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2-[(1,3-Benzodioxol-5-yl)methylene]-6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-one

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The title compound, $C_{14}H_{12}N_2O_3S$, was synthesized by mixing 1,3-benzodioxole-5-carbaldehyde, ethyl chloroacetate and tetrahydropyrimidine-2-thione in ethanol. The nearly planar thiazoline ring is fused to a dihydropyrimidine ring, which has a sofa conformation. The molecules are connected through weak C—H···N and C—H···O interactions.

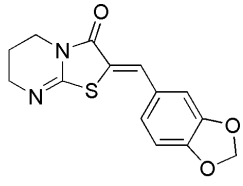
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.114
Data-to-parameter ratio = 14.9

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

Comment

In this paper, the structure of the title compound, (I), is reported. The molecular structure of (I) is illustrated in Fig. 1. The molecule consists of a dihydropyrimidine ring, a thiazoline ring, a benzene ring and a dioxolane ring. The entire molecule, except for C3 and the H atoms, is planar to within 0.031 (3) Å, with C3 lying 0.656 (2) Å out of the plane, such that the dihydropyrimidine ring has a sofa conformation. Molecules are connected by weak C—H···N and C—H···O interactions (Fig. 2). Molecules are connected by weak C—H···N and C—H···O interactions (Fig. 2), with C···O = 3.412 (2) Å and C—H···O = 155°, and C···N = 3.401 (3) Å and C—H···N = 171°.



(I)

Experimental

A mixture of tetrahydropyrimidine-2-thione (0.02 mol), ethyl chloroacetate (0.02 mol) and pyridine (0.02 mol) was stirred under reflux in ethanol (40 ml) for 4 h. Then 1,3-benzodioxole-5-carbaldehyde (0.02 mol) and piperidine (0.02 mol) was added and the mixture was refluxed for 4 h. After cooling and filtration, the title compound was recrystallized from acetic acid. ^{1}H NMR (δ): 1.15 (m, 1 H, –CH_2), 1.65 (m, 1H, —CH_2), 3.25 (m, 2H, —CH_2), 3.37 (m, 2H, —CH_2), 5.90 (s, 1H, —CH), 7.27–7.47 (m, 3H, ArH), 7.74 (s, 1H, —

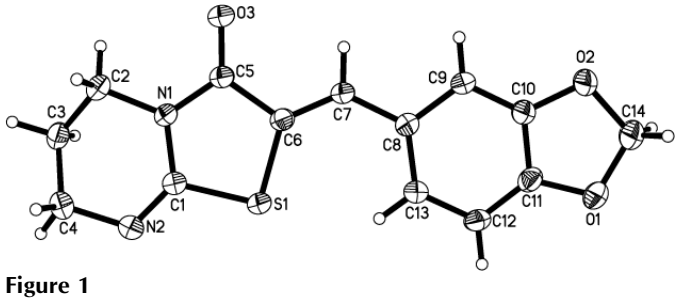


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

CH); m.p. 424–425 K. 20 mg of (I) was dissolved in 15 ml trichloromethane; the solution was kept at room temperature for 15 d. Natural evaporation gave colorless single crystals of (I), suitable for X-ray analysis.

Crystal data

$C_{14}H_{12}N_2O_3S$	$Z = 2$
$M_r = 288.32$	$D_x = 1.447$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.494$ (4) Å	Cell parameters from 887 reflections
$b = 8.050$ (5) Å	$\theta = 3.0$–$26.5°$
$c = 12.097$ (7) Å	$\mu = 0.25$ mm^{-1}
$\alpha = 82.402$ (13)°	$T = 293$ (2) K
$\beta = 79.337$ (12)°	Block, colorless
$\gamma = 67.609$ (12)°	0.22 × 0.18 × 0.10 mm
$V = 661.6$ (7) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2696 independent reflections
	2070 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.017$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$\theta_{max} = 26.6°$
	$h = -9 \rightarrow 8$
$T_{min} = 0.877$, $T_{max} = 0.980$	$k = -10 \rightarrow 9$
3961 measured reflections	$l = -15 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.128P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.039$	
$wR(F^2) = 0.114$	
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2696 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
H-atom parameters constrained	

H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

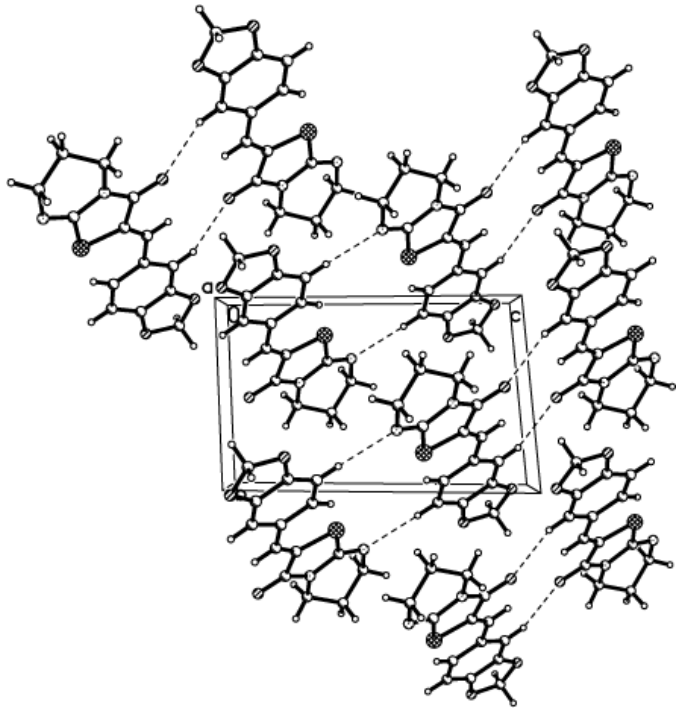


Figure 2
The crystal structure of (I), viewed along the *a* axis. C—H···N and C—H···O interactions are indicated by dashed lines.

References

Bruker (1997). *SADABS, SMART, SAINT* and *SHELXTL*. Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.