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6-(4-Fluorophenyl)-5-morpholino-3-phenyl-2-thioxo-2,3-dihydrothiazolo[4,5-*d*]pyrimidin-7(6*H*)-one

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.113
Data-to-parameter ratio = 18.0

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

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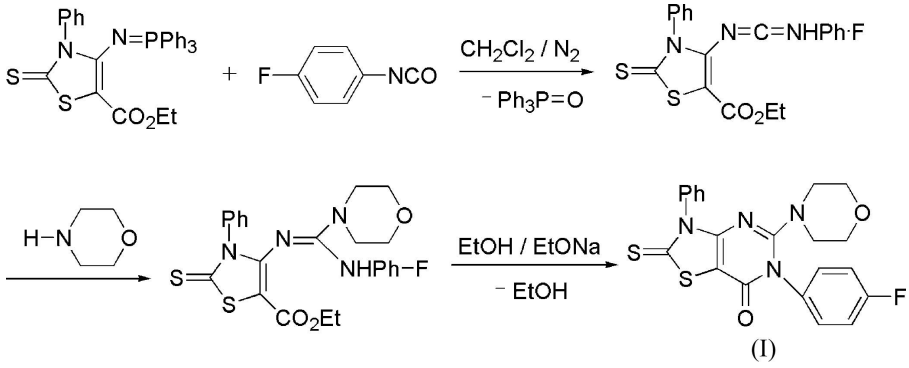
In the structure of the title compound, $C_{21}H_{17}FN_4O_2S_2$, the fused thiazolopyrimidine ring system is nearly planar. The morpholine ring adopts a chair conformation. The crystal structure is stabilized by C—H···O hydrogen bonds and weak π–π stacking interactions.

Comment

Thiazolopyrimidines are widely recognized as pharmaceutically and biologically useful systems due to their structural similarities to purine bases. As such, thiazolopyrimidines have been found to exhibit anti-HIV, anticancer (Habib *et al.*, 1996), anti-inflammatory (Tozkoparan *et al.*, 1999) and antimicrobial activities (Bekhit *et al.*, 2003). An important synthetic route for thiazolo[4,5-*d*]pyrimidines in previous reports is the condensation reaction of 4-aminothiazole-5-carboxylate and an isothiocyanate (Balkan *et al.*, 2002). However, this method often requires a long reaction time. Recently, we have developed a new and versatile annulation process, which proceeds smoothly under mild conditions *via* a tandem aza-Wittig and cyclization reaction, to synthesize novel thiazolo[4,5-*d*]-pyrimidine derivatives. In this paper, we report the structure of the title compound, (I) (Fig. 1).



In the molecule of (I), the thiazolopyrimidine ring system is nearly planar. The planes of both the benzene rings are twisted from that of the central thiazolopyrimidine ring, the dihedral angles being 72.07 (3) (phenyl) and 62.96 (5)° (*p*-fluorophenyl). The terminal morpholine ring adopts a chair conformation. The crystal structure is stabilized by C—H···O hydrogen bonds (Table 1). In addition, short intermolecular distances between the centroids of the C7–C9/N1/S2 ring (*Cg*1) and the C8–C11/N2–N3 ring (*Cg*3), of an adjacent molecule indicate the existence of π–π stacking interactions (Janiak, 2000) [$Cg1\cdots Cg3^{i}$ = 3.8328 (10) Å; symmetry code: (i) 1 − *x*, −*y*, 1 − *z*].

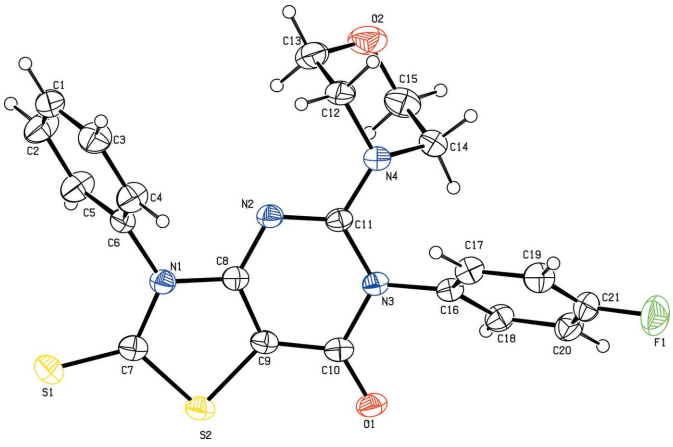


Figure 1
The molecular structure of (I), showing the labelling scheme and 50% probability displacement ellipsoids.

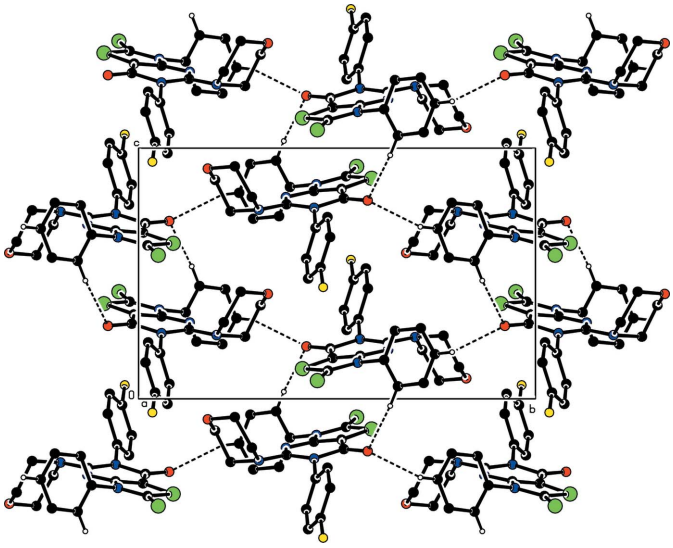


Figure 2
The crystal structure of (I), showing the formation of C—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

Experimental

4-Amino-2,3-dihydro-3-phenyl-2-thioxothiazole-5-carboxylate, (II), was prepared according to a literature procedure in 68.5% yield (Sharaf *et al.*, 1996). The iminophosphorane of (II) was synthesized according to a literature report in 92.6% yield (Wamhoff *et al.*, 1993). To a solution of the iminophosphorane of (II) (1 mmol) in dry CH_2Cl_2 (15 ml) was added 4-fluorophenyl isocyanate (1.1 mmol) under a nitrogen atmosphere at room tempreture (Ding *et al.*, 1999). After reaction, the mixture was allowed to stand for 5–12 h, the solvent was removed under reduced pressure, then diethyl ether (10 ml) and petroleum ether (10 ml) were added to precipitate the side product, triphenylphosphine oxide, which was then removed by filtration. Subsequent removal of the solvent gave the corresponding carbodiimide, which was used directly without further purification. To a solution of the carbodiimide in ethanol (15 ml) was added morpholine (1.1 mmol) and a catalytic amount of sodium ethoxide in ethanol (Wang *et al.*, 2004). After the mixture had been stirred for 4 h at 303 K, the solution was concentrated and the residue was recrystallized from CH_3CN to give colorless blocks of the title compound, (I), after one week.

Crystal data

$C_{21}H_{17}FN_4O_2S_2$
M_r = 440.51
Monoclinic, $P2_1/n$
a = 11.1915 (15) Å
b = 16.982 (2) Å
c = 11.4220 (14) Å
β = 110.077 (2)°
V = 2038.9 (5) Å^3
Z = 4
D_x = 1.435 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.916, T_{max} = 0.943
23710 measured reflections
4869 independent reflections
3524 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.113
S = 0.97
4869 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1^{i}	0.93	2.57	3.399 (2)	149
C5—H5···O1^{ii}	0.93	2.48	3.384 (2)	163

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+1, -y+2, -z+1$.

All H atoms were refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic, C—H = 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH, and C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Bruker, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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