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1-[1-(2-Hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydropyrimidin-5-yl]-1-ethanone[1]

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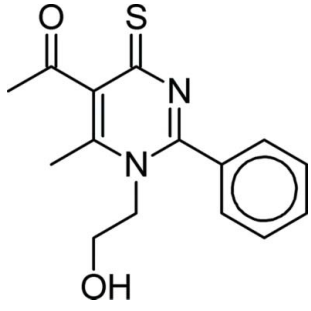
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.212; data-to-parameter ratio = 13.3.

The title compound, $C_{15}H_{16}N_2O_2S$, is of interest with respect to antibacterial and anticancer activity and shows some trypanocidal activity. The crystal structure displays O—H···N hydrogen bonding, forming a chain along [001]. A weak non-classical hydrogen bond of type C—H···S connects molecules across an inversion centre. The packing is also mediated by an intermolecular C=O···π ring interaction connecting centrosymmetrically related molecules. Steric effects are responsible for the molecular conformation.

Related literature

Thioxopyrimidine is an essential structural unit of several heterocycles and displays a wide range of interesting biological and pharmacological properties, such as anticancer and antimicrobial activities (Cocco *et al.*, 1995, 2001). 4-Thioxopyrimidine was obtained by the formal aza-[3+3] cycloaddition reaction of acyclic *N*-alkyl-substituted enaminones with benzoyl isothiocyanate (Cunha *et al.*, 2007).



[1] Structural studies of 4-thioxopyrimidines. Part 1.

Experimental

Crystal data

$C_{15}H_{16}N_2O_2S$
M_r = 288.37
Monoclinic, $P2_1/c$
a = 11.3833 (14) Å
b = 10.4303 (17) Å
c = 12.8157 (14) Å
β = 106.944 (9)°
V = 1455.6 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.00 mm^{-1}
T = 297 (2) K
0.35 × 0.25 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.525, T_{max} = 0.669
2832 measured reflections
2465 independent reflections
2293 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
2 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.212
S = 1.14
2465 reflections
185 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O21—H21···N3^{i}	0.82	2.17	2.972 (3)	167
C13—H13C···S^{ii}	0.96	2.84	3.789 (3)	172

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2101).

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supplementary materials

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1-[1-(2-Hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydropyrimidin-5-yl]-1-ethanone

J. R. Sabino, C. Lariucci, R. M. Bastos and S. Cunha

Comment

Thioxopyrimidine is an essential structural unit of several heterocycles, which displays a wide range of interesting biological, and pharmacological properties, such as anticancer and antimicrobial activities (Cocco *et al.*, 2001, Cocco *et al.*, 1995). Despite these characteristics, solid-state studies of such 4-thioxopyrimidines are scarce. Recently, we described our results concerning the formal aza-[3+3] cycloaddition reaction of acyclic *N*-alkyl-substituted enaminones with benzoyl isotiocyanate for direct 4-thioxopyrimidines synthesis (Cunha *et al.*, 2007). Compound (I) showed some level of trypanocidal activity.

This is the first of a series of three papers devoted to the structural analisys of 4-thioxopyrimidines, Ethyl 1-(2-hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydro-5- pyrimidinecarboxylate and Ethyl 1-butyl-6-methyl-2-phenyl-4-thioxo-1,4-dihydro-5-pyrimidinecarboxylate will be presented in Part 2 and Part 3.

The molecule of (I) is depicted in Fig. 1. The conformation is defined by steric effects, which force a rotation of the phenyl ring relative to the mean plane though the pyrimidine group (rms 0.032 Å) by 84.19 (8) °, and keep the torsion angles C20–C19–N1–C6 of -92.1 (3)° and C4–C5–C14–O15 of 104.8 (3) °. The observed bond distances C4═S, C14═O15, C2═N3 and C5═C6 are indicative of doubly bonded atoms. The bonds C2–C7 and C5–C14 are longer than the expected formal single bond distance by 0.033 and 0.052 Å, respectively, a consequence of the phenyl ring and acetyl group rotations relative to the pyrimidine ring plane and lack of π-orbital overlap on these bonds.

The packing (Fig. 2) is stabilized by an intermolecular H-bond of type O21–H21···N3^{i} and a non-classical intermolecular interaction of type C13–H13C···S^{ii} [Symmetry codes: (i) x, -y+3/2, z+1/2; (ii) -x, -y+1, -z+1]. The former connects molecules in linear chains along to the [0 0 1] direction and the later connects parallel molecules about an inversion center. Also, a non-H weak intermolecular C14═O15···π-ring interaction mediates the molecular packing between molecules related by an inversion center with distances O15···C2^{ii} and O15···N1^{ii} of 3.173 (3) and 2.922 (3) Å, respectively.

Experimental

Compound (I) (m.p. 508.7–509.6 K) was prepared according to a known procedure (Cunha *et al.*, 2007). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in $CHCl_3$ at room temperature.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms or = 1.2U_{eq}(C) for other H atoms. Atom H21 attached to O2 was assigned a bond distance of 0.82 Å and refined with riding constraints using U_{iso}(H16) = 1.2U_{eq}(O). Atom H21 and the methyl H atoms attached to C13 and C16 were allowed to rotate to fit the electron density.

Figures

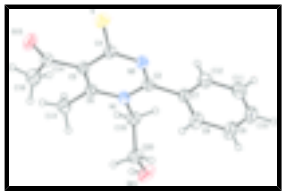

Fig. 1. A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

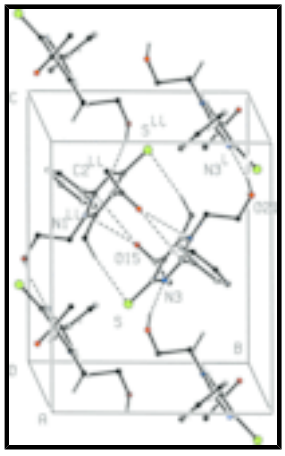

Fig. 2. A packing diagram for (I). Intermolecular contacts are shown as dashed lines. Only the H atoms involved in hydrogen bonds are shown.

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Crystal data

$C_{15}H_{16}N_2O_2S$	F_{000} = 608
M_r = 288.37	D_x = 1.316 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation λ = 1.54180 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 11.3833 (14) Å	θ = 18.4–43.4º
b = 10.4303 (17) Å	μ = 2.00 mm^{-1}
c = 12.8157 (14) Å	T = 297 (2) K
β = 106.944 (9)º	Prism, colourless
V = 1455.6 (3) Å^3	0.35 × 0.25 × 0.2 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	θ_{max} = 67.2º
T = 297(2) K	θ_{min} = 4.1º
non–profiled ω/2θ scans	h = −13→13
Absorption correction: ψ scan (North *et al.*, 1968)	k = 0→12
T_{min} = 0.525, T_{max} = 0.669	l = −1→15
2832 measured reflections	2 standard reflections

2465 independent reflections
2293 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$

every 120 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.212$
$S = 1.14$
2465 reflections
185 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1484P)^2 + 0.5327P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997),
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.049 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.2055 (2)	0.6771 (2)	0.4151 (2)	0.0379 (6)
C4	0.0370 (2)	0.5648 (2)	0.3104 (2)	0.0398 (6)
C5	−0.0344 (2)	0.6197 (2)	0.37357 (19)	0.0381 (6)
C6	0.0206 (2)	0.6935 (2)	0.4626 (2)	0.0380 (6)
C7	0.3353 (2)	0.7183 (3)	0.4316 (2)	0.0432 (7)
C8	0.3584 (3)	0.8187 (3)	0.3714 (3)	0.0565 (8)
H8	0.2934	0.8627	0.3241	0.068*
C9	0.4788 (3)	0.8549 (4)	0.3811 (3)	0.0719 (10)
H9	0.4944	0.9229	0.3401	0.086*
C10	0.5744 (3)	0.7899 (4)	0.4511 (4)	0.0773 (11)
H10	0.6549	0.8139	0.4574	0.093*
C11	0.5523 (3)	0.6903 (4)	0.5115 (4)	0.0806 (12)
H11	0.618	0.6469	0.5587	0.097*
C12	0.4323 (3)	0.6526 (3)	0.5036 (3)	0.0620 (9)
H12	0.4174	0.5851	0.5455	0.074*
C13	−0.0490 (3)	0.7478 (3)	0.5336 (3)	0.0534 (7)
H13A	−0.1354	0.7377	0.499	0.08*
H13B	−0.03	0.8372	0.5456	0.08*
H13C	−0.0266	0.7036	0.6023	0.08*

C14	−0.1707 (2)	0.5926 (3)	0.3418 (2)	0.0453 (7)
C16	−0.2484 (3)	0.6676 (3)	0.2491 (3)	0.0642 (9)
H16A	−0.3296	0.6318	0.2266	0.096*
H16B	−0.2135	0.6646	0.1894	0.096*
H16C	−0.2524	0.755	0.2713	0.096*
C19	0.2104 (2)	0.7881 (3)	0.5869 (2)	0.0453 (7)
H19A	0.2942	0.7566	0.6113	0.054*
H19B	0.1717	0.7682	0.6431	0.054*
C20	0.2126 (3)	0.9326 (3)	0.5732 (2)	0.0550 (8)
H20A	0.2487	0.9539	0.5155	0.066*
H20B	0.1296	0.9662	0.5534	0.066*
N1	0.14383 (18)	0.72046 (19)	0.48503 (16)	0.0379 (5)
N3	0.15686 (18)	0.6024 (2)	0.33238 (17)	0.0421 (6)
O15	−0.2127 (2)	0.5186 (2)	0.3936 (2)	0.0675 (7)
O21	0.2829 (2)	0.9875 (3)	0.67280 (19)	0.0693 (7)
H21	0.2486	0.9747	0.7199	0.104*
S	−0.02136 (6)	0.45695 (7)	0.21138 (6)	0.0530 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0354 (12)	0.0382 (12)	0.0411 (12)	−0.0003 (9)	0.0126 (9)	0.0008 (10)
C4	0.0432 (13)	0.0375 (12)	0.0395 (12)	−0.0037 (9)	0.0134 (10)	0.0021 (10)
C5	0.0372 (12)	0.0360 (12)	0.0414 (13)	−0.0018 (9)	0.0120 (10)	0.0043 (10)
C6	0.0391 (12)	0.0341 (12)	0.0428 (13)	0.0000 (9)	0.0151 (10)	0.0027 (10)
C7	0.0329 (12)	0.0460 (14)	0.0508 (14)	−0.0029 (10)	0.0122 (10)	−0.0084 (12)
C8	0.0454 (15)	0.0673 (19)	0.0604 (17)	−0.0084 (13)	0.0209 (13)	0.0026 (15)
C9	0.0545 (18)	0.078 (2)	0.093 (3)	−0.0160 (16)	0.0357 (17)	−0.002 (2)
C10	0.0455 (17)	0.077 (2)	0.115 (3)	−0.0157 (16)	0.0323 (18)	−0.031 (2)
C11	0.0428 (16)	0.069 (2)	0.118 (3)	0.0060 (15)	0.0043 (18)	−0.015 (2)
C12	0.0437 (15)	0.0530 (16)	0.082 (2)	0.0007 (12)	0.0064 (14)	−0.0023 (16)
C13	0.0537 (15)	0.0545 (16)	0.0603 (17)	−0.0026 (12)	0.0296 (13)	−0.0078 (14)
C14	0.0394 (13)	0.0412 (13)	0.0556 (15)	−0.0030 (10)	0.0144 (11)	0.0005 (12)
C16	0.0471 (15)	0.0654 (19)	0.072 (2)	0.0039 (13)	0.0039 (13)	0.0128 (16)
C19	0.0489 (14)	0.0472 (14)	0.0395 (13)	−0.0029 (11)	0.0123 (11)	−0.0050 (11)
C20	0.0677 (18)	0.0490 (16)	0.0527 (16)	−0.0151 (13)	0.0244 (14)	−0.0123 (13)
N1	0.0388 (10)	0.0379 (11)	0.0378 (11)	−0.0003 (8)	0.0125 (8)	0.0000 (8)
N3	0.0411 (11)	0.0429 (11)	0.0452 (12)	−0.0041 (9)	0.0173 (9)	−0.0055 (10)
O15	0.0474 (12)	0.0666 (14)	0.0890 (17)	−0.0107 (10)	0.0209 (11)	0.0214 (13)
O21	0.0808 (16)	0.0743 (15)	0.0632 (14)	−0.0388 (12)	0.0376 (12)	−0.0265 (13)
S	0.0551 (5)	0.0535 (6)	0.0501 (5)	−0.0125 (3)	0.0146 (3)	−0.0143 (3)

Geometric parameters (Å, °)

C4—S	1.680 (3)	C19—C20	1.519 (4)
C14—O15	1.205 (4)	C20—O21	1.415 (4)
C2—N3	1.302 (3)	C8—H8	0.93
C4—N3	1.368 (3)	C9—H9	0.93
C19—N1	1.483 (3)	C10—H10	0.93

C2—C7	1.493 (3)	C11—H11	0.93
C6—C13	1.482 (4)	C12—H12	0.93
C14—C16	1.480 (4)	C13—H13A	0.96
C2—N1	1.367 (3)	C13—H13B	0.96
C4—C5	1.424 (4)	C13—H13C	0.96
C5—C6	1.368 (4)	C16—H16A	0.96
C5—C14	1.511 (3)	C16—H16B	0.96
C6—N1	1.377 (3)	C16—H16C	0.96
C7—C8	1.370 (4)	C19—H19A	0.97
C7—C12	1.396 (4)	C19—H19B	0.97
C8—C9	1.393 (4)	C20—H20A	0.97
C9—C10	1.371 (6)	C20—H20B	0.97
C10—C11	1.362 (6)	O21—H21	0.82
C11—C12	1.396 (5)		
N3—C2—N1	123.6 (2)	C10—C9—H9	120.1
N3—C2—C7	117.1 (2)	C8—C9—H9	120.1
N1—C2—C7	119.2 (2)	C11—C10—H10	119.8
N3—C4—C5	117.9 (2)	C9—C10—H10	119.8
N3—C4—S	119.93 (19)	C10—C11—H11	119.6
C5—C4—S	122.20 (19)	C12—C11—H11	119.6
C6—C5—C4	120.2 (2)	C7—C12—H12	120.7
C6—C5—C14	120.9 (2)	C11—C12—H12	120.7
C4—C5—C14	118.9 (2)	C6—C13—H13A	109.5
C5—C6—N1	118.7 (2)	C6—C13—H13B	109.5
C5—C6—C13	122.1 (2)	C6—C13—H13C	109.5
N1—C6—C13	119.2 (2)	C14—C16—H16A	109.5
C8—C7—C12	120.2 (3)	C14—C16—H16B	109.5
C8—C7—C2	119.0 (2)	C14—C16—H16C	109.5
C12—C7—C2	120.8 (3)	N1—C19—H19A	109
C7—C8—C9	120.1 (3)	C20—C19—H19A	109
C10—C9—C8	119.8 (4)	N1—C19—H19B	109
C11—C10—C9	120.4 (3)	C20—C19—H19B	109
C10—C11—C12	120.9 (3)	O21—C20—H20A	109.9
C7—C12—C11	118.6 (3)	C19—C20—H20A	109.9
O15—C14—C16	122.7 (3)	O21—C20—H20B	109.9
O15—C14—C5	120.8 (2)	C19—C20—H20B	109.9
C16—C14—C5	116.4 (2)	C20—O21—H21	109.5
N1—C19—C20	113.0 (2)	H13A—C13—H13B	109.5
O21—C20—C19	108.7 (3)	H13A—C13—H13C	109.5
C2—N1—C6	118.9 (2)	H13B—C13—H13C	109.5
C2—N1—C19	120.4 (2)	H16A—C16—H16B	109.5
C6—N1—C19	120.6 (2)	H16A—C16—H16C	109.5
C2—N3—C4	120.1 (2)	H16B—C16—H16C	109.5
C7—C8—H8	119.9	H19A—C19—H19B	107.8
C9—C8—H8	119.9	H20A—C20—H20B	108.3
N3—C4—C5—C6	−8.8 (4)	C6—C5—C14—O15	−74.0 (4)
S—C4—C5—C6	171.19 (19)	C4—C5—C14—O15	104.8 (3)
N3—C4—C5—C14	172.4 (2)	C6—C5—C14—C16	102.4 (3)

S—C4—C5—C14	−7.6 (3)	C4—C5—C14—C16	−78.8 (3)
C4—C5—C6—N1	4.3 (3)	N1—C19—C20—O21	−177.2 (2)
C14—C5—C6—N1	−176.9 (2)	N3—C2—N1—C6	−5.7 (4)
C4—C5—C6—C13	−177.2 (2)	C7—C2—N1—C6	173.1 (2)
C14—C5—C6—C13	1.6 (4)	N3—C2—N1—C19	170.4 (2)
N3—C2—C7—C8	81.4 (3)	C7—C2—N1—C19	−10.8 (3)
N1—C2—C7—C8	−97.4 (3)	C5—C6—N1—C2	2.7 (3)
N3—C2—C7—C12	−95.9 (3)	C13—C6—N1—C2	−175.8 (2)
N1—C2—C7—C12	85.2 (3)	C5—C6—N1—C19	−173.4 (2)
C12—C7—C8—C9	0.7 (5)	C13—C6—N1—C19	8.1 (3)
C2—C7—C8—C9	−176.7 (3)	C20—C19—N1—C2	91.8 (3)
C7—C8—C9—C10	−0.1 (5)	C20—C19—N1—C6	−92.1 (3)
C8—C9—C10—C11	−0.2 (6)	N1—C2—N3—C4	1.1 (4)
C9—C10—C11—C12	−0.1 (6)	C7—C2—N3—C4	−177.7 (2)
C8—C7—C12—C11	−0.9 (5)	C5—C4—N3—C2	6.1 (4)
C2—C7—C12—C11	176.4 (3)	S—C4—N3—C2	−173.90 (19)
C10—C11—C12—C7	0.6 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21···N3^{i}	0.82	2.17	2.972 (3)	167
C13—H13C···S^{ii}	0.96	2.84	3.789 (3)	172

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1

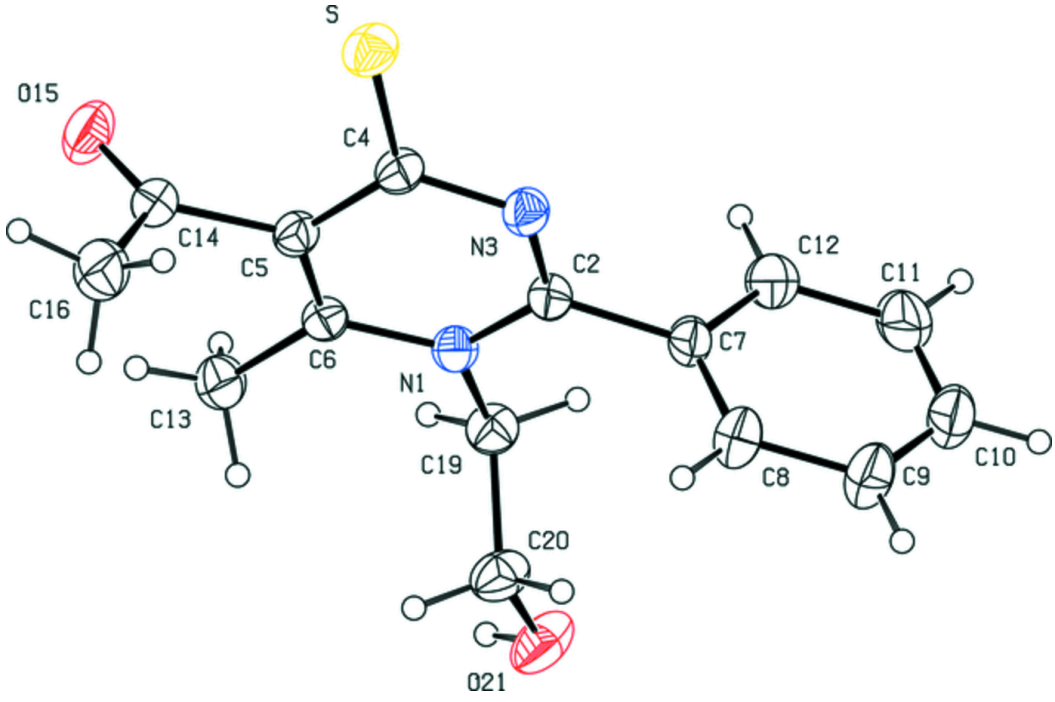

Fig. 2