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Key indicators

Single-crystal X-ray study
T = 292 K
Mean $\sigma(C-C)$ = 0.005 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.155
Data-to-parameter ratio = 16.0

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

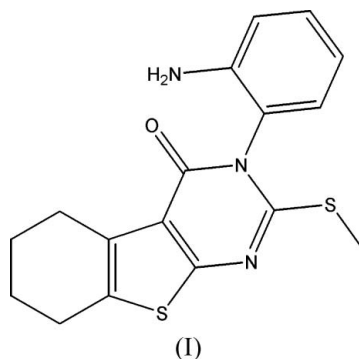
3-(2-Aminophenyl)-2-methylsulfanyl-5,6,7,8-tetrahydrobenzothieno[2,3-d]pyrimidin-4(3H)-one

In the title compound, $C_{17}H_{17}N_3OS_2$, the fused thiophene and pyrimidine rings are almost coplanar. The amino group is involved in both intramolecular and intermolecular hydrogen bonds, the latter linking the molecules into a centrosymmetric dimer.

Received 31 October 2005
Accepted 10 November 2005
Online 19 November 2005

Comment

Pyrimidine derivatives are increasingly attracting the attention of the synthetic community because of the important role played by such systems in many natural products, antibiotics and drugs (Ding *et al.*, 2004). In this paper, we present the X-ray crystallographic analysis of the title compound, (I).



The molecular structure indicates that the thieno[2,3-*d*]pyrimidine group is a conjugated system (Fig. 1). The tetrahydrobenzene ring is disordered. There are two possible conformations, C1–C6 and C1/C2'/C3'/C4–C6, with an occupancy ratio of 0.554 (8):0.446 (8). The amino group of the

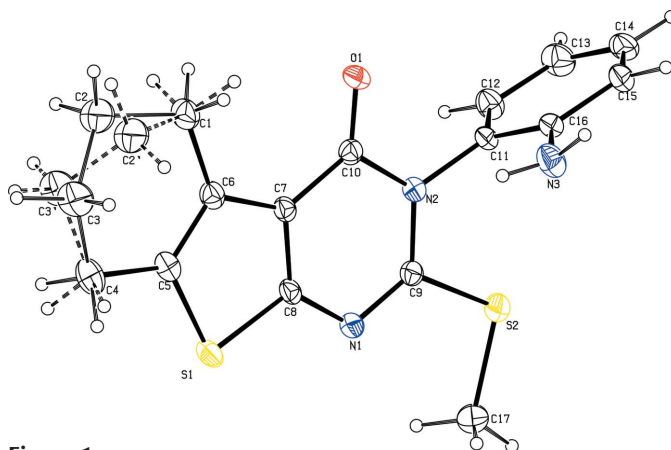


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at 50% probability level. Both disorder components are shown.

molecule forms weak hydrogen bonds with atoms N2 and O1 (Table 2 and Fig. 2). The former is an intramolecular hydrogen bond, which completes a closed five-membered loop. The latter is an intermolecular interaction with a neighbouring molecule related by a centre of inversion. The intermolecular interactions thus link the molecules into a hydrogen-bonded dimer.

Experimental

A mixture of 3-(2-aminophenyl)-2-thioxo-2,3,5,6,7,8-tetrahydrobenzothieno[2,3-*d*]pyrimidin-4(1*H*)-one (1 mmol), methyl iodide (1.3 mmol) and solid potassium carbonate (2 mmol) in CH₃CN (10 ml) was stirred for 1 h at room temperature and filtered. The filtrate was concentrated and the precipitate was recrystallized from methylene chloride/petroleum ether (1:3 *v/v*) to give compound (I) in 80% yield (m.p. 507 K). ¹H NMR (CDCl₃, 400 MHz): δ 7.33–6.89 (*m*, 4H, Ar–H), 3.34 (*br*, 2H, NH₂), 2.96–2.75 (*m*, 4H, 2CH₂), 2.48 (*s*, 3H, SCH₃), 1.91–1.80 (*m*, 4H, 2CH₂); MS (*m/z*, %), 343 (*M*⁺, 100), 326 (18), 295 (65), 267 (27), 254 (7), 117 (6). Analysis calculated for C₁₇H₁₇N₃OS₂: C 59.45, H 4.99, N 12.23%; found: C 59.68, H 4.92, N 12.35%. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from a mixed solvent of hexane and dichloromethane (1:3 *v/v*) at room temperature.

Crystal data

C ₁₇ H ₁₇ N ₃ OS ₂	<i>D</i> _x = 1.388 Mg m ⁻³
<i>M</i> _r = 343.46	Mo Kα radiation
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Cell parameters from 1263 reflections
<i>a</i> = 11.7686 (10) Å	<i>θ</i> = 2.4–19.6°
<i>b</i> = 13.4861 (11) Å	<i>μ</i> = 0.33 mm ⁻¹
<i>c</i> = 11.2137 (10) Å	<i>T</i> = 292 (2) K
<i>β</i> = 112.552 (2)°	Block, colourless
<i>V</i> = 1643.7 (2) Å ³	0.30 × 0.20 × 0.10 mm
<i>Z</i> = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2125 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>φ</i> and <i>ω</i> scans	<i>R</i> _{int} = 0.054
Absorption correction: none	<i>θ</i> _{max} = 27.0°
9565 measured reflections	<i>h</i> = -14 → 15
3575 independent reflections	<i>k</i> = -14 → 17
	<i>l</i> = -12 → 14

Refinement

Refinement on <i>F</i> ²	H atoms treated by a mixture of independent and constrained refinement
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.061	<i>w</i> = 1/[σ ² (<i>F</i> _o ²) + (0.0702 <i>P</i>) ²]
<i>wR</i> (<i>F</i> ²) = 0.155	where <i>P</i> = (<i>F</i> _o ² + 2 <i>F</i> _c ²)/3
<i>S</i> = 0.99	(Δ/σ) _{max} = 0.002
3575 reflections	Δρ _{max} = 0.33 e Å ⁻³
224 parameters	Δρ _{min} = -0.22 e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

C5–C6	1.342 (4)	C9–N2	1.381 (3)
C5–S1	1.747 (3)	C9–S2	1.748 (3)
C6–C7	1.442 (4)	C10–O1	1.228 (3)
C7–C8	1.376 (4)	C10–N2	1.414 (4)
C7–C10	1.431 (4)	C11–N2	1.455 (3)
C8–N1	1.364 (4)	C16–N3	1.381 (4)
C8–S1	1.727 (3)	C17–S2	1.801 (3)
C9–N1	1.298 (4)		
C8–S1–C5	90.46 (14)	C9–S2–C17	99.61 (16)

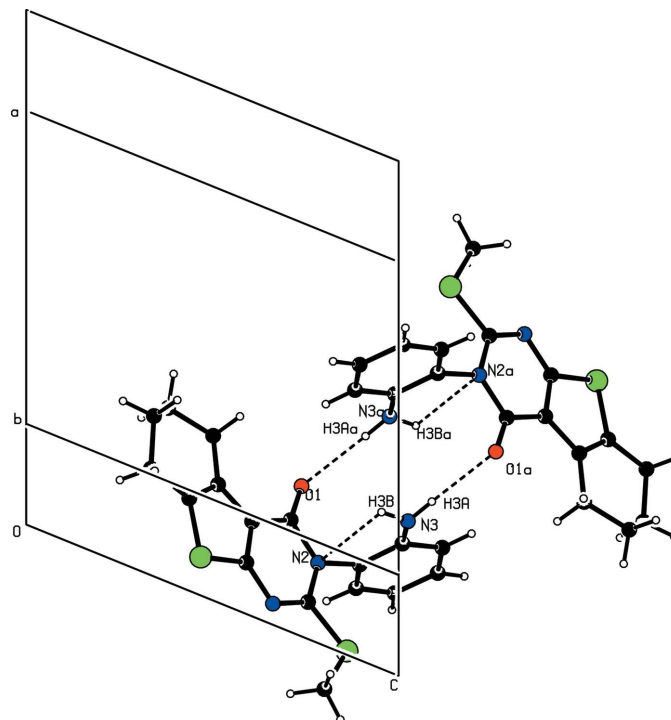


Figure 2

A partial packing diagram, showing a hydrogen-bonded (dashed lines) dimer. The suffix a corresponds to symmetry code *i* in Table 2.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N3–H3B···N2	0.86 (1)	2.51 (4)	2.845 (4)	104 (3)
N3–H3A···O1 ⁱ	0.86 (1)	2.29 (2)	3.139 (3)	170 (2)

Symmetry code: (i) $-x + 1, -y, -z + 2$.

H atoms attached to atom N3 were located in a difference Fourier map and refined with a restraint of N–H = 0.86 (1) Å. The displacement parameter *U*_{iso}(H) was refined freely. Other H atoms were placed at calculated positions and treated as riding atoms, with C–H = 0.93–0.98 Å, and *U*_{iso}(H) = 1.2*U*_{eq}(C), or 1.5*U*_{eq}(C) for CH₃.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge financial support of this work by the National Basic Research Programme of China (2003CB114400) and the National Natural Science Foundation of China (20372023 and 20102001).

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