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1-{6-(4-Chlorophenyl)-1-[(6-chloropyridin-3-yl)-methyl]-2-[(6-chloropyridin-3-yl)methylsulfanyl]-4-methyl-1,6-dihydropyrimidin-5-yl}ethanone

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Key indicators

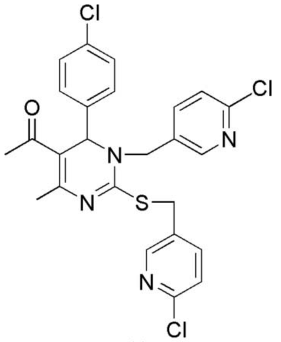
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.133
Data-to-parameter ratio = 17.0

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

In the title compound, $C_{25}H_{21}Cl_3N_4OS$, molecules are linked into centrosymmetric pairs by C—H···O interactions. C—H···Cl interactions link these pairs into a three-dimensional network.

Comment

Biginelli dihydropyrimidines and their derivatives have attracted interest owing to their wide range of therapeutical and pharmacological properties, such as antiviral, antitumor, antibacterial and anti-inflammatory properties (Kappe, 1993).



(I)

The title compound, (I) (Fig. 1), was synthesized by introducing chloropyridine rings into a Biginelli dihydropyrimidine molecular framework. In the crystal structure, intermolecular C5—H5···O1^{i} and C25—H25···O1^{i} [symmetry code: (i) $-x$, $-y$, $-z$] hydrogen bonds link molecules into centrosymmetric pairs (Fig. 2 and Table 1). These pairs are linked into a three-dimensional network by C17—H17···Cl3^{ii} interactions [symmetry code: (ii) $-x + 1$, $-y$, $-z$] (Fig. 2 and Table 1).

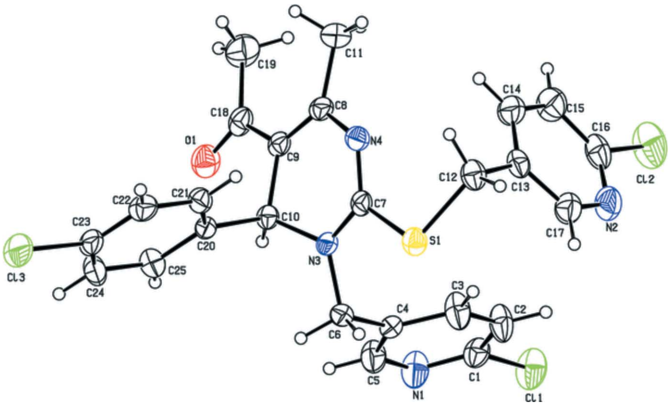


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level for non-H atoms.

Experimental

A solution of 1-[4-(4-chlorophenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydropyrimidin-5-yl]ethanone (2 mmol), 2-chloro-5-chloromethylpyridine (4 mmol) and K_2CO_3 (4 mmol) in anhydrous dimethylformamide (10 ml) was stirred vigorously at room temperature until the reaction was complete (as monitored by thin-layer chromatography). The solid residue was filtered off and the filtrate was concentrated under vacuum then purified by column chromatography on silica gel using (2:1 *v*/*v*) petroleum ether/ethyl acetate as the eluant. Evaporation of the solvent gave a green solid (yield 90%, m.p. 407–408 K). Light-yellow crystals used for X-ray analysis were grown from dichloromethane/hexane (1:1 *v*/*v*).

Crystal data

$C_{25}H_{21}Cl_3N_4OS$
$M_r = 531.87$
Triclinic, $P\overline{1}$
$a = 10.2887$ (11) Å
$b = 11.1487$ (12) Å
$c = 12.6386$ (13) Å
$\alpha = 104.740$ (2)°
$\beta = 95.725$ (2)°
$\gamma = 116.332$ (2)°
$V = 1218.1$ (2) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.49$ mm^{-1}
$T = 298$ (2) K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.909$, $T_{\max} = 0.953$
12693 measured reflections
5262 independent reflections
3900 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.052$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.133$
$S = 1.03$
5262 reflections
309 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.38$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1$^{\rm i}$	0.93	2.42	3.326 (3)	165
C25—H25···O1$^{\rm i}$	0.93	2.55	3.348 (3)	144
C17—H17···Cl3$^{\rm ii}$	0.93	2.80	3.564 (3)	140

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

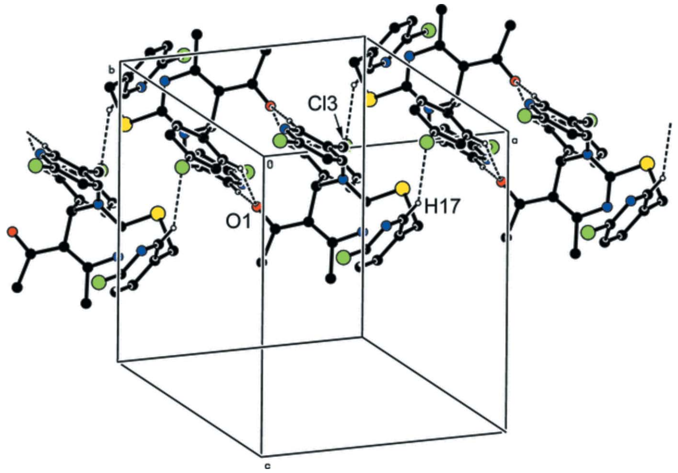


Figure 2
Part of the crystal packing of (I), showing molecules linked into pairs by C—H···O interactions (dashed lines). The pairs are linked further by C—H···Cl interactions (dashed lines). H atoms not involved in these interactions have been omitted.

H atoms were placed in calculated positions with C—H = 0.93–0.98 Å and refined using a riding-model with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C), or $1.5U_{\rm eq}$(C) for the methyl groups. The methyl groups were allowed to rotate about their local threefold axes.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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