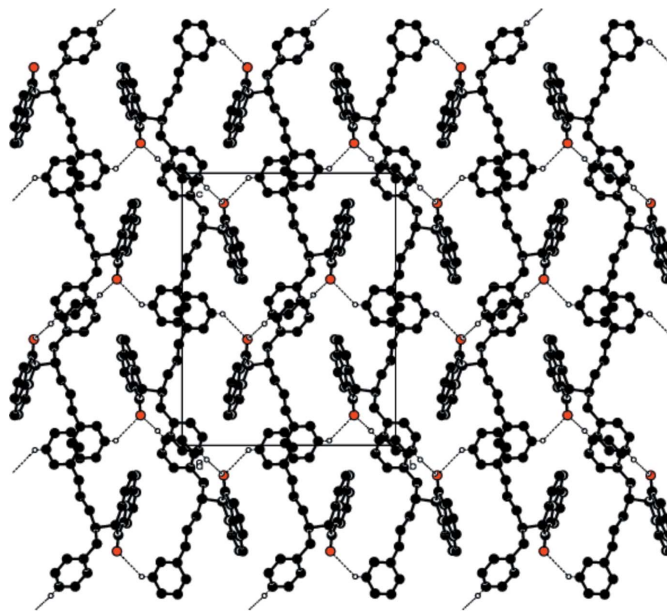


Figure 7

A view of the crystal structure of (II) showing the $R_2^2(20)$ graph-set motifs. Dashed lines indicate weak C—H \cdots O intermolecular interactions. H atoms not involved in the interactions have been omitted for clarity.

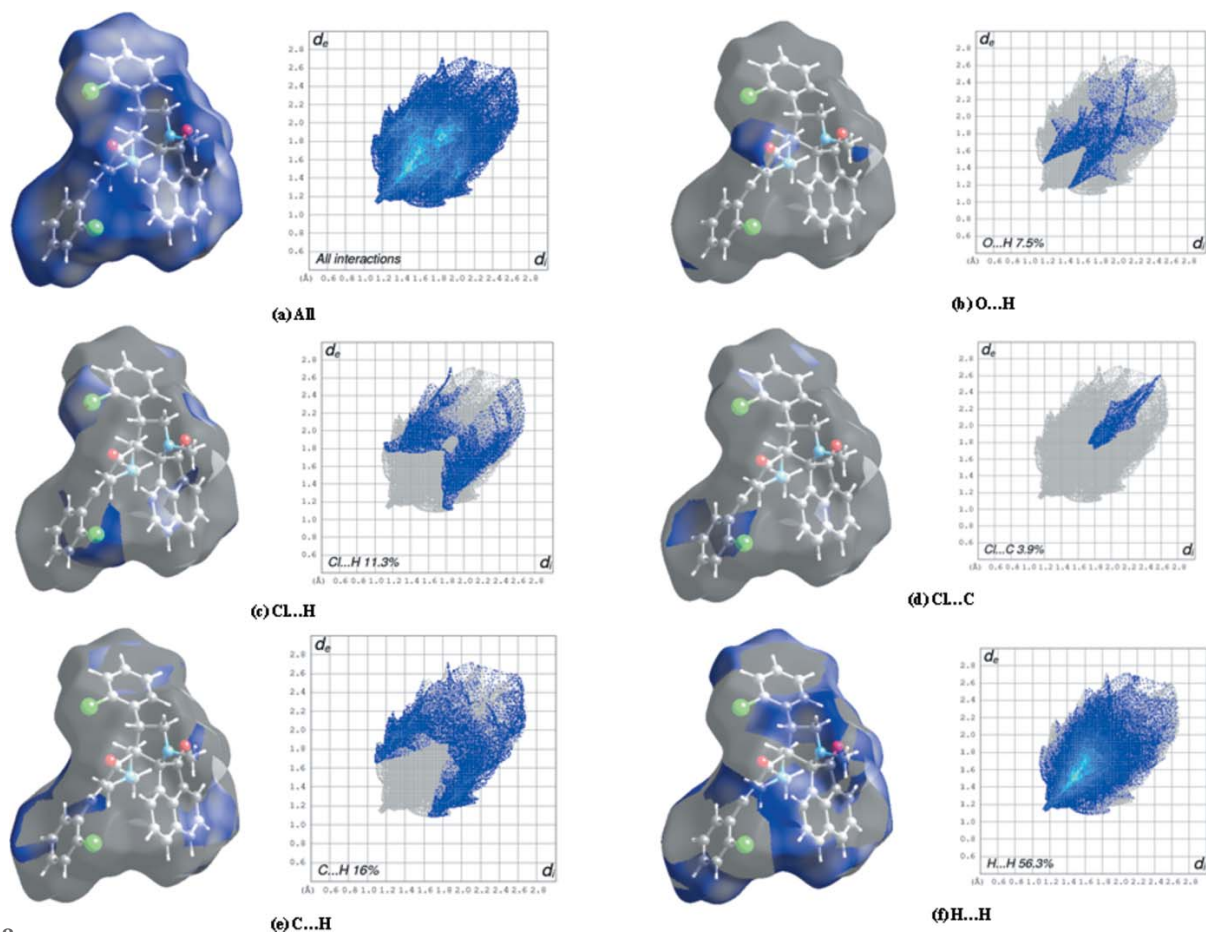


Figure 8 Hirshfeld surface of (I) mapped over shape-index and d_{norm} and decomposed fingerprint plots of the dominant interactions.

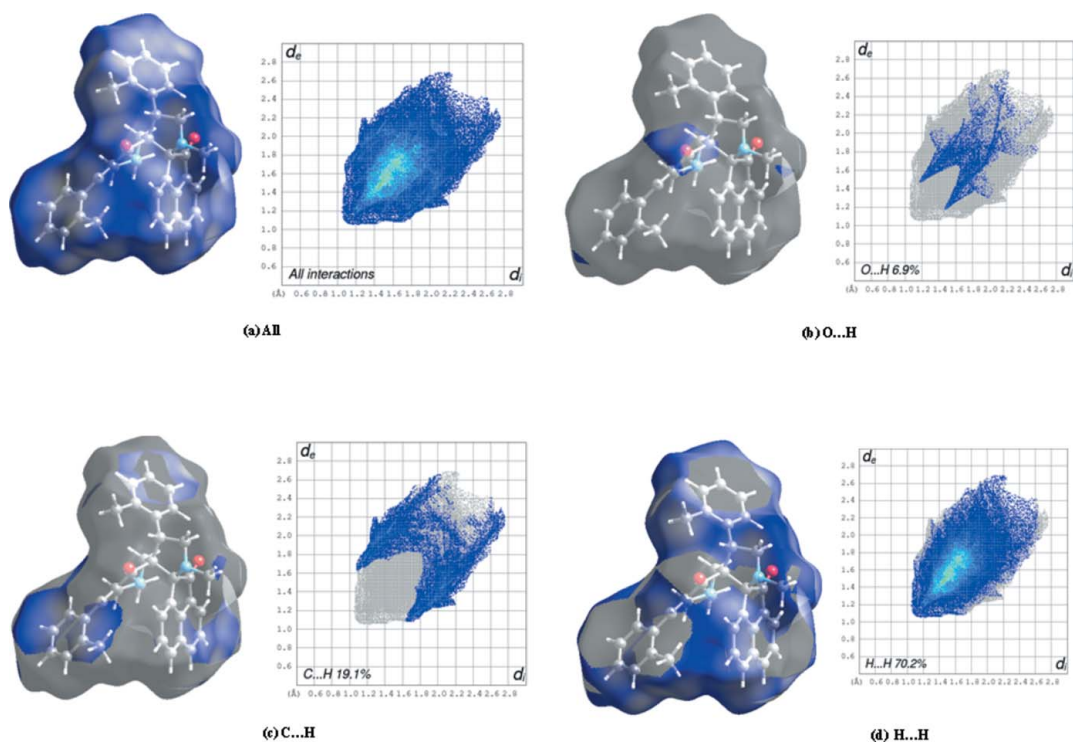


Figure 9 Hirshfeld surface of (II) mapped over shape-index and d_{norm} and decomposed fingerprint plots of the dominant interactions.

Table 5
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C ₃₄ H ₂₈ Cl ₂ N ₂ O ₂	C ₃₆ H ₃₄ N ₂ O ₂
<i>M_r</i>	567.48	526.65
Crystal system, space group	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/c</i>
Temperature (K)	293	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.6710 (4), 15.6756 (7), 20.2284 (9)	8.7507 (5), 15.9089 (8), 20.2879 (10)
β (°)	93.036 (2)	92.935 (2)
<i>V</i> (Å ³)	2745.6 (2)	2820.7 (3)
<i>Z</i>	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	0.27	0.08
Crystal size (mm)	0.31 × 0.22 × 0.19	0.32 × 0.24 × 0.18
Data collection		
Diffractometer	Bruker SMART APEXII CCD	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2001)	Multi-scan (<i>SADABS</i> ; Bruker, 2001)
<i>T_{min}</i> , <i>T_{max}</i>	0.771, 1.000	0.816, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	33739, 7274, 5025	28946, 5339, 3349
<i>R_{int}</i>	0.026	0.040
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.683	0.610
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.044, 0.122, 1.02	0.045, 0.121, 1.02
No. of reflections	7274	5339
No. of parameters	361	362
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.33, -0.46	0.17, -0.15

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXS2013* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

display similar C—H···O intermolecular interactions. The combined O···H and H···O interactions appear symmetrically as distinct spikes at the bottom of the fingerprint plot and contribute 7.5 and 6.9%, respectively, of the total surface in compounds (I) and (II).

The symmetrical internal wing-like projections correspond to C···H/H···C contacts, which account for 16% of the HS in (I) and 19.1% in (II). The dominant contribution is from the H···H contacts [56.3% in (I) and 70.2% in (II)], as shown by the area occupied between the spikes. Such prominent differences may be accounted for by the presence of a Cl···H/H···Cl contact in (I) (11.3% contribution) and its absence in (II).

6. Synthesis and crystallization

For (I), a mixture of 1-methyl-3,5-bis[(*E*)-2-chlorophenylmethylidene] tetrahydro-4(1*H*)-pyridinone (1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) was dissolved in methanol (15 mL) and refluxed for 30 min. After completion of the reaction, as evident from TLC, the mixture was poured into water (50 mL) and the precipitated solid was filtered and washed with water (100 mL) to obtain pure (I) as a yellow solid, (0.31 g, 98%), mp 448–449 K, *R_f* (petroleum ether/EtOAc, 4:1) 0.40. Suitable crystals for single-crystal X-ray studies were obtained by recrystallization of the product from ethanol.

A similar procedure for (II) was adopted by dissolving a mixture of 1-methyl-3,5-bis[(*E*)-2-methylphenylmethylidene]

tetrahydro-4(1*H*)-pyridinone (1 mmol), acenaphthenequinone (1 mmol) and sarcosine (1 mmol) in methanol (15 mL) to yield yellow crystals.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. C-bound H atoms were included in calculated positions and treated as riding, with C—H = 0.95–1.00 Å and *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H atoms or 1.2*U*_{eq}(C) otherwise. The H atoms of the methyl atoms C35 and C36 in (II) were refined as idealized and disordered over two positions since significant residual electron densities were noticed between the three hydrogen atoms of the respective methyl C atoms. The introduction of a disordered model for these two methyl groups had appreciable impact on the final structural parameters.

Acknowledgements

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References

- Aravindan, P. G., Selvanayagam, S., Velmurugan, D., Ravikumar, K., Sridhar, G. & Raghunathan, R. (2004). *Acta Cryst.* E60, o2149–o2151.

- Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2012). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bueno, J. M., Manzano, P., García, M. C., Chicharro, J., Puente, M., Lorenzo, M., García, A., Ferrer, S., Gómez, R. M., Fraile, M. T., Lavandera, J. L., Fiandor, J. M., Vidal, J., Herreros, E. & Gargallo-Viola, D. (2011). *Bioorg. Med. Chem. Lett.* **21**, 5214–5218.
- Cheng, Z., Han, X., Liang, C. & Yang, S. (2018). US Patent Pub. No. US2018/0134705A1.
- Desiraju, G. R. & Sarma, J. A. R. P. (1986). *Proc. - Indian Acad. Sci. Chem. Sci.* **96**, 599–605.
- El-Ayaan, U., Abdel-Aziz, A. A.-M. & Al-Shihry, S. (2007). *Eur. J. Med. Chem.* **42**, 1325–1333.
- Gholap, S. S. (2016). *Eur. J. Med. Chem.* **110**, 13–31.
- Gnanaguru, K., Murthy, G. S., Venkatesan, K. & Ramamurthy, V. (1984). *Chem. Phys. Lett.* **109**, 255–258.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Jiang, L., Zhang, B., Wang, Y., Sun, J., Ma, X., Wang, G., Fu, S., Lin, C. & Li, Y. (2019). *Nat. Prod. Res.* <https://doi.org/10.1080/14786419.2019.1647422>
- Jones, W., Ramdas, S., Theocharis, C. R., Thomas, J. M. & Thomas, N. W. (1981). *J. Phys. Chem.* **85**, 2594–2597.
- Kumar, R. R., Loganayaki, B. & Perumal, S. (2009). *Synth. Commun.* **39**, 3197–3216.
- McDavids, J. E. & Daniels, T. C. (1951). *J. Am. Pharm. Assoc. (Sci. ed.)*, **40**, 325–326.
- McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.
- Nath, N. K. & Nangia, A. (2012). *Cryst. Growth Des.* **12**, 5411–5425.
- Oganov, A. R. (2018). *Faraday Discuss.* **211**, 643–660.
- Pandiarajan, S., Saravanamoorthy, S. N., Kumar, B. R., Kumar, R. R. & Athimoolam, S. (2008). *Acta Cryst.* **E64**, o99–o100.
- Rajni Swamy, V., Müller, P., Srinivasan, N., Perumal, S. & Krishnakumar, R. V. (2013). *Acta Cryst.* **C69**, 412–415.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Sribala, R., Srinivasan, N., Indumathi, S. & Krishnakumar, R. V. (2018). *Acta Cryst.* **E74**, 1267–1271.
- Tan, S. L., Jotani, M. M. & Tiekink, E. R. T. (2019). *Acta Cryst.* **E75**, 308–318.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Wolff, S. K., Grimwood, D. J., McKinnon, J. J., Turner, M. J., Jayatilaka, D. & Spackman, M. A. (2012). *Crystal Explorer*, University of Western Australia.
- Zhu, W., Dai, M., Xu, Y. & Qian, X. (2008). *Bioorg. Med. Chem.* **16**, 3255–3260.

supporting information

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Crystal structure and molecular Hirshfeld surface analysis of acenaphthene derivatives obeying the chlorine–methyl exchange rule

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINTE* (Bruker, 2012); data reduction: *SAINTE* (Bruker, 2012); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

5''-(2-Chlorobenzylidene)-4'-(2-chlorophenyl)-1'-methylspiro[acenaphthene-1,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione (I)

Crystal data

C₃₄H₂₈Cl₂N₂O₂

M_r = 567.48

Monoclinic, *P*2₁/*c*

a = 8.6710 (4) Å

b = 15.6756 (7) Å

c = 20.2284 (9) Å

β = 93.036 (2)°

V = 2745.6 (2) Å³

Z = 4

F(000) = 1184

D_x = 1.373 Mg m⁻³

D_m = 1.37 Mg m⁻³

D_m measured by floatation method

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 7274 reflections

θ = 4.8–57.2°

μ = 0.27 mm⁻¹

T = 293 K

Block, yellow

0.31 × 0.22 × 0.19 mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2001)

T_{min} = 0.771, *T_{max}* = 1.000

33739 measured reflections

7274 independent reflections

5025 reflections with *I* > 2σ(*I*)

R_{int} = 0.026

θ_{max} = 29.1°, θ_{min} = 2.4°

h = -11→11

k = -21→21

l = -27→27

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.044

wR(*F*²) = 0.122

S = 1.01

7274 reflections

361 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0458*P*)² + 1.2817*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.33 e Å⁻³

Δρ_{min} = -0.46 e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.38200 (9)	0.88710 (4)	0.49943 (3)	0.0749 (2)
C12	0.53437 (6)	1.07386 (3)	0.14315 (3)	0.05500 (14)
O1	0.48210 (14)	0.92211 (9)	0.26952 (6)	0.0509 (3)
O2	0.01681 (16)	0.80480 (10)	0.10859 (7)	0.0587 (4)
N1	0.02281 (15)	0.96435 (9)	0.23726 (6)	0.0345 (3)
N2	0.32804 (16)	0.76391 (9)	0.17821 (7)	0.0379 (3)
C1	0.34543 (18)	0.93585 (10)	0.26001 (8)	0.0339 (3)
C2	0.25834 (17)	0.90781 (10)	0.19571 (7)	0.0298 (3)
C3	0.12354 (18)	0.96919 (10)	0.18237 (8)	0.0330 (3)
H3A	0.161841	1.026946	0.177987	0.040*
H3B	0.066923	0.953767	0.141473	0.040*
C4	-0.1310 (2)	0.99694 (13)	0.21990 (10)	0.0486 (4)
H4A	-0.175090	0.965861	0.182623	0.073*
H4B	-0.194961	0.990171	0.256864	0.073*
H4C	-0.124570	1.056321	0.208758	0.073*
C5	0.0931 (2)	1.00991 (11)	0.29351 (8)	0.0412 (4)
H5A	0.029809	1.002742	0.331249	0.049*
H5B	0.096552	1.070296	0.283217	0.049*
C6	0.2542 (2)	0.97887 (10)	0.31150 (8)	0.0367 (3)
C7	0.3209 (2)	0.98384 (12)	0.37223 (9)	0.0453 (4)
H7	0.419755	0.961011	0.377724	0.054*
C8	0.2571 (2)	1.02102 (12)	0.43141 (9)	0.0498 (5)
C9	0.1760 (3)	1.09734 (15)	0.42992 (11)	0.0700 (7)
H9	0.157858	1.125378	0.389746	0.084*
C10	0.1219 (4)	1.13245 (17)	0.48645 (13)	0.0900 (9)
H10	0.067945	1.183728	0.484082	0.108*
C11	0.1467 (4)	1.09265 (18)	0.54595 (12)	0.0925 (10)
H11	0.109239	1.116503	0.584021	0.111*
C12	0.2269 (3)	1.01755 (16)	0.54950 (11)	0.0761 (7)
H12	0.244309	0.990238	0.590018	0.091*
C13	0.2817 (3)	0.98246 (13)	0.49292 (9)	0.0546 (5)
C14	0.40575 (19)	1.04221 (11)	0.07931 (8)	0.0395 (4)
C15	0.3746 (2)	1.09964 (13)	0.02837 (10)	0.0524 (5)
H15	0.424605	1.152112	0.028284	0.063*
C16	0.2695 (3)	1.07856 (15)	-0.02190 (10)	0.0604 (6)
H16	0.247308	1.116892	-0.056185	0.073*
C17	0.1973 (2)	1.00115 (16)	-0.02157 (10)	0.0589 (6)
H17	0.125775	0.986900	-0.055648	0.071*
C18	0.2301 (2)	0.94401 (13)	0.02908 (9)	0.0475 (4)

H18	0.180273	0.891469	0.028376	0.057*
C19	0.33571 (18)	0.96284 (11)	0.08114 (8)	0.0355 (3)
C20	0.37056 (18)	0.90127 (10)	0.13774 (7)	0.0333 (3)
H20	0.474518	0.914578	0.156241	0.040*
C21	0.3698 (2)	0.80722 (11)	0.11831 (9)	0.0418 (4)
H21A	0.294485	0.796255	0.082138	0.050*
H21B	0.470866	0.789228	0.105323	0.050*
C22	0.3020 (3)	0.67327 (12)	0.17056 (11)	0.0576 (5)
H22A	0.275049	0.649399	0.212106	0.086*
H22B	0.219323	0.663728	0.137946	0.086*
H22C	0.394350	0.646490	0.156564	0.086*
C23	0.20239 (17)	0.81269 (10)	0.20545 (8)	0.0322 (3)
C24	0.0411 (2)	0.79856 (11)	0.16774 (9)	0.0410 (4)
C25	-0.06990 (19)	0.77207 (11)	0.21615 (10)	0.0429 (4)
C26	-0.2263 (2)	0.75748 (13)	0.21054 (12)	0.0572 (5)
H26	-0.281931	0.764906	0.170370	0.069*
C27	-0.2990 (2)	0.73129 (15)	0.26675 (14)	0.0684 (7)
H27	-0.405325	0.722892	0.264134	0.082*
C28	-0.2189 (3)	0.71761 (13)	0.32545 (13)	0.0628 (6)
H28	-0.271567	0.698729	0.361561	0.075*
C29	-0.0578 (2)	0.73134 (11)	0.33300 (10)	0.0461 (4)
C30	0.0409 (3)	0.71831 (12)	0.38919 (10)	0.0526 (5)
H30	0.001188	0.698030	0.428006	0.063*
C31	0.1947 (2)	0.73521 (12)	0.38725 (9)	0.0496 (5)
H31	0.258210	0.724857	0.424875	0.060*
C32	0.2616 (2)	0.76802 (11)	0.32996 (9)	0.0410 (4)
H32	0.366809	0.779746	0.330281	0.049*
C33	0.16946 (18)	0.78193 (10)	0.27480 (8)	0.0339 (3)
C34	0.01184 (19)	0.76132 (10)	0.27645 (9)	0.0379 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1164 (5)	0.0651 (4)	0.0426 (3)	0.0162 (3)	-0.0028 (3)	0.0052 (2)
Cl2	0.0520 (3)	0.0486 (3)	0.0634 (3)	-0.0065 (2)	-0.0062 (2)	-0.0027 (2)
O1	0.0375 (6)	0.0762 (9)	0.0382 (7)	0.0023 (6)	-0.0059 (5)	-0.0024 (6)
O2	0.0606 (8)	0.0712 (10)	0.0422 (8)	-0.0164 (7)	-0.0156 (6)	0.0065 (7)
N1	0.0344 (7)	0.0376 (7)	0.0316 (7)	0.0049 (5)	0.0030 (5)	-0.0007 (6)
N2	0.0460 (8)	0.0329 (7)	0.0352 (8)	0.0044 (6)	0.0067 (6)	-0.0027 (6)
C1	0.0384 (8)	0.0354 (8)	0.0275 (8)	-0.0033 (6)	-0.0013 (6)	0.0035 (6)
C2	0.0309 (7)	0.0335 (8)	0.0252 (7)	0.0007 (6)	0.0013 (6)	0.0008 (6)
C3	0.0365 (8)	0.0355 (8)	0.0268 (8)	0.0039 (6)	-0.0005 (6)	0.0016 (6)
C4	0.0411 (9)	0.0537 (11)	0.0511 (11)	0.0131 (8)	0.0035 (8)	0.0005 (9)
C5	0.0517 (10)	0.0408 (9)	0.0315 (9)	0.0077 (7)	0.0039 (7)	-0.0038 (7)
C6	0.0465 (9)	0.0344 (8)	0.0291 (8)	-0.0030 (7)	0.0010 (7)	-0.0008 (6)
C7	0.0571 (11)	0.0466 (10)	0.0318 (9)	-0.0019 (8)	-0.0023 (8)	-0.0012 (7)
C8	0.0732 (13)	0.0468 (10)	0.0288 (9)	-0.0039 (9)	-0.0025 (8)	-0.0063 (8)
C9	0.117 (2)	0.0542 (13)	0.0381 (11)	0.0107 (13)	-0.0047 (12)	-0.0076 (9)

C10	0.153 (3)	0.0611 (15)	0.0555 (15)	0.0301 (16)	0.0057 (16)	-0.0159 (12)
C11	0.163 (3)	0.0710 (17)	0.0448 (14)	0.0170 (18)	0.0209 (16)	-0.0168 (12)
C12	0.133 (2)	0.0629 (14)	0.0337 (11)	0.0001 (15)	0.0128 (13)	-0.0019 (10)
C13	0.0821 (14)	0.0460 (11)	0.0356 (10)	-0.0045 (10)	0.0017 (9)	-0.0034 (8)
C14	0.0358 (8)	0.0465 (9)	0.0369 (9)	0.0042 (7)	0.0078 (7)	0.0023 (7)
C15	0.0570 (11)	0.0509 (11)	0.0507 (12)	0.0045 (9)	0.0165 (9)	0.0135 (9)
C16	0.0635 (13)	0.0777 (15)	0.0409 (11)	0.0173 (11)	0.0104 (9)	0.0262 (10)
C17	0.0525 (11)	0.0899 (17)	0.0337 (10)	0.0036 (11)	-0.0023 (8)	0.0126 (10)
C18	0.0463 (10)	0.0642 (12)	0.0319 (9)	-0.0031 (8)	0.0010 (7)	0.0051 (8)
C19	0.0337 (8)	0.0459 (9)	0.0275 (8)	0.0032 (7)	0.0065 (6)	0.0027 (7)
C20	0.0324 (7)	0.0403 (8)	0.0273 (8)	0.0011 (6)	0.0013 (6)	0.0002 (6)
C21	0.0485 (9)	0.0432 (9)	0.0343 (9)	0.0054 (7)	0.0075 (7)	-0.0040 (7)
C22	0.0804 (14)	0.0358 (10)	0.0577 (13)	0.0030 (9)	0.0133 (11)	-0.0059 (9)
C23	0.0335 (7)	0.0336 (8)	0.0292 (8)	0.0001 (6)	-0.0006 (6)	0.0014 (6)
C24	0.0413 (9)	0.0364 (9)	0.0443 (10)	-0.0033 (7)	-0.0059 (7)	0.0012 (7)
C25	0.0367 (8)	0.0342 (9)	0.0574 (11)	-0.0009 (7)	-0.0002 (8)	0.0006 (8)
C26	0.0391 (9)	0.0519 (11)	0.0798 (15)	-0.0016 (8)	-0.0034 (10)	-0.0004 (10)
C27	0.0395 (10)	0.0635 (14)	0.104 (2)	-0.0058 (9)	0.0173 (12)	-0.0037 (13)
C28	0.0549 (12)	0.0518 (12)	0.0851 (17)	-0.0068 (9)	0.0344 (12)	-0.0017 (11)
C29	0.0539 (10)	0.0309 (8)	0.0555 (12)	0.0016 (7)	0.0216 (9)	0.0003 (8)
C30	0.0744 (13)	0.0377 (9)	0.0482 (11)	0.0050 (9)	0.0253 (10)	0.0054 (8)
C31	0.0711 (13)	0.0406 (10)	0.0374 (10)	0.0110 (9)	0.0051 (9)	0.0074 (8)
C32	0.0461 (9)	0.0382 (9)	0.0389 (9)	0.0067 (7)	0.0033 (7)	0.0048 (7)
C33	0.0377 (8)	0.0288 (7)	0.0355 (9)	0.0029 (6)	0.0049 (6)	0.0026 (6)
C34	0.0403 (8)	0.0261 (7)	0.0479 (10)	0.0012 (6)	0.0090 (7)	0.0000 (7)

Geometric parameters (Å, °)

C11—C13	1.731 (2)	C14—C19	1.386 (2)
C12—C14	1.7336 (18)	C15—C16	1.369 (3)
O1—C1	1.2098 (19)	C15—H15	0.9300
O2—C24	1.208 (2)	C16—C17	1.365 (3)
N1—C5	1.450 (2)	C16—H16	0.9300
N1—C3	1.4506 (19)	C17—C18	1.379 (3)
N1—C4	1.454 (2)	C17—H17	0.9300
N2—C22	1.446 (2)	C18—C19	1.390 (2)
N2—C21	1.451 (2)	C18—H18	0.9300
N2—C23	1.462 (2)	C19—C20	1.516 (2)
C1—C6	1.501 (2)	C20—C21	1.526 (2)
C1—C2	1.533 (2)	C20—H20	0.9800
C2—C3	1.527 (2)	C21—H21A	0.9700
C2—C20	1.566 (2)	C21—H21B	0.9700
C2—C23	1.584 (2)	C22—H22A	0.9600
C3—H3A	0.9700	C22—H22B	0.9600
C3—H3B	0.9700	C22—H22C	0.9600
C4—H4A	0.9600	C23—C33	1.525 (2)
C4—H4B	0.9600	C23—C24	1.573 (2)
C4—H4C	0.9600	C24—C25	1.469 (3)

C5—C6	1.505 (2)	C25—C26	1.374 (2)
C5—H5A	0.9700	C25—C34	1.388 (3)
C5—H5B	0.9700	C26—C27	1.391 (3)
C6—C7	1.332 (2)	C26—H26	0.9300
C7—C8	1.466 (3)	C27—C28	1.360 (3)
C7—H7	0.9300	C27—H27	0.9300
C8—C9	1.387 (3)	C28—C29	1.414 (3)
C8—C13	1.390 (3)	C28—H28	0.9300
C9—C10	1.374 (3)	C29—C30	1.401 (3)
C9—H9	0.9300	C29—C34	1.402 (2)
C10—C11	1.363 (4)	C30—C31	1.362 (3)
C10—H10	0.9300	C30—H30	0.9300
C11—C12	1.367 (4)	C31—C32	1.420 (2)
C11—H11	0.9300	C31—H31	0.9300
C12—C13	1.377 (3)	C32—C33	1.355 (2)
C12—H12	0.9300	C32—H32	0.9300
C14—C15	1.384 (3)	C33—C34	1.406 (2)
C5—N1—C3	109.25 (13)	C16—C17—C18	120.3 (2)
C5—N1—C4	111.01 (13)	C16—C17—H17	119.9
C3—N1—C4	112.25 (13)	C18—C17—H17	119.9
C22—N2—C21	114.51 (14)	C17—C18—C19	121.78 (19)
C22—N2—C23	116.03 (14)	C17—C18—H18	119.1
C21—N2—C23	107.08 (13)	C19—C18—H18	119.1
O1—C1—C6	121.17 (15)	C14—C19—C18	116.24 (16)
O1—C1—C2	121.21 (15)	C14—C19—C20	121.46 (15)
C6—C1—C2	117.60 (13)	C18—C19—C20	122.29 (16)
C3—C2—C1	107.89 (12)	C19—C20—C21	115.01 (13)
C3—C2—C20	114.15 (12)	C19—C20—C2	114.64 (12)
C1—C2—C20	110.87 (12)	C21—C20—C2	105.17 (13)
C3—C2—C23	112.25 (12)	C19—C20—H20	107.2
C1—C2—C23	107.74 (12)	C21—C20—H20	107.2
C20—C2—C23	103.77 (12)	C2—C20—H20	107.2
N1—C3—C2	108.55 (12)	N2—C21—C20	103.58 (13)
N1—C3—H3A	110.0	N2—C21—H21A	111.0
C2—C3—H3A	110.0	C20—C21—H21A	111.0
N1—C3—H3B	110.0	N2—C21—H21B	111.0
C2—C3—H3B	110.0	C20—C21—H21B	111.0
H3A—C3—H3B	108.4	H21A—C21—H21B	109.0
N1—C4—H4A	109.5	N2—C22—H22A	109.5
N1—C4—H4B	109.5	N2—C22—H22B	109.5
H4A—C4—H4B	109.5	H22A—C22—H22B	109.5
N1—C4—H4C	109.5	N2—C22—H22C	109.5
H4A—C4—H4C	109.5	H22A—C22—H22C	109.5
H4B—C4—H4C	109.5	H22B—C22—H22C	109.5
N1—C5—C6	112.12 (13)	N2—C23—C33	111.16 (12)
N1—C5—H5A	109.2	N2—C23—C24	113.88 (13)
C6—C5—H5A	109.2	C33—C23—C24	101.34 (12)

N1—C5—H5B	109.2	N2—C23—C2	101.90 (12)
C6—C5—H5B	109.2	C33—C23—C2	119.06 (13)
H5A—C5—H5B	107.9	C24—C23—C2	110.04 (12)
C7—C6—C1	116.54 (16)	O2—C24—C25	126.70 (16)
C7—C6—C5	123.80 (16)	O2—C24—C23	124.93 (16)
C1—C6—C5	119.60 (14)	C25—C24—C23	108.28 (14)
C6—C7—C8	127.74 (18)	C26—C25—C34	120.53 (18)
C6—C7—H7	116.1	C26—C25—C24	132.12 (19)
C8—C7—H7	116.1	C34—C25—C24	107.35 (15)
C9—C8—C13	116.62 (18)	C25—C26—C27	118.0 (2)
C9—C8—C7	122.54 (18)	C25—C26—H26	121.0
C13—C8—C7	120.78 (18)	C27—C26—H26	121.0
C10—C9—C8	121.5 (2)	C28—C27—C26	121.8 (2)
C10—C9—H9	119.2	C28—C27—H27	119.1
C8—C9—H9	119.2	C26—C27—H27	119.1
C11—C10—C9	120.5 (2)	C27—C28—C29	121.7 (2)
C11—C10—H10	119.8	C27—C28—H28	119.1
C9—C10—H10	119.8	C29—C28—H28	119.1
C10—C11—C12	119.8 (2)	C30—C29—C34	116.10 (17)
C10—C11—H11	120.1	C30—C29—C28	128.53 (19)
C12—C11—H11	120.1	C34—C29—C28	115.4 (2)
C11—C12—C13	119.8 (2)	C31—C30—C29	120.33 (17)
C11—C12—H12	120.1	C31—C30—H30	119.8
C13—C12—H12	120.1	C29—C30—H30	119.8
C12—C13—C8	121.8 (2)	C30—C31—C32	122.50 (18)
C12—C13—C11	118.43 (17)	C30—C31—H31	118.8
C8—C13—C11	119.76 (15)	C32—C31—H31	118.8
C15—C14—C19	122.37 (17)	C33—C32—C31	118.77 (17)
C15—C14—C12	117.45 (15)	C33—C32—H32	120.6
C19—C14—C12	120.17 (13)	C31—C32—H32	120.6
C16—C15—C14	119.5 (2)	C32—C33—C34	118.36 (15)
C16—C15—H15	120.3	C32—C33—C23	132.64 (15)
C14—C15—H15	120.3	C34—C33—C23	108.90 (14)
C17—C16—C15	119.85 (19)	C25—C34—C29	122.39 (17)
C17—C16—H16	120.1	C25—C34—C33	113.76 (15)
C15—C16—H16	120.1	C29—C34—C33	123.85 (17)
O1—C1—C2—C3	-151.50 (15)	C19—C20—C21—N2	-152.40 (13)
C6—C1—C2—C3	30.10 (18)	C2—C20—C21—N2	-25.30 (16)
O1—C1—C2—C20	-25.8 (2)	C22—N2—C23—C33	60.97 (19)
C6—C1—C2—C20	155.77 (13)	C21—N2—C23—C33	-169.74 (13)
O1—C1—C2—C23	87.09 (18)	C22—N2—C23—C24	-52.7 (2)
C6—C1—C2—C23	-91.31 (15)	C21—N2—C23—C24	76.57 (16)
C5—N1—C3—C2	75.82 (16)	C22—N2—C23—C2	-171.16 (15)
C4—N1—C3—C2	-160.58 (14)	C21—N2—C23—C2	-41.87 (15)
C1—C2—C3—N1	-60.62 (16)	C3—C2—C23—N2	147.45 (12)
C20—C2—C3—N1	175.67 (13)	C1—C2—C23—N2	-93.90 (14)
C23—C2—C3—N1	57.94 (16)	C20—C2—C23—N2	23.71 (14)

C3—N1—C5—C6	-54.27 (18)	C3—C2—C23—C33	-89.93 (16)
C4—N1—C5—C6	-178.59 (14)	C1—C2—C23—C33	28.72 (17)
O1—C1—C6—C7	-14.3 (2)	C20—C2—C23—C33	146.34 (13)
C2—C1—C6—C7	164.07 (15)	C3—C2—C23—C24	26.31 (17)
O1—C1—C6—C5	168.41 (16)	C1—C2—C23—C24	144.96 (13)
C2—C1—C6—C5	-13.2 (2)	C20—C2—C23—C24	-97.43 (14)
N1—C5—C6—C7	-153.17 (16)	N2—C23—C24—O2	-51.3 (2)
N1—C5—C6—C1	23.9 (2)	C33—C23—C24—O2	-170.73 (17)
C1—C6—C7—C8	-179.00 (17)	C2—C23—C24—O2	62.4 (2)
C5—C6—C7—C8	-1.9 (3)	N2—C23—C24—C25	125.47 (15)
C6—C7—C8—C9	-42.2 (3)	C33—C23—C24—C25	6.04 (16)
C6—C7—C8—C13	140.7 (2)	C2—C23—C24—C25	-120.86 (14)
C13—C8—C9—C10	-0.5 (4)	O2—C24—C25—C26	-8.4 (3)
C7—C8—C9—C10	-177.7 (3)	C23—C24—C25—C26	174.85 (19)
C8—C9—C10—C11	-0.1 (5)	O2—C24—C25—C34	171.10 (18)
C9—C10—C11—C12	0.4 (5)	C23—C24—C25—C34	-5.60 (18)
C10—C11—C12—C13	-0.2 (5)	C34—C25—C26—C27	-0.4 (3)
C11—C12—C13—C8	-0.4 (4)	C24—C25—C26—C27	179.1 (2)
C11—C12—C13—C11	-179.7 (2)	C25—C26—C27—C28	-2.0 (3)
C9—C8—C13—C12	0.8 (3)	C26—C27—C28—C29	1.6 (3)
C7—C8—C13—C12	178.0 (2)	C27—C28—C29—C30	-178.3 (2)
C9—C8—C13—C11	-179.95 (18)	C27—C28—C29—C34	1.2 (3)
C7—C8—C13—C11	-2.7 (3)	C34—C29—C30—C31	0.5 (3)
C19—C14—C15—C16	0.9 (3)	C28—C29—C30—C31	179.99 (19)
C12—C14—C15—C16	-177.66 (15)	C29—C30—C31—C32	1.4 (3)
C14—C15—C16—C17	-0.4 (3)	C30—C31—C32—C33	-1.0 (3)
C15—C16—C17—C18	-0.2 (3)	C31—C32—C33—C34	-1.4 (2)
C16—C17—C18—C19	0.3 (3)	C31—C32—C33—C23	-177.36 (16)
C15—C14—C19—C18	-0.7 (2)	N2—C23—C33—C32	50.4 (2)
C12—C14—C19—C18	177.79 (13)	C24—C23—C33—C32	171.78 (17)
C15—C14—C19—C20	-179.46 (15)	C2—C23—C33—C32	-67.5 (2)
C12—C14—C19—C20	-0.9 (2)	N2—C23—C33—C34	-125.80 (14)
C17—C18—C19—C14	0.1 (3)	C24—C23—C33—C34	-4.45 (16)
C17—C18—C19—C20	178.84 (17)	C2—C23—C33—C34	116.30 (15)
C14—C19—C20—C21	-145.35 (15)	C26—C25—C34—C29	3.3 (3)
C18—C19—C20—C21	36.0 (2)	C24—C25—C34—C29	-176.29 (15)
C14—C19—C20—C2	92.53 (18)	C26—C25—C34—C33	-177.61 (16)
C18—C19—C20—C2	-86.13 (19)	C24—C25—C34—C33	2.8 (2)
C3—C2—C20—C19	5.57 (19)	C30—C29—C34—C25	175.93 (16)
C1—C2—C20—C19	-116.52 (15)	C28—C29—C34—C25	-3.6 (2)
C23—C2—C20—C19	128.06 (14)	C30—C29—C34—C33	-3.0 (2)
C3—C2—C20—C21	-121.76 (14)	C28—C29—C34—C33	177.43 (16)
C1—C2—C20—C21	116.15 (14)	C32—C33—C34—C25	-175.52 (15)
C23—C2—C20—C21	0.73 (15)	C23—C33—C34—C25	1.33 (19)
C22—N2—C21—C20	173.28 (15)	C32—C33—C34—C29	3.5 (2)
C23—N2—C21—C20	43.13 (17)	C23—C33—C34—C29	-179.62 (15)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C25–C29/C34 ring.

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C10—H10···O2 ⁱ	0.93	2.74	3.492 (3)	139
C16—H16···O2 ⁱⁱ	0.93	2.76	3.481 (3)	135
C5—H5B···Cg1 ⁱ	0.97	2.99	3.9466 (19)	168

Symmetry codes: (i) $-x, y+1/2, -z+1/2$; (ii) $-x, -y+2, -z$.**1'-Methyl-5''-(2-methylbenzylidene)-4'-(2-methylphenyl)dispiro[acenaphthene-1,2'-pyrrolidine-3',3''-piperidine]-2,4''-dione (II)**

Crystal data

C₃₆H₃₄N₂O₂ $M_r = 526.65$ Monoclinic, $P2_1/c$ $a = 8.7507$ (5) Å $b = 15.9089$ (8) Å $c = 20.2879$ (10) Å $\beta = 92.935$ (2)° $V = 2820.7$ (3) Å³ $Z = 4$ $F(000) = 1120$ $D_x = 1.240$ Mg m⁻³ $D_m = 1.24$ Mg m⁻³ D_m measured by floatation methodMo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5358 reflections

 $\theta = 4.7$ – 42.0° $\mu = 0.08$ mm⁻¹ $T = 293$ K

Block, yellow

 $0.32 \times 0.24 \times 0.18$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

 φ and ω scansAbsorption correction: multi-scan
(*SADABS*; Bruker, 2001) $T_{\min} = 0.816$, $T_{\max} = 1.000$

28946 measured reflections

5339 independent reflections

3349 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$ $\theta_{\max} = 25.7^\circ$, $\theta_{\min} = 2.3^\circ$ $h = -10 \rightarrow 10$ $k = -19 \rightarrow 19$ $l = -24 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.045$ $wR(F^2) = 0.121$ $S = 1.02$

5339 reflections

362 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.5169P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.17$ e Å⁻³ $\Delta\rho_{\min} = -0.15$ e Å⁻³

Extinction correction: SHELXL2018

(Sheldrick, 2015),

 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0040 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.47706 (15)	0.91884 (10)	0.26949 (6)	0.0632 (4)	
O2	0.01075 (17)	0.80552 (10)	0.11060 (7)	0.0749 (5)	
N1	0.02197 (15)	0.96279 (9)	0.23966 (7)	0.0438 (4)	
N2	0.32038 (17)	0.76401 (9)	0.17891 (7)	0.0488 (4)	
C1	0.3410 (2)	0.93366 (11)	0.26053 (8)	0.0434 (4)	
C2	0.25341 (18)	0.90633 (11)	0.19667 (8)	0.0377 (4)	
C3	0.12046 (19)	0.96732 (11)	0.18425 (8)	0.0420 (4)	
H3A	0.159005	1.024095	0.179771	0.050*	
H3B	0.063116	0.952451	0.143748	0.050*	
C4	-0.1307 (2)	0.99492 (14)	0.22319 (10)	0.0619 (6)	
H4A	-0.175538	0.964127	0.186372	0.093*	
H4B	-0.192980	0.988480	0.260484	0.093*	
H4C	-0.124579	1.053358	0.211850	0.093*	
C5	0.0937 (2)	1.00778 (12)	0.29519 (9)	0.0529 (5)	
H5A	0.032387	1.000780	0.333317	0.063*	
H5B	0.096585	1.067257	0.284807	0.063*	
C6	0.2541 (2)	0.97734 (11)	0.31209 (8)	0.0459 (4)	
C7	0.3224 (2)	0.98436 (13)	0.37200 (9)	0.0566 (5)	
H7	0.420638	0.962182	0.376842	0.068*	
C8	0.2630 (3)	1.02252 (14)	0.43153 (9)	0.0637 (6)	
C9	0.1907 (3)	1.09994 (16)	0.42926 (11)	0.0862 (8)	
H9	0.176292	1.127477	0.388985	0.103*	
C10	0.1399 (4)	1.13691 (19)	0.48527 (13)	0.1125 (11)	
H10	0.092416	1.189186	0.482807	0.135*	
C11	0.1595 (4)	1.0966 (2)	0.54474 (13)	0.1158 (11)	
H11	0.124439	1.121064	0.582782	0.139*	
C12	0.2304 (4)	1.02073 (18)	0.54774 (12)	0.1011 (10)	
H12	0.242808	0.993784	0.588346	0.121*	
C13	0.2852 (3)	0.98180 (14)	0.49223 (10)	0.0736 (7)	
C14	0.4072 (2)	1.03916 (12)	0.08230 (9)	0.0477 (5)	
C15	0.3741 (2)	1.09458 (14)	0.03059 (10)	0.0627 (6)	
H15	0.425474	1.145727	0.029917	0.075*	
C16	0.2684 (3)	1.07634 (17)	-0.01936 (11)	0.0716 (7)	
H16	0.247654	1.114850	-0.053129	0.086*	
C17	0.1933 (2)	1.00083 (17)	-0.01915 (10)	0.0693 (6)	
H17	0.121043	0.987782	-0.052809	0.083*	
C18	0.2252 (2)	0.94425 (14)	0.03112 (9)	0.0563 (5)	
H18	0.173963	0.893017	0.030703	0.068*	
C19	0.33151 (19)	0.96161 (12)	0.08224 (8)	0.0429 (4)	
C20	0.36354 (19)	0.89945 (11)	0.13800 (8)	0.0420 (4)	
H20	0.466999	0.911443	0.156377	0.050*	
C21	0.3607 (2)	0.80656 (12)	0.11868 (9)	0.0529 (5)	
H21A	0.284808	0.796067	0.083082	0.063*	
H21B	0.460023	0.788322	0.105068	0.063*	
C22	0.2938 (3)	0.67430 (13)	0.17162 (11)	0.0731 (7)	

H22A	0.268011	0.651006	0.213260	0.110*	
H22B	0.211144	0.664885	0.139531	0.110*	
H22C	0.384812	0.647695	0.157250	0.110*	
C23	0.19669 (19)	0.81255 (11)	0.20669 (8)	0.0422 (4)	
C24	0.0359 (2)	0.79910 (12)	0.16969 (10)	0.0523 (5)	
C25	-0.0735 (2)	0.77254 (12)	0.21815 (10)	0.0540 (5)	
C26	-0.2285 (2)	0.75824 (14)	0.21310 (13)	0.0723 (6)	
H26	-0.284279	0.766562	0.173375	0.087*	
C27	-0.2998 (3)	0.73101 (16)	0.26895 (16)	0.0843 (8)	
H27	-0.405192	0.722827	0.266586	0.101*	
C28	-0.2194 (3)	0.71598 (14)	0.32708 (14)	0.0765 (7)	
H28	-0.270670	0.696290	0.363029	0.092*	
C29	-0.0593 (2)	0.72969 (12)	0.33393 (11)	0.0580 (5)	
C30	0.0395 (3)	0.71575 (13)	0.38964 (11)	0.0661 (6)	
H30	0.000748	0.694837	0.428213	0.079*	
C31	0.1921 (3)	0.73275 (12)	0.38742 (10)	0.0618 (6)	
H31	0.255708	0.721647	0.424532	0.074*	
C32	0.2575 (2)	0.76681 (11)	0.33047 (9)	0.0504 (5)	
H32	0.361543	0.778819	0.330644	0.060*	
C33	0.1650 (2)	0.78149 (11)	0.27577 (8)	0.0430 (4)	
C34	0.0085 (2)	0.76075 (11)	0.27794 (9)	0.0473 (5)	
C35	0.3604 (4)	0.89722 (16)	0.49768 (11)	0.0951 (9)	
H35A	0.391046	0.879874	0.454998	0.143*	0.5
H35B	0.448790	0.900568	0.527609	0.143*	0.5
H35C	0.289565	0.857078	0.513916	0.143*	0.5
H35D	0.361888	0.878473	0.542684	0.143*	0.5
H35E	0.304143	0.857779	0.470073	0.143*	0.5
H35F	0.463369	0.901268	0.483766	0.143*	0.5
C36	0.5214 (2)	1.06401 (14)	0.13658 (11)	0.0659 (6)	
H36A	0.531595	1.019372	0.168344	0.099*	0.5
H36B	0.618648	1.074534	0.118366	0.099*	0.5
H36C	0.486814	1.114002	0.157740	0.099*	0.5
H36D	0.559776	1.119233	0.127956	0.099*	0.5
H36E	0.472723	1.064071	0.177934	0.099*	0.5
H36F	0.604557	1.024604	0.138560	0.099*	0.5

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0476 (8)	0.0952 (11)	0.0459 (8)	0.0049 (7)	-0.0050 (6)	-0.0030 (7)
O2	0.0784 (10)	0.0890 (12)	0.0551 (10)	-0.0227 (8)	-0.0182 (8)	0.0099 (8)
N1	0.0438 (8)	0.0493 (9)	0.0386 (9)	0.0075 (7)	0.0052 (7)	0.0005 (7)
N2	0.0621 (10)	0.0420 (9)	0.0434 (9)	0.0056 (7)	0.0121 (7)	-0.0019 (7)
C1	0.0449 (11)	0.0489 (11)	0.0361 (10)	-0.0008 (8)	0.0004 (8)	0.0042 (8)
C2	0.0396 (9)	0.0432 (10)	0.0303 (9)	0.0010 (8)	0.0026 (7)	0.0004 (8)
C3	0.0471 (10)	0.0452 (10)	0.0336 (10)	0.0022 (8)	0.0018 (8)	0.0018 (8)
C4	0.0526 (12)	0.0685 (14)	0.0650 (14)	0.0164 (10)	0.0071 (10)	-0.0003 (11)
C5	0.0666 (13)	0.0533 (12)	0.0391 (11)	0.0101 (10)	0.0072 (9)	-0.0033 (9)

C6	0.0597 (11)	0.0439 (11)	0.0340 (10)	-0.0015 (9)	0.0009 (8)	0.0001 (8)
C7	0.0757 (14)	0.0559 (12)	0.0377 (11)	-0.0012 (10)	-0.0014 (10)	-0.0005 (9)
C8	0.0948 (16)	0.0596 (14)	0.0362 (12)	0.0032 (12)	-0.0021 (10)	-0.0079 (10)
C9	0.143 (2)	0.0701 (16)	0.0441 (14)	0.0211 (16)	-0.0056 (14)	-0.0085 (12)
C10	0.193 (3)	0.0801 (19)	0.0642 (18)	0.040 (2)	0.0039 (19)	-0.0204 (15)
C11	0.202 (3)	0.093 (2)	0.0542 (17)	0.028 (2)	0.0216 (18)	-0.0222 (16)
C12	0.179 (3)	0.0834 (19)	0.0420 (14)	0.013 (2)	0.0156 (16)	-0.0069 (13)
C13	0.118 (2)	0.0631 (15)	0.0393 (12)	0.0007 (14)	0.0031 (12)	-0.0062 (11)
C14	0.0447 (10)	0.0549 (12)	0.0444 (11)	0.0043 (9)	0.0126 (8)	0.0033 (9)
C15	0.0696 (14)	0.0620 (14)	0.0584 (14)	0.0034 (11)	0.0222 (11)	0.0141 (11)
C16	0.0759 (16)	0.0903 (18)	0.0497 (14)	0.0212 (14)	0.0140 (12)	0.0276 (12)
C17	0.0593 (13)	0.107 (2)	0.0415 (12)	0.0025 (13)	0.0008 (10)	0.0154 (12)
C18	0.0545 (12)	0.0767 (14)	0.0377 (11)	-0.0056 (10)	0.0028 (9)	0.0068 (10)
C19	0.0414 (10)	0.0557 (12)	0.0322 (10)	0.0019 (9)	0.0084 (8)	0.0030 (8)
C20	0.0402 (10)	0.0518 (11)	0.0342 (10)	-0.0004 (8)	0.0025 (7)	-0.0005 (8)
C21	0.0609 (12)	0.0562 (12)	0.0425 (11)	0.0046 (9)	0.0117 (9)	-0.0047 (9)
C22	0.1067 (18)	0.0462 (12)	0.0682 (15)	0.0039 (12)	0.0207 (13)	-0.0071 (11)
C23	0.0437 (10)	0.0457 (11)	0.0372 (10)	0.0020 (8)	0.0029 (8)	0.0007 (8)
C24	0.0573 (12)	0.0463 (11)	0.0524 (13)	-0.0040 (9)	-0.0057 (10)	0.0040 (9)
C25	0.0477 (11)	0.0459 (11)	0.0684 (14)	-0.0011 (9)	0.0045 (10)	0.0018 (10)
C26	0.0524 (13)	0.0648 (14)	0.0994 (19)	-0.0021 (11)	0.0012 (12)	-0.0008 (13)
C27	0.0542 (14)	0.0776 (17)	0.123 (2)	-0.0061 (13)	0.0221 (16)	-0.0055 (17)
C28	0.0690 (16)	0.0648 (15)	0.100 (2)	-0.0065 (12)	0.0418 (15)	-0.0021 (14)
C29	0.0696 (14)	0.0404 (11)	0.0664 (14)	0.0048 (10)	0.0249 (12)	0.0005 (10)
C30	0.0901 (17)	0.0501 (13)	0.0613 (14)	0.0083 (12)	0.0364 (13)	0.0071 (10)
C31	0.0919 (17)	0.0501 (12)	0.0439 (12)	0.0184 (12)	0.0088 (11)	0.0087 (9)
C32	0.0591 (12)	0.0465 (11)	0.0460 (11)	0.0112 (9)	0.0069 (9)	0.0061 (9)
C33	0.0509 (11)	0.0366 (10)	0.0422 (11)	0.0069 (8)	0.0082 (8)	0.0033 (8)
C34	0.0519 (11)	0.0349 (10)	0.0563 (12)	0.0028 (8)	0.0149 (10)	0.0026 (9)
C35	0.157 (3)	0.0752 (17)	0.0521 (14)	0.0178 (17)	-0.0015 (15)	0.0049 (12)
C36	0.0637 (13)	0.0622 (14)	0.0717 (15)	-0.0101 (11)	0.0025 (11)	-0.0023 (11)

Geometric parameters (Å, °)

O1—C1	1.218 (2)	C17—H17	0.9300
O2—C24	1.212 (2)	C18—C19	1.385 (2)
N1—C5	1.450 (2)	C18—H18	0.9300
N1—C3	1.453 (2)	C19—C20	1.518 (2)
N1—C4	1.454 (2)	C20—C21	1.529 (3)
N2—C22	1.452 (2)	C20—H20	0.9800
N2—C21	1.456 (2)	C21—H21A	0.9700
N2—C23	1.466 (2)	C21—H21B	0.9700
C1—C6	1.496 (2)	C22—H22A	0.9600
C1—C2	1.534 (2)	C22—H22B	0.9600
C2—C3	1.526 (2)	C22—H22C	0.9600
C2—C20	1.573 (2)	C23—C33	1.525 (2)
C2—C23	1.589 (2)	C23—C24	1.575 (3)
C3—H3A	0.9700	C24—C25	1.469 (3)

C3—H3B	0.9700	C25—C26	1.374 (3)
C4—H4A	0.9600	C25—C34	1.390 (3)
C4—H4B	0.9600	C26—C27	1.390 (3)
C4—H4C	0.9600	C26—H26	0.9300
C5—C6	1.508 (3)	C27—C28	1.363 (3)
C5—H5A	0.9700	C27—H27	0.9300
C5—H5B	0.9700	C28—C29	1.417 (3)
C6—C7	1.331 (2)	C28—H28	0.9300
C7—C8	1.470 (3)	C29—C34	1.398 (3)
C7—H7	0.9300	C29—C30	1.405 (3)
C8—C9	1.384 (3)	C30—C31	1.366 (3)
C8—C13	1.396 (3)	C30—H30	0.9300
C9—C10	1.374 (3)	C31—C32	1.423 (3)
C9—H9	0.9300	C31—H31	0.9300
C10—C11	1.369 (4)	C32—C33	1.360 (2)
C10—H10	0.9300	C32—H32	0.9300
C11—C12	1.358 (4)	C33—C34	1.412 (2)
C11—H11	0.9300	C35—H35A	0.9600
C12—C13	1.392 (3)	C35—H35B	0.9600
C12—H12	0.9300	C35—H35C	0.9600
C13—C35	1.500 (3)	C35—H35D	0.9600
C14—C15	1.390 (3)	C35—H35E	0.9600
C14—C19	1.400 (3)	C35—H35F	0.9600
C14—C36	1.501 (3)	C36—H36A	0.9600
C15—C16	1.368 (3)	C36—H36B	0.9600
C15—H15	0.9300	C36—H36C	0.9600
C16—C17	1.369 (3)	C36—H36D	0.9600
C16—H16	0.9300	C36—H36E	0.9600
C17—C18	1.378 (3)	C36—H36F	0.9600
C5—N1—C3	109.05 (14)	C19—C20—C21	116.01 (14)
C5—N1—C4	111.16 (15)	C19—C20—C2	114.95 (14)
C3—N1—C4	112.30 (14)	C21—C20—C2	104.99 (14)
C22—N2—C21	114.57 (15)	C19—C20—H20	106.8
C22—N2—C23	116.08 (15)	C21—C20—H20	106.8
C21—N2—C23	107.05 (13)	C2—C20—H20	106.8
O1—C1—C6	120.91 (16)	N2—C21—C20	103.63 (13)
O1—C1—C2	120.90 (16)	N2—C21—H21A	111.0
C6—C1—C2	118.18 (15)	C20—C21—H21A	111.0
C3—C2—C1	107.64 (14)	N2—C21—H21B	111.0
C3—C2—C20	114.25 (13)	C20—C21—H21B	111.0
C1—C2—C20	111.07 (13)	H21A—C21—H21B	109.0
C3—C2—C23	112.19 (13)	N2—C22—H22A	109.5
C1—C2—C23	107.70 (13)	N2—C22—H22B	109.5
C20—C2—C23	103.83 (13)	H22A—C22—H22B	109.5
N1—C3—C2	108.55 (13)	N2—C22—H22C	109.5
N1—C3—H3A	110.0	H22A—C22—H22C	109.5
C2—C3—H3A	110.0	H22B—C22—H22C	109.5

N1—C3—H3B	110.0	N2—C23—C33	110.81 (14)
C2—C3—H3B	110.0	N2—C23—C24	113.84 (14)
H3A—C3—H3B	108.4	C33—C23—C24	101.21 (14)
N1—C4—H4A	109.5	N2—C23—C2	101.76 (13)
N1—C4—H4B	109.5	C33—C23—C2	119.65 (14)
H4A—C4—H4B	109.5	C24—C23—C2	110.12 (13)
N1—C4—H4C	109.5	O2—C24—C25	126.46 (18)
H4A—C4—H4C	109.5	O2—C24—C23	124.94 (17)
H4B—C4—H4C	109.5	C25—C24—C23	108.48 (16)
N1—C5—C6	112.26 (15)	C26—C25—C34	120.42 (19)
N1—C5—H5A	109.2	C26—C25—C24	132.3 (2)
C6—C5—H5A	109.2	C34—C25—C24	107.31 (16)
N1—C5—H5B	109.2	C25—C26—C27	118.3 (2)
C6—C5—H5B	109.2	C25—C26—H26	120.9
H5A—C5—H5B	107.9	C27—C26—H26	120.9
C7—C6—C1	117.20 (17)	C28—C27—C26	121.8 (2)
C7—C6—C5	123.34 (17)	C28—C27—H27	119.1
C1—C6—C5	119.41 (15)	C26—C27—H27	119.1
C6—C7—C8	128.6 (2)	C27—C28—C29	121.4 (2)
C6—C7—H7	115.7	C27—C28—H28	119.3
C8—C7—H7	115.7	C29—C28—H28	119.3
C9—C8—C13	119.01 (19)	C34—C29—C30	116.11 (19)
C9—C8—C7	121.34 (19)	C34—C29—C28	115.7 (2)
C13—C8—C7	119.6 (2)	C30—C29—C28	128.2 (2)
C10—C9—C8	121.3 (2)	C31—C30—C29	120.30 (19)
C10—C9—H9	119.3	C31—C30—H30	119.8
C8—C9—H9	119.3	C29—C30—H30	119.8
C11—C10—C9	119.8 (3)	C30—C31—C32	122.5 (2)
C11—C10—H10	120.1	C30—C31—H31	118.8
C9—C10—H10	120.1	C32—C31—H31	118.8
C12—C11—C10	119.5 (2)	C33—C32—C31	118.75 (19)
C12—C11—H11	120.3	C33—C32—H32	120.6
C10—C11—H11	120.3	C31—C32—H32	120.6
C11—C12—C13	122.4 (2)	C32—C33—C34	118.17 (17)
C11—C12—H12	118.8	C32—C33—C23	132.64 (16)
C13—C12—H12	118.8	C34—C33—C23	109.07 (15)
C12—C13—C8	118.0 (2)	C25—C34—C29	122.33 (19)
C12—C13—C35	120.5 (2)	C25—C34—C33	113.55 (16)
C8—C13—C35	121.46 (19)	C29—C34—C33	124.10 (19)
C15—C14—C19	118.55 (18)	C13—C35—H35A	109.5
C15—C14—C36	119.47 (18)	C13—C35—H35B	109.5
C19—C14—C36	121.98 (17)	H35A—C35—H35B	109.5
C16—C15—C14	122.1 (2)	C13—C35—H35C	109.5
C16—C15—H15	118.9	H35A—C35—H35C	109.5
C14—C15—H15	118.9	H35B—C35—H35C	109.5
C15—C16—C17	119.4 (2)	H35D—C35—H35E	109.5
C15—C16—H16	120.3	H35D—C35—H35F	109.5
C17—C16—H16	120.3	H35E—C35—H35F	109.5

C16—C17—C18	119.8 (2)	C14—C36—H36A	109.5
C16—C17—H17	120.1	C14—C36—H36B	109.5
C18—C17—H17	120.1	H36A—C36—H36B	109.5
C17—C18—C19	121.8 (2)	C14—C36—H36C	109.5
C17—C18—H18	119.1	H36A—C36—H36C	109.5
C19—C18—H18	119.1	H36B—C36—H36C	109.5
C18—C19—C14	118.42 (17)	H36D—C36—H36E	109.5
C18—C19—C20	121.30 (17)	H36D—C36—H36F	109.5
C14—C19—C20	120.26 (15)	H36E—C36—H36F	109.5
O1—C1—C2—C3	-152.18 (17)	C19—C20—C21—N2	-153.51 (14)
C6—C1—C2—C3	29.1 (2)	C2—C20—C21—N2	-25.45 (17)
O1—C1—C2—C20	-26.4 (2)	C22—N2—C23—C33	60.2 (2)
C6—C1—C2—C20	154.89 (15)	C21—N2—C23—C33	-170.39 (14)
O1—C1—C2—C23	86.66 (19)	C22—N2—C23—C24	-53.0 (2)
C6—C1—C2—C23	-92.04 (17)	C21—N2—C23—C24	76.33 (18)
C5—N1—C3—C2	76.18 (17)	C22—N2—C23—C2	-171.47 (15)
C4—N1—C3—C2	-160.14 (15)	C21—N2—C23—C2	-42.10 (16)
C1—C2—C3—N1	-60.49 (17)	C3—C2—C23—N2	147.72 (13)
C20—C2—C3—N1	175.66 (13)	C1—C2—C23—N2	-94.01 (15)
C23—C2—C3—N1	57.82 (17)	C20—C2—C23—N2	23.85 (15)
C3—N1—C5—C6	-54.01 (19)	C3—C2—C23—C33	-89.87 (17)
C4—N1—C5—C6	-178.36 (15)	C1—C2—C23—C33	28.4 (2)
O1—C1—C6—C7	-12.8 (3)	C20—C2—C23—C33	146.26 (14)
C2—C1—C6—C7	165.91 (16)	C3—C2—C23—C24	26.65 (18)
O1—C1—C6—C5	169.68 (17)	C1—C2—C23—C24	144.93 (14)
C2—C1—C6—C5	-11.6 (2)	C20—C2—C23—C24	-97.21 (15)
N1—C5—C6—C7	-154.62 (18)	N2—C23—C24—O2	-51.5 (2)
N1—C5—C6—C1	22.8 (2)	C33—C23—C24—O2	-170.37 (18)
C1—C6—C7—C8	-178.84 (19)	C2—C23—C24—O2	62.1 (2)
C5—C6—C7—C8	-1.4 (3)	N2—C23—C24—C25	124.97 (16)
C6—C7—C8—C9	-46.1 (3)	C33—C23—C24—C25	6.06 (18)
C6—C7—C8—C13	136.6 (2)	C2—C23—C24—C25	-121.50 (15)
C13—C8—C9—C10	-0.4 (4)	O2—C24—C25—C26	-8.7 (4)
C7—C8—C9—C10	-177.7 (3)	C23—C24—C25—C26	174.9 (2)
C8—C9—C10—C11	-0.5 (5)	O2—C24—C25—C34	170.5 (2)
C9—C10—C11—C12	0.6 (6)	C23—C24—C25—C34	-5.9 (2)
C10—C11—C12—C13	0.2 (5)	C34—C25—C26—C27	-0.7 (3)
C11—C12—C13—C8	-1.1 (5)	C24—C25—C26—C27	178.4 (2)
C11—C12—C13—C35	-179.0 (3)	C25—C26—C27—C28	-2.0 (4)
C9—C8—C13—C12	1.2 (4)	C26—C27—C28—C29	1.8 (4)
C7—C8—C13—C12	178.6 (2)	C27—C28—C29—C34	1.1 (3)
C9—C8—C13—C35	179.0 (3)	C27—C28—C29—C30	-178.6 (2)
C7—C8—C13—C35	-3.6 (4)	C34—C29—C30—C31	0.3 (3)
C19—C14—C15—C16	1.2 (3)	C28—C29—C30—C31	-179.9 (2)
C36—C14—C15—C16	-178.30 (19)	C29—C30—C31—C32	1.6 (3)
C14—C15—C16—C17	-0.7 (3)	C30—C31—C32—C33	-1.3 (3)
C15—C16—C17—C18	-0.1 (3)	C31—C32—C33—C34	-1.0 (3)

C16—C17—C18—C19	0.3 (3)	C31—C32—C33—C23	-176.46 (17)
C17—C18—C19—C14	0.3 (3)	N2—C23—C33—C32	50.5 (3)
C17—C18—C19—C20	178.85 (17)	C24—C23—C33—C32	171.57 (19)
C15—C14—C19—C18	-1.0 (3)	C2—C23—C33—C32	-67.4 (2)
C36—C14—C19—C18	178.52 (17)	N2—C23—C33—C34	-125.23 (15)
C15—C14—C19—C20	-179.60 (16)	C24—C23—C33—C34	-4.16 (17)
C36—C14—C19—C20	-0.1 (3)	C2—C23—C33—C34	116.91 (16)
C18—C19—C20—C21	36.9 (2)	C26—C25—C34—C29	3.7 (3)
C14—C19—C20—C21	-144.55 (17)	C24—C25—C34—C29	-175.53 (17)
C18—C19—C20—C2	-86.1 (2)	C26—C25—C34—C33	-177.43 (17)
C14—C19—C20—C2	92.47 (19)	C24—C25—C34—C33	3.3 (2)
C3—C2—C20—C19	6.9 (2)	C30—C29—C34—C25	175.91 (18)
C1—C2—C20—C19	-115.06 (16)	C28—C29—C34—C25	-3.9 (3)
C23—C2—C20—C19	129.45 (15)	C30—C29—C34—C33	-2.8 (3)
C3—C2—C20—C21	-121.77 (16)	C28—C29—C34—C33	177.44 (17)
C1—C2—C20—C21	116.24 (16)	C32—C33—C34—C25	-175.63 (17)
C23—C2—C20—C21	0.75 (16)	C23—C33—C34—C25	0.8 (2)
C22—N2—C21—C20	173.69 (16)	C32—C33—C34—C29	3.2 (3)
C23—N2—C21—C20	43.47 (18)	C23—C33—C34—C29	179.61 (17)

Hydrogen-bond geometry (Å, °)

Cg2 is the centroid of the C8—C13 ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C36—H36 <i>A</i> ...O1	0.96	2.66	3.586 (3)	161
C10—H10...O2 ⁱ	0.93	2.77	3.529 (3)	140
C16—H16...O2 ⁱⁱ	0.93	2.79	3.530 (3)	137
C35—H35 <i>F</i> ...Cg2 ⁱⁱⁱ	0.96	2.94	3.805 (4)	151

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