

5-Methyl-2-morpholino-3-*p*-tolyl-8,9,10,11-tetrahydro-2-benzothieno[2',3':6,5]pyrido[4,3-*d*]-pyrimidin-4(3*H*)-one

He-Lian Chen and Hong-Wu He*

Key Laboratory of Pesticides and Chemical Biology, College of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China

Correspondence e-mail:
he1208@mail.ccnu.edu.cn

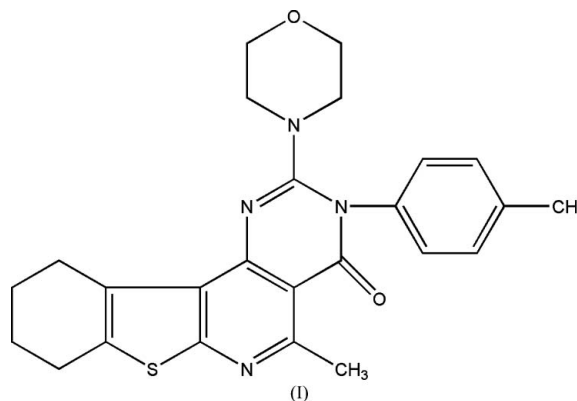
Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.050
 wR factor = 0.123
Data-to-parameter ratio = 16.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, $\text{C}_{25}\text{H}_{26}\text{N}_4\text{O}_2\text{S}$, the central tricyclic system is essentially planar. The crystal stacking is governed mainly due to π - π interactions.

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Comment

Among many known heterocyclic compounds, derivatives containing a pyridine system have received much attention since they have been demonstrated to possess significant biological activity (Augusto *et al.*, 1995). The title compound, (I), belongs to this family of heterocyclic compounds and we present its crystal structure here.

Selected bond lengths and angles are listed in Table 1. In (I) (Fig. 1), the C—S bonds are longer than those observed in free thiophene [1.714 (3) Å; Bonham & Momany, 1963] and thieno[2,3-*c*]pyridine [1.728 (1) and 1.731 (1) Å; Nerenz *et al.*, 1997]. The C8—S1—C angle in (I) is slightly narrower than that observed in free thiophene [92.2 (2)°]. C3 and C4 of the cyclohexene ring are each disordered over two positions.

The short intermolecular distances between the centroids of the pyridine ($Cg3$) and pyrimidine ($Cg4$) rings [$Cg3 \cdots Cg4^i = 3.7799$ (11) Å; symmetry code: (i) $1 - x, y, 1 - z$] indicate the existence of π - π stacking interactions (Janiak, 2000), which stabilize the crystal packing (Fig. 2).

Experimental

4-Amino-3-ethoxycarbonyl-2-methyl-5,6,7,8-(4*H*)-tetrahydro-2-benzothieno[2,3-*b*]pyridine, (II), was prepared according to a literature procedure (Augusto *et al.*, 1995) in 90% yield. The iminophosphorane of (II) was obtained in 82% yield according to a literature synthetic method (Wamhoff *et al.*, 1993). To a solution of iminophosphorane of (II) (1 mmol) in dry CH_2Cl_2 (20 ml) and a catalytic amount of K_2CO_3 (0.05 mmol) was added 4-methylphenyl isocyanate (1.1 mmol) (Ding *et al.*, 1999). After the reaction mixture had been left to stand for 6 h, the solvent was removed under

reduced pressure and Et₂O/petroleum ether (2:1 v/v) was 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) were added morphine (1.1 mmol) and a catalytic amount of sodium ethoxide in ethanol (Wang *et al.*, 2004). After the mixture had been stirred for 12 h at 300 K, the solution was concentrated and the residue was recrystallized from CH₃CN, giving colorless prismatic block-shaped crystals of the title compound after one week.

Crystal data

C ₂₅ H ₂₆ N ₄ O ₂ S	Z = 2
<i>M_r</i> = 446.56	<i>D_x</i> = 1.353 Mg m ⁻³
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 9.5216 (9) Å	Cell parameters from 3408 reflections
<i>b</i> = 11.0760 (10) Å	θ = 2.5–28.2°
<i>c</i> = 12.5294 (11) Å	μ = 0.18 mm ⁻¹
α = 105.036 (2)°	<i>T</i> = 292 (2) K
β = 101.193 (2)°	Block, colorless
γ = 114.050 (2)°	0.30 × 0.20 × 0.20 mm
<i>V</i> = 1095.93 (17) Å ³	

Data collection

Bruker SMART CCD area-detector diffractometer	3570 reflections with <i>I</i> > 2σ(<i>I</i>)
φ and ω scans	<i>R</i> _{int} = 0.069
Absorption correction: none	θ _{max} = 27.5°
12852 measured reflections	<i>h</i> = -12 → 12
4978 independent reflections	<i>k</i> = -14 → 14
	<i>l</i> = -16 → 16

Refinement

Refinement on <i>F</i> ²	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.050$	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2]$
$wR(F^2) = 0.123$	where $P = (F_o^2 + 2F_c^2)/3$
<i>S</i> = 0.95	(Δ/σ) _{max} < 0.001
4978 reflections	$\Delta\rho_{max} = 0.24 \text{ e \AA}^{-3}$
310 parameters	$\Delta\rho_{min} = -0.29 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C1–S1	1.7444 (17)	C8–S1	1.7302 (17)
C1–C2–C3–C4	-42.7 (3)	C18–C17–N4–C14	151.08 (15)
C1–C2–C3'–C4'	47.1 (14)	C17–C18–O2–C16	-58.5 (2)
C6–C7–C8–N1	-179.08 (16)	C15–C16–O2–C18	57.3 (2)
C9–C11–C12–N2	179.64 (15)		

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.93–0.97 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(methyl C). Two of the C atoms in the six-membered ring are disordered over two positions, and the occupancy factors for the disordered positions C3/C3' and C4/C4' were refined to 0.80 (1):0.19 (1). Similarity restraints were applied to distances involving disordered atoms.

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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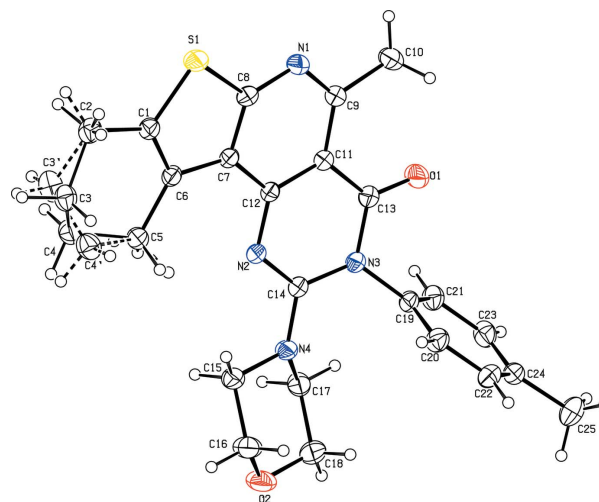


Figure 1
View of (I), showing the labelling scheme and 50% probability displacement ellipsoids. Both disorder components are shown.

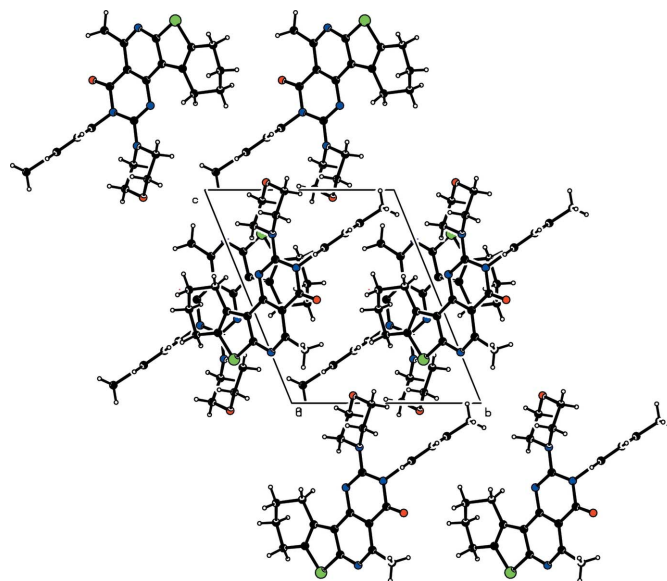


Figure 2
The packing of (I). Only one disorder component is shown.

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