Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(4-Chlorophenyl)-2-(2,4-dichlorophenoxy)-5-methyl-8,9,10,11-tetrahydro-2-benzothieno[2′,3′:2,3]pyrido[4,5-*d*]-pyrimidin-4(3*H*)-one dichloromethane solvate

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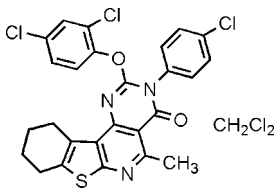
Received 6 October 2007; accepted 29 October 2007

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.007 Å; disorder in solvent or counterion; R factor = 0.078; wR factor = 0.234; data-to-parameter ratio = 14.8.

In the structure of the title compound, $C_{26}H_{18}Cl_3N_3O_2S\cdot CH_2Cl_2$, the C—S bond lengths in the thiophene ring [1.733 (5) and 1.735 (5) Å] are equivalent and long compared with the values observed in both free thiophene, measured using electron diffraction, and thieno[2,3-*c*]pyridine. The central thienopyridine ring system is planar. The pyrimidinone ring forms a dihedral angle of 3.1 (2)° with the pyridine ring. The chloro- and dichlorophenyl rings form dihedral angles of 75.2 (2) and 85.5 (2)°, respectively, with the pyrimidinone ring. The dihedral angle between the chlorophenyl rings is 69.7 (2)°. The cyclohexene ring adopts a half-chair conformation. There is a C—H—O hydrogen bond between the main molecule and the dichoromethane molecule. The dichloromethane molecule is disordered over two positions, with refined occupancies of 0.775 (6) and 0.225 (6).

Related literature

For related literature, see: Bonham & Momany (1963); Bridges *et al.* (1997); Ghosh & Simonsen (1993); Ismail & Wibberley (1967); Liu *et al.* (2006).



Experimental

Crystal data

$C_{26}H_{18}Cl_3N_3O_2S\cdot CH_2Cl_2$
M_r = 627.77
Orthorhombic, *Pbca*
a = 18.295 (6) Å
b = 10.851 (4) Å
c = 28.097 (9) Å
V = 5578 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 292 (2) K
0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.885, T_{max} = 0.929
29034 measured reflections
5488 independent reflections
3296 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.234
S = 1.04
5488 reflections
372 parameters
41 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C27—H27A···O1^{i}	0.97	2.44	3.23 (2)	138

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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, 1998); software used to prepare material for publication: *SHELXTL*.

The author thanks the National Natural Science Foundation of China (grant No. 20672041) and the Post Doctoral Science Foundation of Central South University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2481).

References

Bonham, R. A. & Momany, F. A. (1963). *J. Phys. Chem.* **67**, 2474–2477.
Bridges, A. J., Denny, W. A., Fry, D., Kraker, A., Meyer, R. F., Rewcastle, G. W. & Thompson, A. M. (1997). US Patent No. 5 654 307.
Bruker (1998). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ghosh, R. & Simonsen, S. H. (1993). *Acta Cryst.* C**49**, 1031–1032.
Ismail, A. G. & Wibberley, D. G. (1967). *J. Chem. Soc. C*, pp. 2613–2616.
Liu, J. C., He, H. W., Ren, Q. Y. & Ding, M. W. (2006). *Helv. Chim. Acta*, **89**, 1337–1343.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*, University of Göttingen, Germany.

 doi:10.1107/S1600536807054013

supplementary materials

Acta Cryst. (2007). E**63**, o4571 [**doi:10.1107/S1600536807054013**]

3-(4-Chlorophenyl)-2-(2,4-dichlorophenoxy)-5-methyl-8,9,10,11-tetrahydro-2-benzothieno[2',3':2,3]pyrido[4,5-*d*]pyrimidin-4(3*H*)-one dichloromethane solvate

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Comment

The pyridopyrimidine scaffold is a well known pharmacophore in drug design and it is associated with a wide range of biological properties. For example, many pyrido[4,3-*d*]pyrimidines have been described as inhibitors of tyrosine kinases of the epidermal growth factor receptor family (Bridges *et al.*, 1997). An important synthetic route to pyrido[4,3-*d*]pyrimidine is the condensation reaction of 4-aminonicotinic acid and amines (Ismail & Wibberley, 1967). However, this method often require long reaction time. Recently, we have developed a new and facile regioselective annulation process, which proceeds smoothly under mild condition *via* a tandem aza-Wittig and cyclization reaction, to synthesize novel pyrido[4,3-*d*]pyrimidine derivates (Liu *et al.*, 2006). In this paper, the crystal structure of the title compound is reported. The structure of the title compound was also characterized by ^{1}H NMR, MS and elemental analysis.

The molecular structure of (1) is shown in Fig. 1. The C—S bond lengths in the thiophene ring [1.733 (5) and 1.735 (5) Å] are almost equal and long compared with the values observed in free thiophene (1.714 Å; Bonham & Momany, 1963). The C8—S1—C5 [91.7 (2)°] angle is comparable to that observed in free thiophene [92.2 (2)°]. The C8—N1—C9 angle of 116.9 (4)° is typical of a non-protonated ring system, being smaller than 120° (Ghosh & Simonsen, 1993). The central thienopyridine ring system is nearly planar and the dihedral angle between the thiophene and pyridine planes is 1.6 (2)°. The pyrimidinone ring forms a dihedral angle of 3.1 (2)° with the pyridine ring. The chloro- and dichloro-phenyl rings form dihedral angles of 75.2 (2) and 85.5 (2)°, respectively, with the pyrimidinone ring. The dihedral angle between the chlorophenyl rings is 69.7 (2)°. The cyclohexene ring adopts a half-chair conformation. There exists a C27—H27A—O1 hydrogen bond between the main molecule and the dichoromethane molecule.

Experimental

The title compound was prepared according to the literature procedure of Liu *et al.* (2006) and suitable crystals were obtained by evaporation of a dichoromethane solution (m.p. 563–565 K). Analysis, calculated for $C_{27}H_{20}Cl_5N_3O_2S$: C 51.66, H 3.31, N 6.69%; found: C 51.71, H 3.40, N 6.78%. IR (KBr, ν, cm^{-1}): 3125(Ph—H), 2931,2863(C—H), 1705(C=O), 1620, 1562, 1517, 1490, 1365, 1252, 1092, 843. ^{1}H NMR($CDCl_3$, TMS, 400 MHz): 1.62–1.80(m, 4H, $2CH_2$), 2.34–2.79(m, 4H, $2CH_2$), 3.03(s, 3H, CH_3), 7.15–7.58(m, 7H, Ar—H). MS(EI, %): 544 (100), 543(M^+ 90), 396 (16), 380 (20).

Refinement

The dichloromethane molecule is disordered over two positions, with refined occupancies of 0.775 (6) and 0.225 (6). The disorder was modelled with a C—Cl distance restraint of 1.732 (9) Å, and the Cl···Cl distance in both disorder components were restrained to be equal. The displacement parameters of disordered atoms were restrained to an approximate isotropic

behaviour. H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms, with U_{iso}(H) = kU_{eq}(C) (k=1.5 for methyl and 1.2 for the other C atoms).

Figures

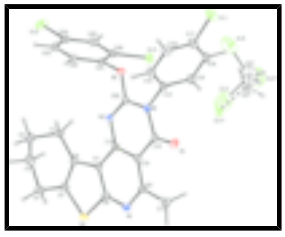


Fig. 1. The molecular structure of the title compound, showing 50% probablity displacement ellipsoids.

3-(4-Chlorophenyl)-2-(2,4-dichlorophenoxy)-5-methyl-8,9,10,11-tetrahydro- 2-benzothieno[2',3':2,3]pyrido[4,5-*d*]pyrimidin-4(3*H*)-one dichloromethane solvate

Crystal data

$C_{26}H_{18}Cl_3N_3O_2S·CH_2Cl_2$	F_{000} = 2560
M_r = 627.77	D_x = 1.495 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3031 reflections
a = 18.295 (6) Å	θ = 2.2–19.0º
b = 10.851 (4) Å	μ = 0.63 mm^{-1}
c = 28.097 (9) Å	*T* = 292 (2) K
V = 5578 (3) Å^3	Block, colourless
Z = 8	0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	5488 independent reflections
Radiation source: fine-focus sealed tube	3296 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.079
T = 292(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −21→22
T_{min} = 0.885, T_{max} = 0.929	*k* = −13→13
29034 measured reflections	*l* = −22→34

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.078	H-atom parameters constrained

$wR(F^2) = 0.234$	$w = 1/[\sigma^2(F_o^2) + (0.1242P)^2 + 1.4543P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
5488 reflections	$\Delta\rho_{max} = 0.64$ e Å^{-3}
372 parameters	$\Delta\rho_{min} = -0.52$ e Å^{-3}
41 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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)
S1	0.21511 (7)	0.80443 (11)	0.23989 (5)	0.0657 (4)	
O1	0.3537 (2)	0.4856 (4)	0.05713 (12)	0.0791 (11)	
O2	0.49710 (15)	0.3654 (3)	0.17882 (10)	0.0512 (7)	
N1	0.2325 (2)	0.7211 (4)	0.15028 (16)	0.0623 (11)	
N2	0.42421 (19)	0.4285 (3)	0.11922 (12)	0.0476 (9)	
N3	0.40778 (18)	0.5042 (3)	0.19699 (11)	0.0427 (8)	
Cl1	0.60808 (11)	0.10969 (19)	−0.00222 (6)	0.1144 (7)	
Cl2	0.61639 (11)	0.41703 (16)	0.37059 (5)	0.1062 (7)	
Cl3	0.61136 (8)	0.55092 (12)	0.18665 (5)	0.0791 (5)	
C1	0.3714 (2)	0.5894 (4)	0.29686 (16)	0.0510 (11)	
H1A	0.4207	0.6198	0.2920	0.061*	
H1B	0.3707	0.5026	0.2886	0.061*	
C2	0.3508 (3)	0.6043 (5)	0.34868 (18)	0.0732 (15)	
H2A	0.3103	0.5496	0.3556	0.088*	
H2B	0.3918	0.5785	0.3681	0.088*	
C3	0.3304 (3)	0.7282 (5)	0.3624 (2)	0.0808 (16)	
H3A	0.3719	0.7825	0.3578	0.097*	
H3B	0.3182	0.7289	0.3960	0.097*	
C4	0.2656 (3)	0.7775 (5)	0.33410 (19)	0.0667 (13)	
H4A	0.2205	0.7449	0.3472	0.080*	
H4B	0.2640	0.8666	0.3366	0.080*	
C5	0.2719 (2)	0.7413 (4)	0.28293 (17)	0.0541 (11)	
C6	0.3202 (2)	0.6575 (3)	0.26505 (16)	0.0460 (10)	
C7	0.3112 (2)	0.6435 (4)	0.21415 (15)	0.0447 (10)	

C8	0.2551 (2)	0.7161 (4)	0.19556 (17)	0.0536 (11)	
C9	0.2674 (2)	0.6507 (5)	0.11937 (17)	0.0609 (13)	
C10	0.3273 (2)	0.5734 (4)	0.13337 (15)	0.0494 (11)	
C11	0.2414 (3)	0.6608 (7)	0.06876 (19)	0.0904 (19)	
H11A	0.2814	0.6845	0.0487	0.136*	
H11B	0.2035	0.7217	0.0668	0.136*	
H11C	0.2226	0.5826	0.0585	0.136*	
C12	0.3655 (2)	0.4967 (4)	0.09934 (17)	0.0559 (11)	
C13	0.3492 (2)	0.5717 (3)	0.18081 (14)	0.0423 (10)	
C14	0.4672 (2)	0.3507 (4)	0.08798 (15)	0.0507 (11)	
C15	0.4589 (3)	0.2252 (4)	0.08967 (17)	0.0594 (12)	
H15	0.4249	0.1902	0.1102	0.071*	
C16	0.5012 (3)	0.1510 (5)	0.06094 (18)	0.0699 (14)	
H16	0.4957	0.0658	0.0616	0.084*	
C17	0.5513 (3)	0.2048 (6)	0.03150 (18)	0.0717 (15)	
C18	0.5596 (3)	0.3282 (6)	0.02942 (18)	0.0747 (15)	
H18	0.5939	0.3626	0.0089	0.090*	
C19	0.5168 (3)	0.4039 (5)	0.05788 (17)	0.0650 (13)	
H19	0.5218	0.4891	0.0565	0.078*	
C20	0.4402 (2)	0.4372 (4)	0.16607 (15)	0.0445 (10)	
C21	0.5227 (2)	0.3813 (4)	0.22526 (14)	0.0427 (10)	
C22	0.4961 (2)	0.3099 (4)	0.26109 (16)	0.0540 (11)	
H22	0.4590	0.2534	0.2553	0.065*	
C23	0.5249 (3)	0.3223 (5)	0.30592 (17)	0.0631 (13)	
H23	0.5068	0.2749	0.3309	0.076*	
C24	0.5799 (3)	0.4040 (5)	0.31384 (17)	0.0627 (13)	
C25	0.6071 (3)	0.4726 (4)	0.27805 (19)	0.0623 (13)	
H25	0.6455	0.5269	0.2837	0.075*	
C26	0.5783 (2)	0.4624 (4)	0.23344 (16)	0.0528 (11)	
C27	0.6539 (11)	0.7036 (15)	0.0314 (6)	0.249 (11)	0.775 (6)
H27A	0.6307	0.6766	0.0022	0.298*	0.775 (6)
H27B	0.6912	0.7639	0.0237	0.298*	0.775 (6)
Cl4	0.5897 (7)	0.7657 (11)	0.0703 (3)	0.335 (6)	0.775 (6)
Cl5	0.6906 (2)	0.5827 (6)	0.06091 (14)	0.211 (3)	0.775 (6)
C27'	0.6306 (10)	0.731 (2)	0.0331 (7)	0.072 (7)	0.225 (6)
H27C	0.6128	0.7420	0.0009	0.086*	0.225 (6)
H27D	0.6482	0.6468	0.0368	0.086*	0.225 (6)
Cl5'	0.6977 (11)	0.838 (2)	0.0475 (7)	0.267 (11)	0.225 (6)
Cl4'	0.5647 (6)	0.7663 (8)	0.0747 (3)	0.085 (3)	0.225 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0499 (7)	0.0544 (7)	0.0928 (10)	0.0157 (6)	0.0065 (6)	0.0087 (6)
O1	0.082 (3)	0.109 (3)	0.047 (2)	0.015 (2)	−0.0146 (17)	0.0038 (19)
O2	0.0472 (17)	0.0563 (17)	0.0500 (18)	0.0139 (14)	−0.0046 (13)	−0.0038 (14)
N1	0.044 (2)	0.071 (3)	0.071 (3)	0.0063 (19)	−0.001 (2)	0.024 (2)
N2	0.046 (2)	0.053 (2)	0.043 (2)	0.0004 (16)	−0.0011 (16)	0.0026 (16)

N3	0.0421 (19)	0.0427 (18)	0.043 (2)	0.0042 (15)	−0.0007 (15)	0.0063 (16)
Cl1	0.1140 (14)	0.1339 (15)	0.0954 (13)	0.0158 (11)	0.0231 (10)	−0.0474 (11)
Cl2	0.1456 (16)	0.1045 (12)	0.0685 (10)	0.0484 (11)	−0.0462 (9)	−0.0280 (8)
Cl3	0.0886 (10)	0.0617 (8)	0.0870 (10)	−0.0128 (7)	0.0238 (7)	0.0028 (7)
C1	0.050 (2)	0.043 (2)	0.061 (3)	0.0013 (19)	−0.004 (2)	−0.001 (2)
C2	0.089 (4)	0.072 (3)	0.059 (3)	0.007 (3)	−0.006 (3)	0.002 (3)
C3	0.084 (4)	0.089 (4)	0.069 (4)	0.000 (3)	0.007 (3)	−0.018 (3)
C4	0.057 (3)	0.057 (3)	0.085 (4)	0.002 (2)	0.009 (3)	−0.012 (3)
C5	0.050 (3)	0.048 (2)	0.064 (3)	−0.004 (2)	0.009 (2)	0.003 (2)
C6	0.038 (2)	0.033 (2)	0.067 (3)	−0.0028 (17)	0.0056 (19)	0.0030 (19)
C7	0.033 (2)	0.043 (2)	0.058 (3)	−0.0026 (18)	0.0029 (18)	0.014 (2)
C8	0.042 (2)	0.048 (2)	0.071 (3)	−0.0013 (19)	0.005 (2)	0.017 (2)
C9	0.045 (3)	0.075 (3)	0.062 (3)	0.001 (2)	−0.006 (2)	0.023 (3)
C10	0.041 (2)	0.055 (3)	0.052 (3)	−0.001 (2)	−0.0029 (19)	0.015 (2)
C11	0.064 (3)	0.138 (5)	0.070 (4)	0.026 (4)	−0.014 (3)	0.030 (3)
C12	0.048 (3)	0.067 (3)	0.052 (3)	−0.004 (2)	−0.003 (2)	0.016 (2)
C13	0.037 (2)	0.039 (2)	0.051 (3)	−0.0041 (17)	−0.0036 (18)	0.0141 (18)
C14	0.048 (3)	0.062 (3)	0.042 (2)	−0.006 (2)	−0.0007 (19)	−0.001 (2)
C15	0.061 (3)	0.060 (3)	0.058 (3)	−0.013 (2)	0.004 (2)	−0.002 (2)
C16	0.070 (3)	0.071 (3)	0.069 (3)	−0.009 (3)	−0.002 (3)	−0.018 (3)
C17	0.070 (3)	0.091 (4)	0.053 (3)	0.000 (3)	0.000 (3)	−0.025 (3)
C18	0.069 (3)	0.102 (5)	0.053 (3)	−0.001 (3)	0.018 (3)	−0.003 (3)
C19	0.071 (3)	0.064 (3)	0.060 (3)	−0.007 (3)	0.013 (3)	0.004 (2)
C20	0.041 (2)	0.044 (2)	0.049 (3)	−0.0026 (19)	−0.0001 (19)	0.005 (2)
C21	0.043 (2)	0.043 (2)	0.042 (2)	0.0144 (19)	−0.0028 (18)	−0.0020 (18)
C22	0.050 (3)	0.053 (3)	0.058 (3)	0.008 (2)	−0.001 (2)	0.004 (2)
C23	0.078 (3)	0.059 (3)	0.053 (3)	0.019 (3)	0.004 (2)	0.011 (2)
C24	0.077 (3)	0.057 (3)	0.055 (3)	0.027 (3)	−0.017 (3)	−0.015 (2)
C25	0.057 (3)	0.055 (3)	0.075 (4)	0.007 (2)	−0.012 (3)	−0.014 (3)
C26	0.050 (3)	0.045 (2)	0.064 (3)	0.007 (2)	0.008 (2)	−0.004 (2)
C27	0.278 (14)	0.259 (13)	0.208 (13)	−0.023 (10)	0.037 (10)	0.018 (9)
Cl4	0.387 (12)	0.353 (10)	0.266 (8)	0.012 (9)	−0.060 (8)	−0.009 (8)
Cl5	0.132 (3)	0.355 (7)	0.148 (3)	−0.034 (3)	−0.025 (2)	−0.082 (4)
C27'	0.070 (10)	0.098 (11)	0.047 (9)	−0.004 (8)	0.040 (8)	−0.002 (8)
Cl5'	0.300 (19)	0.288 (18)	0.213 (15)	−0.001 (15)	−0.024 (13)	0.008 (13)
Cl4'	0.127 (6)	0.092 (5)	0.037 (4)	0.006 (4)	0.029 (4)	−0.001 (3)

Geometric parameters (Å, °)

S1—C8	1.733 (5)	C9—C11	1.504 (7)
S1—C5	1.735 (5)	C10—C13	1.392 (6)
O1—C12	1.211 (5)	C10—C12	1.447 (7)
O2—C20	1.349 (5)	C11—H11A	0.96
O2—C21	1.397 (5)	C11—H11B	0.96
N1—C9	1.321 (6)	C11—H11C	0.96
N1—C8	1.338 (6)	C14—C19	1.369 (6)
N2—C20	1.352 (5)	C14—C15	1.372 (6)
N2—C12	1.419 (6)	C15—C16	1.377 (7)
N2—C14	1.449 (5)	C15—H15	0.93

N3—C20	1.279 (5)	C16—C17	1.367 (8)
N3—C13	1.375 (5)	C16—H16	0.93
Cl1—C17	1.744 (5)	C17—C18	1.349 (8)
Cl2—C24	1.735 (5)	C18—C19	1.387 (7)
Cl3—C26	1.737 (5)	C18—H18	0.93
C1—C6	1.490 (6)	C19—H19	0.93
C1—C2	1.513 (7)	C21—C22	1.360 (6)
C1—H1A	0.97	C21—C26	1.364 (6)
C1—H1B	0.97	C22—C23	1.372 (6)
C2—C3	1.447 (7)	C22—H22	0.93
C2—H2A	0.97	C23—C24	1.358 (7)
C2—H2B	0.97	C23—H23	0.93
C3—C4	1.525 (7)	C24—C25	1.347 (7)
C3—H3A	0.97	C25—C26	1.364 (6)
C3—H3B	0.97	C25—H25	0.93
C4—C5	1.495 (7)	C27—Cl5	1.691 (9)
C4—H4A	0.97	C27—Cl4	1.739 (9)
C4—H4B	0.97	C27—H27A	0.97
C5—C6	1.364 (6)	C27—H27B	0.97
C6—C7	1.448 (6)	C27'—Cl4'	1.724 (9)
C7—C8	1.396 (6)	C27'—Cl5'	1.740 (9)
C7—C13	1.403 (6)	C27'—H27C	0.97
C9—C10	1.435 (6)	C27'—H27D	0.97
C8—S1—C5	91.7 (2)	O1—C12—C10	128.2 (4)
C20—O2—C21	115.8 (3)	N2—C12—C10	113.9 (4)
C9—N1—C8	116.9 (4)	N3—C13—C10	123.2 (4)
C20—N2—C12	120.7 (4)	N3—C13—C7	117.5 (4)
C20—N2—C14	120.9 (3)	C10—C13—C7	119.3 (4)
C12—N2—C14	118.4 (4)	C19—C14—C15	120.8 (4)
C20—N3—C13	116.1 (3)	C19—C14—N2	119.3 (4)
C6—C1—C2	111.6 (4)	C15—C14—N2	119.8 (4)
C6—C1—H1A	109.3	C14—C15—C16	119.9 (5)
C2—C1—H1A	109.3	C14—C15—H15	120.0
C6—C1—H1B	109.3	C16—C15—H15	120.0
C2—C1—H1B	109.3	C17—C16—C15	118.8 (5)
H1A—C1—H1B	108.0	C17—C16—H16	120.6
C3—C2—C1	114.9 (4)	C15—C16—H16	120.6
C3—C2—H2A	108.6	C18—C17—C16	121.7 (5)
C1—C2—H2A	108.5	C18—C17—Cl1	119.8 (4)
C3—C2—H2B	108.5	C16—C17—Cl1	118.4 (5)
C1—C2—H2B	108.5	C17—C18—C19	119.9 (5)
H2A—C2—H2B	107.5	C17—C18—H18	120.0
C2—C3—C4	112.8 (5)	C19—C18—H18	120.0
C2—C3—H3A	109.0	C14—C19—C18	118.8 (5)
C4—C3—H3A	109.0	C14—C19—H19	120.6
C2—C3—H3B	109.0	C18—C19—H19	120.6
C4—C3—H3B	109.0	N3—C20—O2	120.4 (4)
H3A—C3—H3B	107.8	N3—C20—N2	126.9 (4)
C5—C4—C3	110.5 (4)	O2—C20—N2	112.6 (4)

C5—C4—H4A	109.6	C22—C21—C26	120.6 (4)
C3—C4—H4A	109.6	C22—C21—O2	120.1 (4)
C5—C4—H4B	109.6	C26—C21—O2	119.2 (4)
C3—C4—H4B	109.6	C21—C22—C23	119.1 (5)
H4A—C4—H4B	108.1	C21—C22—H22	120.4
C6—C5—C4	125.4 (4)	C23—C22—H22	120.4
C6—C5—S1	113.2 (4)	C24—C23—C22	119.9 (5)
C4—C5—S1	121.4 (3)	C24—C23—H23	120.0
C5—C6—C7	111.1 (4)	C22—C23—H23	120.0
C5—C6—C1	121.1 (4)	C25—C24—C23	120.8 (5)
C7—C6—C1	127.7 (4)	C25—C24—Cl2	119.9 (4)
C8—C7—C13	115.4 (4)	C23—C24—Cl2	119.3 (4)
C8—C7—C6	113.2 (4)	C24—C25—C26	119.8 (5)
C13—C7—C6	131.4 (4)	C24—C25—H25	120.1
N1—C8—C7	127.2 (4)	C26—C25—H25	120.1
N1—C8—S1	122.1 (3)	C21—C26—C25	119.7 (4)
C7—C8—S1	110.7 (4)	C21—C26—Cl3	119.2 (4)
N1—C9—C10	121.8 (4)	C25—C26—Cl3	121.0 (4)
N1—C9—C11	115.3 (4)	Cl5—C27—Cl4	105.1 (9)
C10—C9—C11	122.9 (5)	Cl5—C27—H27A	110.7
C13—C10—C9	119.3 (4)	Cl4—C27—H27A	110.7
C13—C10—C12	119.1 (4)	Cl5—C27—H27B	110.7
C9—C10—C12	121.6 (4)	Cl4—C27—H27B	110.7
C9—C11—H11A	109.5	H27A—C27—H27B	108.8
C9—C11—H11B	109.5	Cl4'—C27'—Cl5'	100.7 (10)
H11A—C11—H11B	109.5	Cl4'—C27'—H27C	111.6
C9—C11—H11C	109.5	Cl5'—C27'—H27C	111.6
H11A—C11—H11C	109.5	Cl4'—C27'—H27D	111.6
H11B—C11—H11C	109.5	Cl5'—C27'—H27D	111.6
O1—C12—N2	117.9 (4)	H27C—C27'—H27D	109.4
C6—C1—C2—C3	−43.0 (6)	C9—C10—C13—C7	1.3 (6)
C1—C2—C3—C4	58.8 (7)	C12—C10—C13—C7	−178.4 (4)
C2—C3—C4—C5	−40.6 (6)	C8—C7—C13—N3	176.2 (3)
C3—C4—C5—C6	11.5 (7)	C6—C7—C13—N3	−3.0 (6)
C3—C4—C5—S1	−169.1 (4)	C8—C7—C13—C10	−2.5 (5)
C8—S1—C5—C6	0.8 (3)	C6—C7—C13—C10	178.3 (4)
C8—S1—C5—C4	−178.7 (4)	C20—N2—C14—C19	−103.7 (5)
C4—C5—C6—C7	179.5 (4)	C12—N2—C14—C19	75.4 (5)
S1—C5—C6—C7	0.1 (4)	C20—N2—C14—C15	74.8 (5)
C4—C5—C6—C1	2.1 (7)	C12—N2—C14—C15	−106.1 (5)
S1—C5—C6—C1	−177.4 (3)	C19—C14—C15—C16	0.2 (7)
C2—C1—C6—C5	12.5 (6)	N2—C14—C15—C16	−178.2 (4)
C2—C1—C6—C7	−164.5 (4)	C14—C15—C16—C17	0.7 (7)
C5—C6—C7—C8	−1.2 (5)	C15—C16—C17—C18	−1.0 (8)
C1—C6—C7—C8	176.1 (4)	C15—C16—C17—Cl1	176.4 (4)
C5—C6—C7—C13	178.1 (4)	C16—C17—C18—C19	0.4 (9)
C1—C6—C7—C13	−4.7 (7)	Cl1—C17—C18—C19	−177.0 (4)
C9—N1—C8—C7	−0.7 (7)	C15—C14—C19—C18	−0.8 (7)
C9—N1—C8—S1	179.3 (3)	N2—C14—C19—C18	177.6 (4)

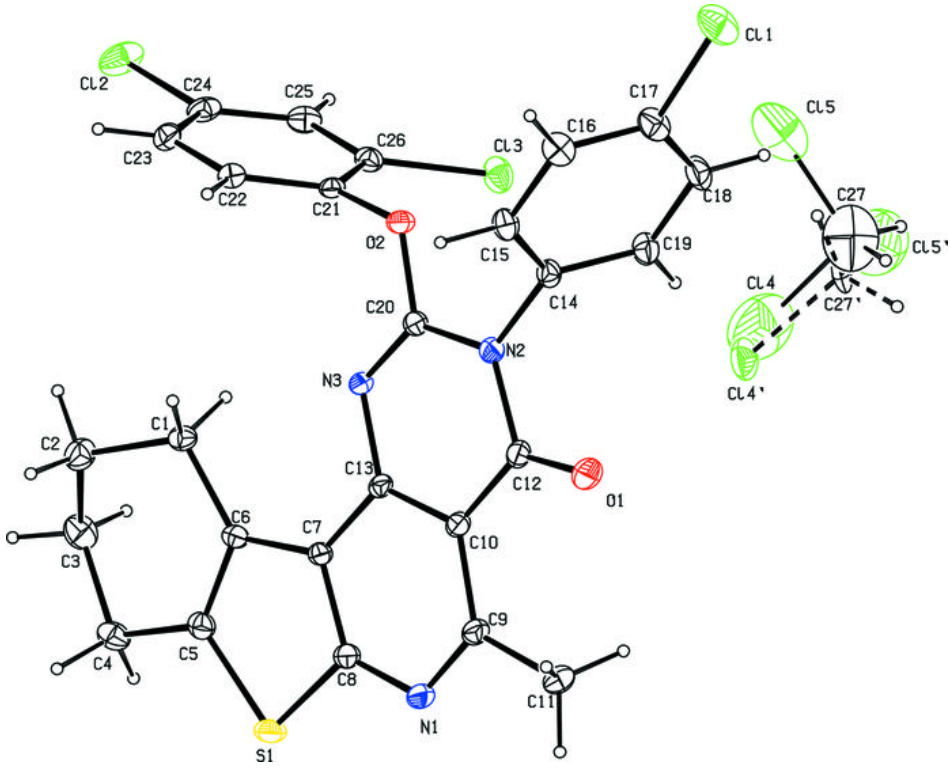
C13—C7—C8—N1	2.3 (6)	C17—C18—C19—C14	0.5 (8)
C6—C7—C8—N1	−178.3 (4)	C13—N3—C20—O2	−178.3 (3)
C13—C7—C8—S1	−177.6 (3)	C13—N3—C20—N2	1.9 (6)
C6—C7—C8—S1	1.7 (4)	C21—O2—C20—N3	−7.7 (5)
C5—S1—C8—N1	178.6 (4)	C21—O2—C20—N2	172.2 (3)
C5—S1—C8—C7	−1.4 (3)	C12—N2—C20—N3	−0.7 (6)
C8—N1—C9—C10	−0.7 (7)	C14—N2—C20—N3	178.3 (4)
C8—N1—C9—C11	−178.9 (4)	C12—N2—C20—O2	179.5 (3)
N1—C9—C10—C13	0.4 (7)	C14—N2—C20—O2	−1.5 (5)
C11—C9—C10—C13	178.4 (5)	C20—O2—C21—C22	92.4 (4)
N1—C9—C10—C12	−179.9 (4)	C20—O2—C21—C26	−92.6 (4)
C11—C9—C10—C12	−1.8 (7)	C26—C21—C22—C23	1.4 (6)
C20—N2—C12—O1	179.8 (4)	O2—C21—C22—C23	176.4 (4)
C14—N2—C12—O1	0.7 (6)	C21—C22—C23—C24	−0.9 (7)
C20—N2—C12—C10	0.5 (6)	C22—C23—C24—C25	−0.5 (7)
C14—N2—C12—C10	−178.6 (4)	C22—C23—C24—Cl2	−179.2 (3)
C13—C10—C12—O1	179.3 (5)	C23—C24—C25—C26	1.5 (7)
C9—C10—C12—O1	−0.4 (8)	Cl2—C24—C25—C26	−179.9 (3)
C13—C10—C12—N2	−1.6 (6)	C22—C21—C26—C25	−0.5 (6)
C9—C10—C12—N2	178.7 (4)	O2—C21—C26—C25	−175.6 (4)
C20—N3—C13—C10	−3.0 (6)	C22—C21—C26—Cl3	179.4 (3)
C20—N3—C13—C7	178.3 (3)	O2—C21—C26—Cl3	4.4 (5)
C9—C10—C13—N3	−177.3 (4)	C24—C25—C26—C21	−0.9 (7)
C12—C10—C13—N3	2.9 (6)	C24—C25—C26—Cl3	179.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C27—H27A···O1^{i}	0.97	2.44	3.23 (2)	138

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*.

Fig. 1


Cl1
Cl2
C24
C25
C17
C16
Cl5
C26
Cl3
C23
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C27
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C19
Cl5'
Cl4
C14
C27'
C20
N2
N3
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C1
C2
C13
C12
O1
C6
C7
C10
C3
C9
C4
C5
C8
C11
N1
S1