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2-(4-Chloro-3-methylphenoxy)-3-(4-chlorophenyl)-5-methyl-8,9,10,11-tetrahydro-1-benzothieno-[2′,3′:2,3]pyrido[4,3-*d*]pyrimidin-4(3*H*)-one dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.087
wR factor = 0.211
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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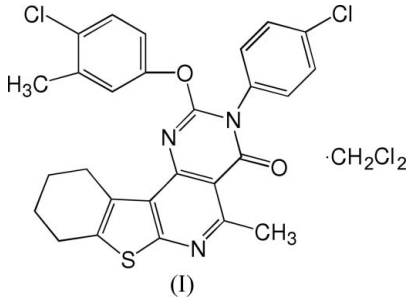
In the structure of the title compound, $C_{27}H_{21}Cl_2N_3O_2S\cdot CH_2Cl_2$, the C—S bond lengths in the thiophene ring [1.744 (5) and 1.745 (5) Å] are equivalent and long compared with the values observed in both free thiophene, measured using electron diffraction, and thieno[2,3-*c*]pyridine. The central thienopyridine ring system is nearly planar and the dihedral angle between the thiophene and pyridine planes is 0.9 (1)°.

Comment

Many pyrido[4,3-*d*]pyrimidines have pharmaceutical activity and germicidal action (Anderson & Broom, 1977). 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 requires a long reaction time. Recently, we have developed a new and facile regioselective annulation process, which proceeds smoothly under mild conditions *via* a tandem aza-Wittig and cyclization reaction, to synthesize novel pyrido[4,3-*d*]pyrimidine derivatives (Zhou *et al.*, 2005). In this paper, the crystal structure of the title compound, (I), is reported. The structure of (I) was also characterized by ^{1}H NMR, MS and elemental analyses.



(I)

The molecular structure of (I) is shown in Fig. 1. The C—S bond lengths in the thiophene ring [1.744 (5) and 1.745 (5) Å] are equivalent and long compared with the values observed in both free thiophene (1.714 Å; Bonham & Momany, 1963). The C11—N1—C7 angle of 116.6 (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 0.9 (1)°.

Experimental

The title compound was prepared according to the literature procedure of Zhou *et al.* (2005). Suitable crystals of (I) were obtained by

evaporation of a dichoromethane solution (m.p. 533–534 K). Analysis, calculated for $C_{28}H_{23}Cl_4N_3O_2S$: C 55.37, H 3.82, N 6.92%; found: C 55.26, H 3.93, N 7.15%. Spectroscopis analysis: IR (KBr, ν, cm^{-1}): 3124 (Ph—H), 2936, 2859 (C—H), 1701 (C═O), 1616, 1562, 1517, 1489, 1161, 1051, 748; ^{1}H NMR ($CDCl_3$, TMS, 400 MHz, δ, p.p.m.): 1.65–1.81 (*m*, 2H, $2CH_2$), 2.47 (*s*, 3H, CH_3), 2.48–2.82 (*m*, 4H, $2CH_2$), 3.05 (*s*, 3H, CH_3), 6.91–7.58 (*m*, 7H, Ar—H). MS (EI, %): 524 (M^+ + 2 62), 523 (M^+ + 1 49), 522 (M^+ 100), 506 (19), 493 (14), 396 (17), 380 (28).

Crystal data

$C_{27}H_{21}Cl_2N_3O_2S\cdot CH_2Cl_2$
M_r = 607.35
Orthorhombic, *Pbca*
a = 18.564 (3) Å
b = 10.6834 (17) Å
c = 28.510 (4) Å
V = 5654.3 (15) Å^3
Z = 8
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3753 reflections
θ = 2.3–19.8°
μ = 0.52 mm^{-1}
T = 293 (2) K
Plate, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
26881 measured reflections
4964 independent reflections
3655 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°
$h = -17 \rightarrow 22$
$k = -12 \rightarrow 12$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.087
$wR(F^2)$ = 0.211
S = 1.10
4964 reflections
346 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 7.6281P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C6—S1	1.744 (5)	C14—N2	1.275 (5)
C7—N1	1.338 (6)	C14—N3	1.370 (5)
C7—S1	1.745 (5)	C15—N3	1.444 (5)
C9—N2	1.382 (5)	C16—C17	1.377 (7)
C11—N1	1.324 (6)	C17—C18	1.376 (7)
C13—N3	1.419 (5)		
N2—C14—N3	126.5 (4)	C14—N3—C15	121.2 (3)
O2—C14—N3	112.0 (3)	C13—N3—C15	118.5 (3)
C11—N1—C7	116.6 (4)	C14—O2—C21	116.5 (3)
C14—N2—C9	116.6 (3)	C6—S1—C7	91.2 (2)
C14—N3—C13	120.2 (3)		

H atoms were refined with fixed geometry, with C—H distances in the range 0.93–0.97 Å, riding on their carrier atoms, with U_{iso}(H) set to 1.2 (1.5 for the methyl H atoms) times U_{eq} of the parent atom.

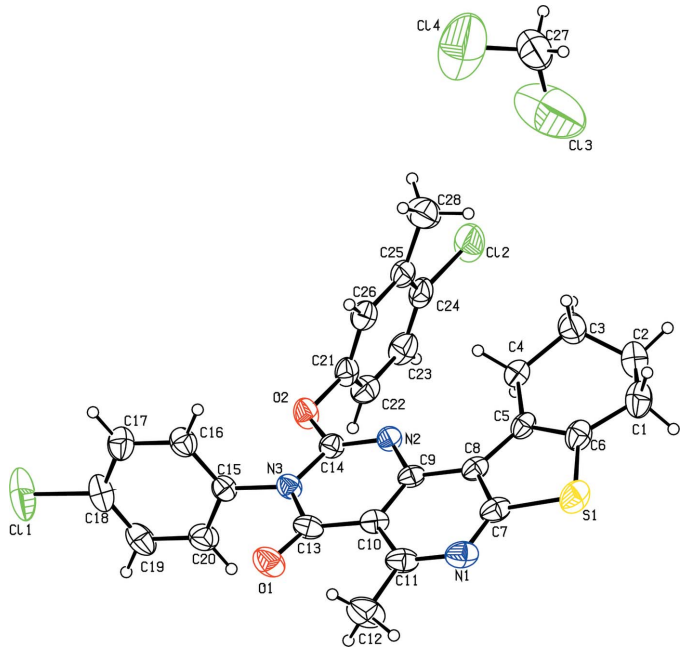


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

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*.

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