Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7,7-Dimethyl-5-phenyl-10-(2-thienyl)-7,8-dihydro-5*H*-indeno[1,2-*b*]quinoline-9,11(6*H*,10*H*)-dione dimethylformamide hemisolvate

Weijun Zhu,[a]* Chunmei Li[b] and Yan Xiang[c]

[a]Xuzhou College of Industrial Technology, Xuzhou 221140, People's Republic of China, [b]School of Chemistry and Chemical Engineering, Xuzhou Normal University, Xuzhou 221116, People's Republic of China, and [c]Xuzhou Medicine High Vocational School, Xuzhou 221116, People's Republic of China
Correspondence e-mail: laotu2001@263.net

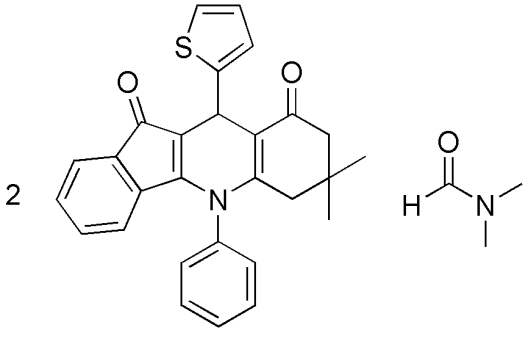
Received 14 August 2007; accepted 23 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.067; wR factor = 0.216; data-to-parameter ratio = 13.8.

The asymmetric unit of the title compound, $C_{28}H_{23}NO_2S\cdot0.5C_3H_7NO$, contains two 7,7-dimethyl-5-phenyl-10-(2-thienyl)-7,8-dihydro-5*H*-indeno[1,2-*b*]quinoline-9,11(6*H*,10*H*)-dione molecules and one dimethylformamide solvent molecule. The two thiophene rings are disordered over two positions each; site-occupancy factors range from 0.457 (6) to 0.543 (6). The two dihydropyridine rings adopt boat conformations, while the two cyclohexene rings have envelope conformations.

Related literature

For related literature, see: Stout & Meyers (1982); Yamato *et al.* (1989); Deady *et al.* (1999, 2000); Chen *et al.* (2002); Tu *et al.* (2006); Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{28}H_{23}NO_2S\cdot0.5C_3H_7NO$
M_r = 474.08
Monoclinic, $P2_1/c$
a = 15.081 (2) Å
b = 9.4381 (14) Å
c = 35.102 (3) Å
β = 92.041 (3)°
V = 4992.9 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 298 (2) K
0.43 × 0.32 × 0.28 mm

Data collection

Bruker CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.934, T_{max} = 0.957
25441 measured reflections
8790 independent reflections
3935 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.216
S = 1.00
8790 reflections
636 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

The authors are grateful to the National Science Foundation of China (grant No. 20672090) and the Natural Science Foundation of Jiangsu Province (grant No. BK 2006033) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2311).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, J. J., Deady, L. W., Kaye, A. J., Finlay, G. J., Baguley, B. C. & Denny, W. A. (2002). *Bioorg. Med. Chem.* **10**, 2381–2386.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Deady, L. W., Desneves, J., Kaye, A. J., Finlay, G. J., Baguley, B. C. & Denny, W. A. (2000). *Bioorg. Med. Chem.* **8**, 977–984.
Deady, L. W., Desneves, J., Kaye, A. J., Thompson, M., Finlay, G. J., Baguley, B. C. & Denny, W. A. (1999). *Bioorg. Med. Chem.* **7**, 2801–2809.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stout, D. M. & Meyers, A. I. (1982). *Chem. Rev.* **82**, 223–243.
Tu, S. J., Jiang, B., Jia, R. H., Zhang, J. Y., Zhang, Y., Yao, C. S. & Shi, F. (2006). *Org. Biomol. Chem.* **4**, 3664–3668.
Yamato, M., Takeuchi, Y., Hashigaki, K., Ikeda, Y., Mingrong, C., Takeuchi, K., Matsushima, M., Tsuro, T., Tasiro, T., Tsukagoshi, S., Yamashita, Y. & Nakano, H. (1989). *J. Med. Chem.* **32**, 1295–1300.

  doi:10.1107/S1600536807041621

supplementary materials

Acta Cryst. (2007). E**63**, o3910 [**doi:10.1107/S1600536807041621**]

7,7-Dimethyl-5-phenyl-10-(2-thienyl)-7,8-dihydro-5*H*-indeno[1,2-*b*]quinoline-9,11(6*H*,10*H*)-dione dimethylformamide hemisolvate

W. Zhu, C. Li and Y. Xiang

Comment

1,4-Dihydropyridines (1,4-DHPs) are well known compounds because of their pharmacological profiles as calcium channel modulators (Stout & Meyers, 1982). With a 1,4-DHP parent nucleus, indenoquinoline belongs to a class of compounds which are special not only because of their interesting chemical and physical properties, but also due to their immense utility in the pharmaceutical industries. The discovery of indenoquinoline, as new potent cytotoxic and antitumor agents, has attracted the attention of organic chemists (Yamato *et al.*, 1989; Deady *et al.*, 2000; Chen *et al.*, 2002). It is well established that the chemical modifications on the indenoquinoline skeletons may bring remarkable changes of biological activity (Deady *et al.*, 1999). We report herein the crystal structure of the title compound, (I).

The asymmetric unit of the title compound, (I), contains two $C_{28}H_{23}NO_2S$ molecules and one dimethylformamide (C_3H_7NO) molecule (Fig. 1). The bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987).

When the crystal structure was solved,the atoms S1, S2, C26, H26, C54 and H54 were found to be disordered. So, for instance, the positions of C26 and C54 atoms may be occupied by S1 and S2 atoms, respectively. In fact, it may not be possible to separate S atoms from C atoms, and that is the reason why the C—S bonds are longer than C═C bonds, but shorter than normal C—S single bonds.

Rings A (C1—C6), B (N1/C1/C6—C8/C16), A' (C29—C34) and B' (N2/C29/C34—C36/C44) are not planar, having total puckering amplitudes, Q_T, of 0.467 (3), 0.278 (2), 0.456 (3) and 0.231 (3) Å, respectively. Rings B and B' adopt boat conformations [$\varphi = -59.19\ (3)°$, $\theta = 110.20\ (3)°$ and $\varphi = -118.31\ (2)°$, $\theta = 69.55\ (3)°$, respectively] (Cremer & Pople, 1975). Rings A and A' have envelope conformations with atoms C3 and C31 displaced by 0.639 (3) Å and −0.622 (2) Å from the planes of the other ring atoms, respectively. Rings C (C10—C15), D (C8—C10/C15/C16), E (C17—C22), F (C25—C28/S1) and C' (C38—C43), D' (C36—C38/C43/C44), E' (C45—C50) and F' (C53—C55/S2) are, of course, planar and rings C, D and C', D' are also coplanar with dihedral angles of 0.97 (3)° and 0.58 (3)°, respectively.

Experimental

The title compound, (I), was prepared by the reaction of thiophene-2 -carbaldehyde (110 mg, 1 mmol), 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone (220 mg, 1 mmol) with 1,3-indanedione (150 mg, 1 mmol) in acetic acid (1.5 ml) under microwave irradiation for 4 min at 200 W power and 393 K (microwave oven is EmrysTM Creator from Personal Chemistry, Uppsala, Sweden). Upon completion, monitored by TLC, the reaction mixture was cooled to room temperature and then poured into cold water. The solid product was filtered, washed with water and EtOH (95%), and subsequently dried and recrystallized from EtOH (95%) to give the pure product. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a 95% aqueous ethanol solution (yield; 370 mg, 85%; m.p. 503–505 K).

Refinement

When the crystal structure was solved,the atoms S1, S2, C26, H26, C54 and H54 were found to be disordered. During refinement with isotropic thermal parameters, the occupancies of disordered H atoms were refined as H26 = 0.543 (6), H26' = 0.457 (6), H54 = 0.476 (7) and H54' = 0.524 (7). The remaining site occupancy factors were also refined as S1 = 0.543 (6), S1' = 0.457 (6), S2 = 0.476 (7), S2' = 0.524 (7), C26 = 0.543 (6), C26' = 0.457 (6), C54 = 0.476 (7) and C54' = 0.524 (7), during anisotropic refinement. H atoms were positioned geometrically with C—H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

Figures

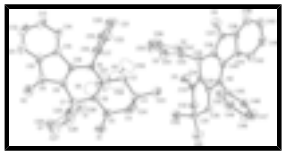


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

7,7-Dimethyl-5-phenyl-10-(2-thienyl)-7,8-dihydro-5*H*-indeno[1,2-*b*]quinoline-\ 9,11(6*H*,10H)-dione dimethylformamide hemisolvate

Crystal data

$C_{28}H_{23}NO_2S·0.5C_3H_7NO$	$F_{000} = 2000$
$M_r = 474.08$	$D_x = 1.261$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 503-505 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 15.081$ (2) Å	Cell parameters from 3416 reflections
$b = 9.4381$ (14) Å	$\theta = 2.2$–20.7º
$c = 35.102$ (3) Å	$\mu = 0.16$ mm^{-1}
$\beta = 92.041$ (3)º	$T = 298$ (2) K
$V = 4992.9$ (11) Å^3	Block, red
$Z = 8$	0.43 × 0.32 × 0.28 mm

Data collection

Bruker CCD area-detector diffractometer	8790 independent reflections
Radiation source: fine-focus sealed tube	3935 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.058$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -17 \rightarrow 17$
$T_{min} = 0.934$, $T_{max} = 0.957$	$k = -11 \rightarrow 11$

25441 measured reflections

$l = -41 \rightarrow 41$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.216$

$S = 1.00$

8790 reflections

636 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

$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 5.7834P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.47$ e Å^{-3}

$\Delta\rho_{min} = -0.38$ e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
N1	0.6520 (2)	0.8962 (4)	0.18639 (9)	0.0471 (9)	
O1	0.5219 (2)	1.1267 (4)	0.28990 (10)	0.0831 (11)	
O2	0.8552 (2)	0.8958 (4)	0.29221 (10)	0.0787 (10)	
S1	0.7025 (6)	1.2602 (12)	0.2105 (2)	0.0685 (15)	0.543 (6)
S1'	0.8025 (4)	1.2516 (13)	0.2777 (3)	0.0926 (19)	0.457 (6)
C1	0.5801 (3)	0.9650 (4)	0.20350 (11)	0.0446 (10)	
C2	0.4947 (3)	0.9692 (5)	0.18048 (12)	0.0530 (12)	
H2A	0.4677	0.8759	0.1808	0.064*	
H2B	0.5074	0.9912	0.1542	0.064*	
C3	0.4284 (3)	1.0774 (5)	0.19476 (14)	0.0585 (12)	
C4	0.4248 (3)	1.0620 (6)	0.23788 (13)	0.0679 (14)	
H4A	0.3857	1.1342	0.2475	0.082*	
H4B	0.3996	0.9704	0.2437	0.082*	
C5	0.5135 (3)	1.0749 (5)	0.25804 (14)	0.0564 (12)	
C6	0.5905 (3)	1.0203 (4)	0.23887 (12)	0.0459 (11)	
C7	0.6800 (3)	1.0348 (5)	0.25897 (12)	0.0498 (11)	
H7	0.6740	1.0222	0.2865	0.060*	

C8	0.7389 (3)	0.9208 (5)	0.24394 (12)	0.0490 (11)	
C9	0.8224 (3)	0.8699 (5)	0.26047 (14)	0.0564 (12)	
C10	0.8621 (3)	0.7786 (5)	0.23056 (13)	0.0544 (12)	
C11	0.9410 (3)	0.7068 (6)	0.23092 (16)	0.0706 (15)	
H11	0.9790	0.7087	0.2524	0.085*	
C12	0.9632 (3)	0.6312 (6)	0.19881 (18)	0.0786 (17)	
H12	1.0170	0.5830	0.1984	0.094*	
C13	0.9065 (3)	0.6274 (6)	0.16784 (16)	0.0738 (16)	
H13	0.9220	0.5752	0.1466	0.089*	
C14	0.8258 (3)	0.6994 (5)	0.16693 (13)	0.0599 (13)	
H14	0.7876	0.6953	0.1456	0.072*	
C15	0.8040 (3)	0.7768 (4)	0.19858 (13)	0.0498 (11)	
C16	0.7269 (3)	0.8678 (4)	0.20865 (12)	0.0445 (10)	
C17	0.6483 (3)	0.8550 (5)	0.14690 (11)	0.0474 (11)	
C18	0.6921 (3)	0.9378 (5)	0.12114 (13)	0.0623 (13)	
H18	0.7196	1.0213	0.1292	0.075*	
C19	0.6952 (4)	0.8976 (7)	0.08379 (15)	0.0793 (17)	
H19	0.7249	0.9528	0.0664	0.095*	
C20	0.6540 (4)	0.7749 (8)	0.07245 (15)	0.0832 (18)	
H20	0.6567	0.7461	0.0472	0.100*	
C21	0.6089 (4)	0.6938 (6)	0.09748 (16)	0.0785 (16)	
H21	0.5802	0.6118	0.0891	0.094*	
C22	0.6059 (3)	0.7337 (5)	0.13550 (13)	0.0610 (13)	
H22	0.5755	0.6790	0.1528	0.073*	
C23	0.3377 (3)	1.0490 (6)	0.17598 (17)	0.0883 (18)	
H23A	0.3411	1.0593	0.1489	0.132*	
H23B	0.2955	1.1154	0.1854	0.132*	
H23C	0.3193	0.9544	0.1818	0.132*	
C24	0.4563 (3)	1.2286 (5)	0.18438 (16)	0.0802 (16)	
H24A	0.4587	1.2370	0.1572	0.120*	
H24B	0.5137	1.2484	0.1958	0.120*	
H24C	0.4139	1.2948	0.1937	0.120*	
C25	0.7215 (3)	1.1783 (5)	0.25128 (13)	0.0554 (12)	
C26	0.7775 (17)	1.261 (4)	0.2764 (13)	0.0926 (19)	0.543 (6)
H26	0.7894	1.2442	0.3022	0.111*	0.543 (6)
C26'	0.712 (3)	1.265 (5)	0.2174 (12)	0.0685 (15)	0.457 (6)
H26'	0.6744	1.2440	0.1967	0.082*	0.457 (6)
S2	0.2967 (4)	−0.0878 (10)	0.0720 (3)	0.0936 (17)	0.476 (7)
S2'	0.4021 (4)	0.0998 (7)	0.0333 (2)	0.1040 (18)	0.524 (7)
N2	0.0808 (2)	0.1273 (4)	0.08187 (9)	0.0493 (9)	
O3	0.2963 (2)	0.3737 (4)	0.01357 (9)	0.0806 (11)	
O4	0.1601 (2)	−0.1504 (4)	−0.02383 (9)	0.0763 (10)	
C27	0.8141 (5)	1.3828 (8)	0.2523 (3)	0.123 (3)	
H27	0.8601	1.4435	0.2598	0.148*	
C28	0.7699 (4)	1.3866 (7)	0.2198 (2)	0.098 (2)	
H28	0.7773	1.4603	0.2026	0.117*	
C29	0.1360 (3)	0.2450 (4)	0.07598 (11)	0.0442 (10)	
C30	0.1206 (3)	0.3714 (5)	0.10037 (12)	0.0542 (12)	
H30A	0.1128	0.3398	0.1263	0.065*	

H30B	0.0659	0.4168	0.0916	0.065*	
C31	0.1951 (3)	0.4805 (5)	0.10045 (12)	0.0559 (12)	
C32	0.2206 (4)	0.5062 (5)	0.05950 (13)	0.0661 (14)	
H32A	0.1719	0.5537	0.0459	0.079*	
H32B	0.2716	0.5688	0.0595	0.079*	
C33	0.2422 (3)	0.3727 (5)	0.03884 (12)	0.0572 (12)	
C34	0.1954 (3)	0.2432 (5)	0.04826 (11)	0.0460 (10)	
C35	0.2177 (3)	0.1104 (5)	0.02597 (13)	0.0569 (12)	
H35	0.2257	0.1364	−0.0007	0.068*	
C36	0.1408 (3)	0.0124 (5)	0.02794 (11)	0.0486 (11)	
C37	0.1215 (3)	−0.1105 (5)	0.00424 (13)	0.0556 (12)	
C38	0.0437 (3)	−0.1811 (5)	0.02148 (13)	0.0539 (12)	
C39	−0.0007 (3)	−0.3029 (5)	0.01027 (14)	0.0662 (14)	
H39	0.0160	−0.3534	−0.0111	0.079*	
C40	−0.0705 (3)	−0.3475 (5)	0.03154 (16)	0.0699 (14)	
H40	−0.1008	−0.4300	0.0248	0.084*	
C41	−0.0953 (3)	−0.2719 (6)	0.06239 (15)	0.0683 (14)	
H41	−0.1424	−0.3042	0.0764	0.082*	
C42	−0.0524 (3)	−0.1487 (5)	0.07346 (14)	0.0619 (13)	
H42	−0.0713	−0.0972	0.0942	0.074*	
C43	0.0185 (3)	−0.1034 (5)	0.05336 (12)	0.0498 (11)	
C44	0.0815 (3)	0.0184 (4)	0.05583 (11)	0.0458 (11)	
C45	0.0243 (3)	0.1215 (5)	0.11418 (11)	0.0468 (11)	
C46	−0.0549 (3)	0.1942 (5)	0.11302 (13)	0.0583 (12)	
H46	−0.0719	0.2474	0.0917	0.070*	
C47	−0.1084 (3)	0.1871 (6)	0.14389 (15)	0.0687 (14)	
H47	−0.1616	0.2371	0.1437	0.082*	
C48	−0.0838 (4)	0.1068 (6)	0.17489 (16)	0.0767 (16)	
H48	−0.1202	0.1026	0.1957	0.092*	
C49	−0.0059 (4)	0.0328 (6)	0.17558 (14)	0.0773 (16)	
H49	0.0099	−0.0232	0.1965	0.093*	
C50	0.0495 (3)	0.0413 (5)	0.14496 (13)	0.0608 (13)	
H50	0.1033	−0.0071	0.1454	0.073*	
C51	0.1621 (4)	0.6175 (5)	0.11844 (14)	0.0810 (16)	
H51A	0.1467	0.5995	0.1443	0.121*	
H51B	0.1108	0.6512	0.1042	0.121*	
H51C	0.2081	0.6878	0.1181	0.121*	
C52	0.2756 (3)	0.4261 (6)	0.12348 (14)	0.0776 (16)	
H52A	0.2596	0.4096	0.1493	0.116*	
H52B	0.3222	0.4953	0.1231	0.116*	
H52C	0.2957	0.3391	0.1125	0.116*	
C53	0.3016 (3)	0.0397 (5)	0.04134 (14)	0.0641 (13)	
C54	0.391 (2)	0.056 (3)	0.0365 (10)	0.1040 (18)	0.476 (7)
H54	0.4115	0.1232	0.0196	0.125*	0.476 (7)
C54'	0.3161 (18)	−0.075 (4)	0.0645 (9)	0.0936 (17)	0.524 (7)
H54'	0.2679	−0.1257	0.0730	0.112*	0.524 (7)
C55	0.4506 (4)	−0.0278 (8)	0.0567 (2)	0.105 (2)	
H55	0.5122	−0.0235	0.0571	0.126*	
C56	0.4000 (4)	−0.1158 (7)	0.07549 (19)	0.098 (2)	

H56	0.4238	−0.1900	0.0900	0.117*
N3	0.6516 (7)	0.3356 (7)	0.0652 (2)	0.135 (3)
O5	0.7733 (7)	0.4293 (9)	0.0982 (3)	0.244 (5)
C57	0.7339 (10)	0.3999 (12)	0.0655 (3)	0.162 (4)
H57	0.7607	0.4219	0.0428	0.194*
C58	0.6104 (8)	0.3013 (9)	0.0998 (3)	0.200 (5)
H58A	0.5541	0.2571	0.0942	0.300*
H58B	0.6016	0.3863	0.1142	0.300*
H58C	0.6477	0.2374	0.1144	0.300*
C59	0.6157 (7)	0.3105 (10)	0.0280 (3)	0.182 (4)
H59A	0.5589	0.2655	0.0295	0.273*
H59B	0.6550	0.2501	0.0144	0.273*
H59C	0.6089	0.3990	0.0147	0.273*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.044 (2)	0.053 (2)	0.045 (2)	0.0089 (18)	0.0081 (17)	0.0009 (17)
N2	0.049 (2)	0.049 (2)	0.050 (2)	0.0016 (19)	0.0102 (17)	−0.0059 (18)
N3	0.190 (8)	0.088 (5)	0.130 (6)	−0.009 (5)	0.038 (6)	0.000 (4)
O1	0.089 (3)	0.091 (3)	0.072 (2)	0.002 (2)	0.026 (2)	−0.025 (2)
O2	0.073 (2)	0.091 (3)	0.071 (2)	0.004 (2)	−0.010 (2)	0.008 (2)
O3	0.100 (3)	0.074 (3)	0.070 (2)	−0.009 (2)	0.037 (2)	−0.0011 (18)
O4	0.082 (2)	0.083 (3)	0.064 (2)	0.013 (2)	0.0086 (19)	−0.0265 (19)
O5	0.287 (10)	0.195 (8)	0.243 (9)	0.078 (7)	−0.096 (8)	−0.033 (7)
S1	0.069 (3)	0.0574 (16)	0.080 (3)	−0.0026 (15)	0.022 (2)	0.012 (2)
S2	0.063 (3)	0.102 (3)	0.117 (4)	0.010 (3)	0.008 (2)	0.033 (3)
C26'	0.069 (3)	0.0574 (16)	0.080 (3)	−0.0026 (15)	0.022 (2)	0.012 (2)
C54'	0.063 (3)	0.102 (3)	0.117 (4)	0.010 (3)	0.008 (2)	0.033 (3)
C1	0.042 (2)	0.043 (3)	0.049 (3)	0.004 (2)	0.010 (2)	0.004 (2)
C2	0.047 (3)	0.052 (3)	0.060 (3)	0.009 (2)	0.004 (2)	−0.006 (2)
C3	0.041 (3)	0.059 (3)	0.076 (3)	0.011 (2)	0.006 (2)	0.001 (2)
C4	0.048 (3)	0.077 (4)	0.081 (4)	0.006 (3)	0.023 (3)	−0.007 (3)
C5	0.057 (3)	0.052 (3)	0.061 (3)	−0.003 (2)	0.021 (2)	−0.005 (2)
C6	0.044 (2)	0.045 (3)	0.050 (3)	−0.001 (2)	0.011 (2)	0.002 (2)
C7	0.056 (3)	0.051 (3)	0.043 (2)	−0.003 (2)	0.007 (2)	0.003 (2)
C8	0.050 (3)	0.044 (3)	0.052 (3)	−0.001 (2)	0.003 (2)	0.005 (2)
C9	0.054 (3)	0.055 (3)	0.060 (3)	−0.003 (2)	0.001 (2)	0.016 (2)
C10	0.045 (3)	0.052 (3)	0.066 (3)	0.002 (2)	0.004 (2)	0.019 (2)
C11	0.051 (3)	0.078 (4)	0.084 (4)	0.008 (3)	0.005 (3)	0.030 (3)
C12	0.057 (3)	0.083 (4)	0.098 (4)	0.026 (3)	0.028 (3)	0.030 (3)
C13	0.063 (3)	0.076 (4)	0.084 (4)	0.026 (3)	0.023 (3)	0.019 (3)
C14	0.054 (3)	0.061 (3)	0.065 (3)	0.017 (3)	0.009 (2)	0.012 (2)
C15	0.047 (3)	0.043 (3)	0.060 (3)	0.010 (2)	0.016 (2)	0.012 (2)
C16	0.043 (2)	0.039 (3)	0.052 (3)	0.003 (2)	0.006 (2)	0.007 (2)
C17	0.044 (2)	0.054 (3)	0.044 (2)	0.011 (2)	0.007 (2)	0.002 (2)
C18	0.060 (3)	0.072 (4)	0.055 (3)	0.007 (3)	0.012 (2)	0.008 (3)
C19	0.080 (4)	0.103 (5)	0.056 (3)	0.024 (4)	0.019 (3)	0.013 (3)

C20	0.086 (4)	0.116 (6)	0.048 (3)	0.038 (4)	0.006 (3)	−0.006 (3)
C21	0.086 (4)	0.074 (4)	0.075 (4)	0.014 (3)	0.004 (3)	−0.025 (3)
C22	0.064 (3)	0.059 (3)	0.061 (3)	0.006 (3)	0.012 (2)	−0.003 (3)
C23	0.056 (3)	0.095 (5)	0.115 (5)	0.015 (3)	0.005 (3)	−0.015 (4)
C24	0.072 (4)	0.062 (4)	0.107 (4)	0.018 (3)	0.008 (3)	0.009 (3)
C25	0.050 (3)	0.045 (3)	0.071 (3)	−0.002 (2)	0.014 (2)	0.002 (2)
C26	0.073 (4)	0.086 (3)	0.117 (3)	−0.014 (4)	−0.010 (4)	−0.0083 (19)
S1'	0.073 (4)	0.086 (3)	0.117 (3)	−0.014 (4)	−0.010 (4)	−0.0083 (19)
C27	0.072 (5)	0.080 (5)	0.218 (9)	−0.026 (4)	0.008 (5)	−0.019 (6)
C28	0.078 (4)	0.062 (4)	0.157 (7)	−0.003 (4)	0.047 (4)	0.026 (4)
C29	0.047 (2)	0.041 (3)	0.045 (2)	0.006 (2)	0.002 (2)	−0.003 (2)
C30	0.057 (3)	0.051 (3)	0.056 (3)	0.000 (2)	0.008 (2)	−0.006 (2)
C31	0.067 (3)	0.049 (3)	0.052 (3)	0.002 (3)	0.008 (2)	0.002 (2)
C32	0.084 (4)	0.052 (3)	0.063 (3)	−0.001 (3)	0.017 (3)	0.001 (2)
C33	0.063 (3)	0.060 (3)	0.049 (3)	0.004 (3)	0.011 (2)	0.004 (2)
C34	0.050 (2)	0.044 (3)	0.044 (2)	0.002 (2)	0.008 (2)	−0.002 (2)
C35	0.051 (3)	0.061 (3)	0.060 (3)	0.007 (2)	0.013 (2)	0.003 (2)
C36	0.048 (3)	0.052 (3)	0.046 (2)	0.011 (2)	0.000 (2)	−0.006 (2)
C37	0.056 (3)	0.059 (3)	0.051 (3)	0.016 (3)	−0.007 (2)	−0.007 (2)
C38	0.049 (3)	0.050 (3)	0.062 (3)	0.011 (2)	−0.010 (2)	−0.006 (2)
C39	0.063 (3)	0.061 (4)	0.073 (3)	0.014 (3)	−0.017 (3)	−0.018 (3)
C40	0.055 (3)	0.058 (4)	0.096 (4)	0.005 (3)	−0.014 (3)	−0.014 (3)
C41	0.051 (3)	0.062 (4)	0.092 (4)	−0.002 (3)	−0.005 (3)	−0.009 (3)
C42	0.048 (3)	0.060 (3)	0.078 (3)	−0.002 (3)	0.004 (2)	−0.008 (3)
C43	0.044 (2)	0.049 (3)	0.056 (3)	0.008 (2)	−0.008 (2)	−0.010 (2)
C44	0.045 (2)	0.046 (3)	0.046 (2)	0.007 (2)	−0.001 (2)	−0.007 (2)
C45	0.051 (3)	0.047 (3)	0.043 (2)	−0.006 (2)	0.007 (2)	−0.008 (2)
C46	0.056 (3)	0.060 (3)	0.059 (3)	0.001 (3)	0.010 (2)	−0.001 (2)
C47	0.060 (3)	0.073 (4)	0.074 (4)	−0.001 (3)	0.016 (3)	−0.007 (3)
C48	0.070 (4)	0.090 (4)	0.072 (4)	−0.023 (3)	0.027 (3)	−0.011 (3)
C49	0.087 (4)	0.088 (4)	0.057 (3)	−0.021 (4)	0.006 (3)	0.010 (3)
C50	0.060 (3)	0.065 (3)	0.058 (3)	−0.001 (3)	0.000 (2)	0.007 (2)
C51	0.108 (4)	0.055 (3)	0.081 (4)	−0.008 (3)	0.031 (3)	−0.017 (3)
C52	0.074 (4)	0.089 (4)	0.069 (3)	−0.009 (3)	−0.008 (3)	−0.002 (3)
C53	0.046 (3)	0.067 (4)	0.080 (3)	0.009 (3)	0.008 (2)	0.005 (3)
C54	0.054 (2)	0.107 (4)	0.152 (3)	0.003 (2)	0.0224 (19)	0.049 (3)
S2'	0.054 (2)	0.107 (4)	0.152 (3)	0.003 (2)	0.0224 (19)	0.049 (3)
C55	0.056 (4)	0.113 (6)	0.147 (6)	0.018 (4)	0.002 (4)	0.015 (5)
C56	0.074 (4)	0.096 (5)	0.123 (5)	0.025 (4)	−0.010 (4)	0.023 (4)
C57	0.212 (13)	0.125 (9)	0.148 (9)	0.054 (9)	0.000 (9)	0.007 (7)
C58	0.310 (14)	0.101 (7)	0.196 (10)	0.007 (8)	0.125 (10)	0.024 (6)
C59	0.231 (11)	0.161 (9)	0.153 (9)	−0.078 (8)	−0.009 (8)	−0.003 (7)

Geometric parameters (Å, °)

N1—C16	1.377 (5)	C25—C26	1.43 (4)
N1—C1	1.416 (5)	C25—S1'	1.659 (12)
N1—C17	1.439 (5)	C26—C27	1.54 (4)
N2—C44	1.376 (5)	C26—H26	0.9300

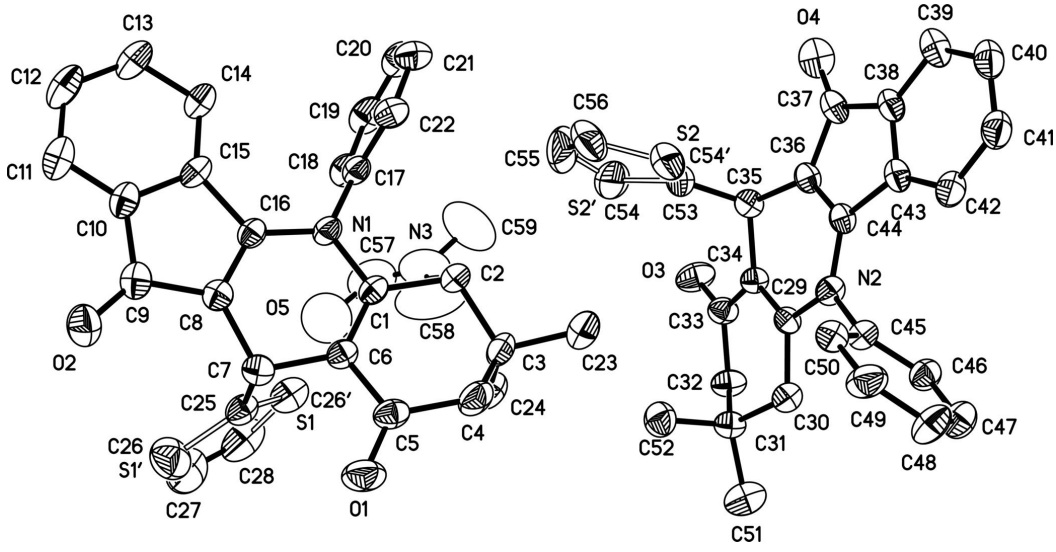
N2—C29	1.408 (5)	S1'—C27	1.539 (16)
N2—C45	1.444 (5)	C27—C28	1.302 (9)
N3—C57	1.381 (13)	C27—H27	0.9300
N3—C59	1.417 (10)	C28—H28	0.9300
N3—C58	1.422 (9)	C29—C34	1.346 (5)
O1—C5	1.223 (5)	C29—C30	1.491 (5)
O2—C9	1.228 (5)	C30—C31	1.524 (6)
O3—C33	1.226 (5)	C30—H30A	0.9700
O4—C37	1.222 (5)	C30—H30B	0.9700
O5—C57	1.305 (11)	C31—C32	1.521 (6)
S1—C28	1.593 (14)	C31—C52	1.524 (6)
S1—C25	1.643 (11)	C31—C51	1.530 (6)
S2—C56	1.581 (10)	C32—C33	1.496 (6)
S2—C53	1.617 (10)	C32—H32A	0.9700
C26'—C28	1.44 (5)	C32—H32B	0.9700
C26'—C25	1.44 (4)	C33—C34	1.456 (6)
C26'—H26'	0.9300	C34—C35	1.521 (6)
C54'—C53	1.37 (3)	C35—C36	1.487 (6)
C54'—C56	1.37 (3)	C35—C53	1.512 (6)
C54'—H54'	0.9300	C35—H35	0.9800
C1—C6	1.351 (5)	C36—C44	1.350 (5)
C1—C2	1.497 (6)	C36—C37	1.451 (6)
C2—C3	1.526 (6)	C37—C38	1.496 (6)
C2—H2A	0.9700	C38—C39	1.380 (6)
C2—H2B	0.9700	C38—C43	1.402 (6)
C3—C23	1.521 (6)	C39—C40	1.378 (7)
C3—C4	1.523 (6)	C39—H39	0.9300
C3—C24	1.535 (6)	C40—C41	1.360 (6)
C4—C5	1.496 (6)	C40—H40	0.9300
C4—H4A	0.9700	C41—C42	1.380 (6)
C4—H4B	0.9700	C41—H41	0.9300
C5—C6	1.456 (6)	C42—C43	1.370 (6)
C6—C7	1.508 (6)	C42—H42	0.9300
C7—C8	1.502 (6)	C43—C44	1.492 (6)
C7—C25	1.520 (6)	C45—C50	1.362 (6)
C7—H7	0.9800	C45—C46	1.376 (6)
C8—C16	1.342 (5)	C46—C47	1.376 (6)
C8—C9	1.449 (6)	C46—H46	0.9300
C9—C10	1.500 (6)	C47—C48	1.367 (7)
C10—C11	1.369 (6)	C47—H47	0.9300
C10—C15	1.400 (6)	C48—C49	1.365 (7)
C11—C12	1.385 (7)	C48—H48	0.9300
C11—H11	0.9300	C49—C50	1.388 (6)
C12—C13	1.360 (7)	C49—H49	0.9300
C12—H12	0.9300	C50—H50	0.9300
C13—C14	1.393 (6)	C51—H51A	0.9600
C13—H13	0.9300	C51—H51B	0.9600
C14—C15	1.379 (6)	C51—H51C	0.9600
C14—H14	0.9300	C52—H52A	0.9600

C15—C16	1.497 (5)	C52—H52B	0.9600
C17—C22	1.365 (6)	C52—H52C	0.9600
C17—C18	1.381 (6)	C53—C54	1.38 (3)
C18—C19	1.367 (6)	C53—S2'	1.652 (7)
C18—H18	0.9300	C54—C55	1.37 (3)
C19—C20	1.367 (8)	C54—H54	0.9300
C19—H19	0.9300	S2'—C55	1.619 (10)
C20—C21	1.365 (8)	C55—C56	1.319 (8)
C20—H20	0.9300	C55—H55	0.9300
C21—C22	1.389 (6)	C56—H56	0.9300
C21—H21	0.9300	C57—H57	0.9300
C22—H22	0.9300	C58—H58A	0.9600
C23—H23A	0.9600	C58—H58B	0.9600
C23—H23B	0.9600	C58—H58C	0.9600
C23—H23C	0.9600	C59—H59A	0.9600
C24—H24A	0.9600	C59—H59B	0.9600
C24—H24B	0.9600	C59—H59C	0.9600
C24—H24C	0.9600		
C16—N1—C1	118.3 (3)	S1—C28—H28	121.2
C16—N1—C17	119.8 (3)	C34—C29—N2	120.6 (4)
C1—N1—C17	121.9 (3)	C34—C29—C30	123.1 (4)
C44—N2—C29	118.2 (3)	N2—C29—C30	116.2 (3)
C44—N2—C45	120.8 (3)	C29—C30—C31	114.3 (3)
C29—N2—C45	120.9 (3)	C29—C30—H30A	108.7
C57—N3—C59	113.2 (9)	C31—C30—H30A	108.7
C57—N3—C58	120.9 (10)	C29—C30—H30B	108.7
C59—N3—C58	125.9 (10)	C31—C30—H30B	108.7
C28—S1—C25	94.8 (5)	H30A—C30—H30B	107.6
C56—S2—C53	96.2 (4)	C32—C31—C30	108.5 (4)
C28—C26'—C25	111 (3)	C32—C31—C52	109.4 (4)
C28—C26'—H26'	124.3	C30—C31—C52	110.3 (4)
C25—C26'—H26'	124.3	C32—C31—C51	110.6 (4)
C53—C54'—C56	121.4 (18)	C30—C31—C51	108.8 (4)
C53—C54'—H54'	119.3	C52—C31—C51	109.3 (4)
C56—C54'—H54'	119.3	C33—C32—C31	113.0 (4)
C6—C1—N1	120.1 (4)	C33—C32—H32A	109.0
C6—C1—C2	123.7 (4)	C31—C32—H32A	109.0
N1—C1—C2	116.2 (3)	C33—C32—H32B	109.0
C1—C2—C3	113.7 (4)	C31—C32—H32B	109.0
C1—C2—H2A	108.8	H32A—C32—H32B	107.8
C3—C2—H2A	108.8	O3—C33—C34	120.8 (4)
C1—C2—H2B	108.8	O3—C33—C32	120.4 (4)
C3—C2—H2B	108.8	C34—C33—C32	118.7 (4)
H2A—C2—H2B	107.7	C29—C34—C33	119.7 (4)
C23—C3—C4	110.5 (4)	C29—C34—C35	123.2 (4)
C23—C3—C2	109.2 (4)	C33—C34—C35	117.2 (3)
C4—C3—C2	108.0 (4)	C36—C35—C53	110.6 (4)
C23—C3—C24	108.1 (4)	C36—C35—C34	107.5 (3)
C4—C3—C24	110.1 (4)	C53—C35—C34	112.2 (4)

C2—C3—C24	110.8 (4)	C36—C35—H35	108.8
C5—C4—C3	113.6 (4)	C53—C35—H35	108.8
C5—C4—H4A	108.9	C34—C35—H35	108.8
C3—C4—H4A	108.9	C44—C36—C37	108.9 (4)
C5—C4—H4B	108.9	C44—C36—C35	123.1 (4)
C3—C4—H4B	108.9	C37—C36—C35	127.7 (4)
H4A—C4—H4B	107.7	O4—C37—C36	128.1 (5)
O1—C5—C6	120.4 (4)	O4—C37—C38	126.3 (4)
O1—C5—C4	121.8 (4)	C36—C37—C38	105.6 (4)
C6—C5—C4	117.8 (4)	C39—C38—C43	121.4 (4)
C1—C6—C5	119.6 (4)	C39—C38—C37	129.4 (4)
C1—C6—C7	122.5 (4)	C43—C38—C37	109.2 (4)
C5—C6—C7	117.8 (4)	C40—C39—C38	118.2 (5)
C8—C7—C6	107.5 (3)	C40—C39—H39	120.9
C8—C7—C25	108.9 (3)	C38—C39—H39	120.9
C6—C7—C25	111.4 (4)	C41—C40—C39	120.5 (5)
C8—C7—H7	109.7	C41—C40—H40	119.7
C6—C7—H7	109.7	C39—C40—H40	119.7
C25—C7—H7	109.7	C40—C41—C42	121.8 (5)
C16—C8—C9	109.4 (4)	C40—C41—H41	119.1
C16—C8—C7	122.0 (4)	C42—C41—H41	119.1
C9—C8—C7	127.8 (4)	C43—C42—C41	119.0 (5)
O2—C9—C8	127.9 (5)	C43—C42—H42	120.5
O2—C9—C10	126.2 (4)	C41—C42—H42	120.5
C8—C9—C10	105.9 (4)	C42—C43—C38	119.1 (4)
C11—C10—C15	121.4 (5)	C42—C43—C44	135.9 (4)
C11—C10—C9	130.3 (5)	C38—C43—C44	105.0 (4)
C15—C10—C9	108.3 (4)	C36—C44—N2	122.3 (4)
C10—C11—C12	118.9 (5)	C36—C44—C43	111.3 (4)
C10—C11—H11	120.6	N2—C44—C43	126.4 (4)
C12—C11—H11	120.6	C50—C45—C46	121.2 (4)
C13—C12—C11	120.0 (5)	C50—C45—N2	119.2 (4)
C13—C12—H12	120.0	C46—C45—N2	119.5 (4)
C11—C12—H12	120.0	C47—C46—C45	119.0 (5)
C12—C13—C14	122.0 (5)	C47—C46—H46	120.5
C12—C13—H13	119.0	C45—C46—H46	120.5
C14—C13—H13	119.0	C48—C47—C46	120.2 (5)
C15—C14—C13	118.2 (5)	C48—C47—H47	119.9
C15—C14—H14	120.9	C46—C47—H47	119.9
C13—C14—H14	120.9	C49—C48—C47	120.5 (5)
C14—C15—C10	119.5 (4)	C49—C48—H48	119.8
C14—C15—C16	134.7 (4)	C47—C48—H48	119.8
C10—C15—C16	105.8 (4)	C48—C49—C50	119.9 (5)
C8—C16—N1	122.1 (4)	C48—C49—H49	120.1
C8—C16—C15	110.6 (4)	C50—C49—H49	120.1
N1—C16—C15	127.3 (4)	C45—C50—C49	119.2 (5)
C22—C17—C18	120.9 (4)	C45—C50—H50	120.4
C22—C17—N1	120.7 (4)	C49—C50—H50	120.4
C18—C17—N1	118.3 (4)	C31—C51—H51A	109.5

C19—C18—C17	120.3 (5)	C31—C51—H51B	109.5
C19—C18—H18	119.9	H51A—C51—H51B	109.5
C17—C18—H18	119.9	C31—C51—H51C	109.5
C18—C19—C20	118.9 (5)	H51A—C51—H51C	109.5
C18—C19—H19	120.5	H51B—C51—H51C	109.5
C20—C19—H19	120.5	C31—C52—H52A	109.5
C21—C20—C19	121.3 (5)	C31—C52—H52B	109.5
C21—C20—H20	119.3	H52A—C52—H52B	109.5
C19—C20—H20	119.3	C31—C52—H52C	109.5
C20—C21—C22	120.0 (5)	H52A—C52—H52C	109.5
C20—C21—H21	120.0	H52B—C52—H52C	109.5
C22—C21—H21	120.0	C54'—C53—C54	91.3 (16)
C17—C22—C21	118.6 (5)	C54'—C53—C35	132.6 (11)
C17—C22—H22	120.7	C54—C53—C35	136.0 (13)
C21—C22—H22	120.7	C54—C53—S2	103.4 (12)
C3—C23—H23A	109.5	C35—C53—S2	120.5 (4)
C3—C23—H23B	109.5	C54'—C53—S2'	104.2 (11)
H23A—C23—H23B	109.5	C35—C53—S2'	123.2 (4)
C3—C23—H23C	109.5	S2—C53—S2'	115.7 (5)
H23A—C23—H23C	109.5	C55—C54—C53	119.9 (18)
H23B—C23—H23C	109.5	C55—C54—H54	120.0
C3—C24—H24A	109.5	C53—C54—H54	120.0
C3—C24—H24B	109.5	C55—S2'—C53	93.3 (4)
H24A—C24—H24B	109.5	C56—C55—C54	104.0 (11)
C3—C24—H24C	109.5	C56—C55—S2'	117.6 (5)
H24A—C24—H24C	109.5	C56—C55—H55	128.0
H24B—C24—H24C	109.5	C54—C55—H55	128.0
C26—C25—C26'	104 (3)	S2'—C55—H55	114.0
C26—C25—C7	127.8 (18)	C55—C56—C54'	103.1 (12)
C26'—C25—C7	128.4 (19)	C55—C56—S2	116.2 (6)
C26—C25—S1	111.3 (18)	C55—C56—H56	121.9
C7—C25—S1	120.9 (5)	C54'—C56—H56	134.7
C26'—C25—S1'	105.8 (19)	S2—C56—H56	121.9
C7—C25—S1'	124.8 (5)	O5—C57—N3	118.7 (13)
S1—C25—S1'	113.2 (6)	O5—C57—H57	120.6
C25—C26—C27	106 (3)	N3—C57—H57	120.6
C25—C26—H26	126.8	N3—C58—H58A	109.5
C27—C26—H26	126.8	N3—C58—H58B	109.5
C27—S1'—C25	96.2 (7)	H58A—C58—H58B	109.5
C28—C27—S1'	117.7 (7)	N3—C58—H58C	109.5
C28—C27—C26	108.6 (15)	H58A—C58—H58C	109.5
C28—C27—H27	125.7	H58B—C58—H58C	109.5
S1'—C27—H27	115.5	N3—C59—H59A	109.5
C26—C27—H27	125.7	N3—C59—H59B	109.5
C27—C28—C26'	108.5 (18)	H59A—C59—H59B	109.5
C27—C28—S1	117.5 (6)	N3—C59—H59C	109.5
C27—C28—H28	121.2	H59A—C59—H59C	109.5
C26'—C28—H28	130.3	H59B—C59—H59C	109.5

Fig. 1


C13
C12
C14
C20
C21
C19
C22
C11
C15
C18
C17
C16
C10
N1
C57
N3
C59
C2
C9
C8
O5
C1
C58
O2
C7
C6
C3
C23
C25
C26'
S1
C24
C26
S1'
C28
C5
C4
C27
O1
O4
C39
C40
C38
C37
C56
S2
C41
C55
C54'
C36
C35
S2'
C54
C53
C43
C42
C44
C34
O3
C29
N2
C33
C45
C50
C46
C32
C30
C49
C47
C52
C31
C48
C51