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Ethyl 1-(2-hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydropyrimidine-5-carboxylate[1]

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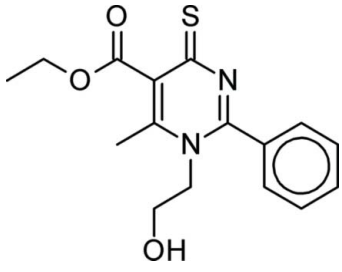
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; R factor = 0.061; wR factor = 0.156; data-to-parameter ratio = 14.0.

The title compound, $C_{16}H_{18}N_2O_3S$, is of interest with respect to antibacterial and anticancer activity and shows some trypanocidal activity. The crystal packing displays O—H···N and C—H···S hydrogen bonds, forming a chain parallel to the [100] direction. The crystal structure also shows a short intermolecular C=O···π-ring interaction connecting centrosymmetrically related molecules.

Related literature

For the synthesis, see: Cunha *et al.* (2007). The corresponding compound with an acetyl group on C5 is described by Sabino *et al.* (2007). Despite the different chemical nature of these substituent groups, both compounds pack in a similar manner.



Experimental

Crystal data

$C_{16}H_{18}N_2O_3S$
M_r = 318.39
Triclinic, $P\overline{1}$
a = 8.2262 (15) Å
b = 11.0683 (18) Å
c = 11.112 (2) Å
α = 60.317 (15)°
β = 74.555 (16)°
γ = 69.765 (16)°
V = 819.1 (3) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 1.88 mm^{-1}
T = 297 (2) K
0.35 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.597, T_{max} = 0.686
2997 measured reflections
2837 independent reflections
2630 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
2 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.156
S = 1.18
2837 reflections
203 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 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	2.21	2.986 (2)	159
C13—H13B···S^{i}	0.96	2.73	3.640 (2)	160

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

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; 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: *ORTEP-3* (Farrugia, 1997) and *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: CF2102).

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[1] Structural studies of 4-thioxopyrimidines. Part 2

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supplementary materials

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Ethyl 1-(2-hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydropyrimidine-5-carboxylate

J. R. Sabino, I. Vencato, R. M. Bastos and S. Cunha

Comment

In continuation of our structural studies of bioactive thioxopyrimidine (Sabino *et al.*, 2007), we present the analysis of the 4-thioxopyrimidine derivative **I**, which exhibited some level of trypanocidal activity. This derivative differs from 1-[1-(2-hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydro-5-pyrimidinyl]-1-ethanone by a substituent on the ring atom C5, where (I) has a carboxylate group instead of an acetyl group.

The molecular structure of (I) is depicted in Fig. 1. The conformation of **I** is defined by steric effects. The angle between the pyrimidine mean plane (rms 0.017 Å) and the mean plane through the phenyl ring is 87.71 (8)°. The torsion angle C20—C19—N1—C6 is -89.1 (2)° and C4—C5—C14—O15 is 91.1 (3)°. Bond lengths are within the expected ranges with the exception of the C2–C7 and C5–C14 bonds which present the same elongation of 0.03 Å from the formal single bond distance.

The crystal packing (Fig. 2) is mediated by a hydrogen bond of type O21–H21···$N3^{ii}$ and a non-classical hydrogen bond of type C13–H13B···S^{ii} [Symmetry code: (ii) 1-x, -y, 1-z], which connect neighboring molecules in a linear chain along [100]. Intermolecular contacts of type C14═O15^{i}···π-ring [Symmetry code: (i)1-x, -y, 1-z] mediate the packing via the sp^2 ring atoms C6, N1 and C2, building a dimer about an inversion center. The distance N1···$O15^{i}$ is 2.858 (2) Å, 0.21 Å shorter than the sum of van der Waals radii of the atoms involved.

Experimental

Compound (I) [m.p. >573 K] was prepared according to a known procedure (Cunha *et al.*, 2007). Single crystals of I were obtained by slow evaporation of a solution in $CHCl_3$ at room temperature.

Refinement

All H atoms were positioned in idealized coordinates with distances 0.93–0.97 and 0.82 Å for the C atoms and O atoms, respectively. H atoms were refined isotropicaly and allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(methyl C,*O*).

Figures

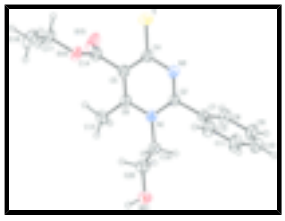

Fig. 1. View of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitray radius.

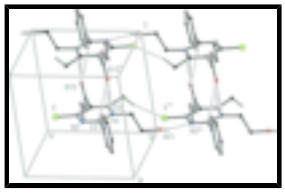

Fig. 2. Packing diagram of (I). Intra- and intermolecular contacts are shown as dashed lines. Only the H atoms involved in H-bonds are shown.

ethyl 1-(2-hydroxyethyl)-6-methyl-2-phenyl-4-thioxo-1,4-dihydropyrimidine- 5-carboxylate

Crystal data

$C_{16}H_{18}N_2O_3S$	$Z = 2$
$M_r = 318.39$	$F_{000} = 336$
Triclinic, $P\overline{1}$	$D_x = 1.291$ Mg m^{-3}
Hall symbol: -P 1	Cu $K\alpha$ radiation $\lambda = 1.5418$ Å
$a = 8.2262$ (15) Å	Cell parameters from 25 reflections
$b = 11.0683$ (18) Å	$\theta = 16.4$–50.6°
$c = 11.112$ (2) Å	$\mu = 1.88$ mm^{-1}
$\alpha = 60.317$ (15)°	$T = 297$ (2) K
$\beta = 74.555$ (16)°	Prism, yellow
$\gamma = 69.765$ (16)°	0.35 × 0.3 × 0.2 mm
$V = 819.1$ (3) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$\theta_{max} = 67.1^\circ$
non–profiled $\omega/2\theta$ scans	$\theta_{min} = 4.6^\circ$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -9 \rightarrow 9$
$T_{min} = 0.597$, $T_{max} = 0.686$	$k = -11 \rightarrow 13$
2997 measured reflections	$l = 0 \rightarrow 13$
2837 independent reflections	2 standard reflections
2630 reflections with $I > 2\sigma(I)$	every 120 min
$R_{int} = 0.025$	intensity decay: 1%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.156$

$S = 1.18$

2837 reflections

203 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2 + 0.1677P]$

where $P = (F_o^2 + 2F_c^2)/3$

$\Delta\rho_{max} = 0.33$ e Å^{-3}

$\Delta\rho_{min} = -0.38$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.181 (8)

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}
S	0.87159 (7)	0.12970 (7)	0.20413 (8)	0.0745 (3)
N1	0.30304 (19)	0.27798 (15)	0.30438 (15)	0.0430 (4)
C2	0.4169 (2)	0.35827 (18)	0.27024 (18)	0.0430 (4)
N3	0.5847 (2)	0.31579 (16)	0.24112 (17)	0.0495 (4)
C4	0.6574 (3)	0.1819 (2)	0.2448 (2)	0.0488 (5)
C5	0.5435 (2)	0.09323 (18)	0.28549 (18)	0.0456 (5)
C6	0.3693 (2)	0.13928 (18)	0.31766 (18)	0.0449 (4)
C7	0.3467 (2)	0.50303 (18)	0.2683 (2)	0.0468 (5)
C8	0.3321 (3)	0.5140 (2)	0.3891 (2)	0.0645 (6)
H8	0.3674	0.4323	0.4703	0.077*
C9	0.2646 (4)	0.6472 (3)	0.3889 (3)	0.0757 (7)
H9	0.2532	0.6551	0.4705	0.091*
C10	0.2147 (4)	0.7674 (3)	0.2692 (3)	0.0799 (8)
H10	0.1683	0.8566	0.2698	0.096*
C11	0.2326 (4)	0.7569 (2)	0.1498 (3)	0.0876 (9)
H11	0.1994	0.8392	0.0685	0.105*
C12	0.2999 (4)	0.6243 (2)	0.1476 (2)	0.0686 (6)
H12	0.3132	0.6177	0.0652	0.082*
C13	0.2481 (3)	0.0445 (2)	0.3715 (3)	0.0632 (6)
H13A	0.3096	−0.0439	0.3656	0.095*
H13B	0.1524	0.0924	0.3165	0.095*
H13C	0.2045	0.0243	0.4669	0.095*
C14	0.6211 (3)	−0.0571 (2)	0.2998 (2)	0.0520 (5)
O15	0.6649 (3)	−0.15931 (16)	0.40618 (18)	0.0888 (7)
O16	0.63106 (19)	−0.06239 (13)	0.18248 (14)	0.0546 (4)
C17	0.7073 (3)	−0.2034 (2)	0.1833 (2)	0.0651 (6)
H17A	0.6387	−0.2702	0.2507	0.078*

H17B	0.8254	−0.2418	0.208	0.078*
C18	0.7075 (4)	−0.1824 (3)	0.0412 (3)	0.0876 (9)
H18A	0.7589	−0.2726	0.037	0.131*
H18B	0.7738	−0.1147	−0.0246	0.131*
H18C	0.5897	−0.1462	0.0188	0.131*
C19	0.1130 (2)	0.3349 (2)	0.3320 (2)	0.0515 (5)
H19A	0.0928	0.4045	0.3667	0.062*
H19B	0.0628	0.2568	0.4035	0.062*
C20	0.0236 (3)	0.4058 (2)	0.2020 (3)	0.0636 (6)
H20A	0.0689	0.487	0.1312	0.076*
H20B	0.0452	0.3377	0.1651	0.076*
O21	−0.1572 (2)	0.45270 (19)	0.2374 (3)	0.0871 (6)
H21	−0.21	0.4136	0.2198	0.131*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0453 (4)	0.0706 (4)	0.1179 (6)	0.0022 (3)	−0.0192 (3)	−0.0558 (4)
N1	0.0458 (8)	0.0357 (8)	0.0484 (8)	−0.0048 (6)	−0.0136 (6)	−0.0191 (6)
C2	0.0487 (10)	0.0354 (9)	0.0458 (9)	−0.0077 (7)	−0.0110 (7)	−0.0181 (8)
N3	0.0491 (9)	0.0390 (8)	0.0632 (10)	−0.0073 (6)	−0.0104 (7)	−0.0255 (7)
C4	0.0513 (10)	0.0429 (10)	0.0552 (10)	−0.0033 (8)	−0.0176 (8)	−0.0242 (8)
C5	0.0572 (11)	0.0347 (9)	0.0467 (10)	−0.0034 (7)	−0.0190 (7)	−0.0185 (8)
C6	0.0565 (10)	0.0351 (9)	0.0466 (9)	−0.0073 (7)	−0.0192 (7)	−0.0172 (8)
C7	0.0478 (10)	0.0353 (9)	0.0580 (11)	−0.0076 (7)	−0.0078 (8)	−0.0226 (8)
C8	0.0837 (15)	0.0487 (11)	0.0625 (13)	−0.0103 (10)	−0.0110 (10)	−0.0287 (10)
C9	0.0861 (17)	0.0671 (15)	0.0901 (17)	−0.0139 (12)	−0.0023 (13)	−0.0545 (14)
C10	0.0803 (16)	0.0480 (13)	0.124 (2)	0.0007 (11)	−0.0272 (15)	−0.0513 (15)
C11	0.120 (2)	0.0358 (11)	0.105 (2)	−0.0075 (12)	−0.0478 (17)	−0.0210 (12)
C12	0.0980 (18)	0.0420 (11)	0.0653 (13)	−0.0137 (11)	−0.0219 (12)	−0.0202 (10)
C13	0.0647 (13)	0.0467 (11)	0.0855 (15)	−0.0147 (9)	−0.0221 (11)	−0.0281 (11)
C14	0.0648 (12)	0.0380 (10)	0.0541 (11)	−0.0025 (8)	−0.0212 (8)	−0.0211 (9)
O15	0.1527 (19)	0.0406 (8)	0.0626 (10)	0.0108 (9)	−0.0489 (10)	−0.0206 (8)
O16	0.0703 (9)	0.0381 (7)	0.0556 (8)	0.0038 (6)	−0.0226 (6)	−0.0250 (6)
C17	0.0788 (15)	0.0417 (11)	0.0746 (14)	0.0060 (9)	−0.0219 (11)	−0.0334 (10)
C18	0.119 (2)	0.0667 (15)	0.0732 (15)	0.0008 (15)	−0.0080 (14)	−0.0458 (13)
C19	0.0454 (10)	0.0467 (10)	0.0645 (12)	−0.0078 (8)	−0.0085 (8)	−0.0278 (9)
C20	0.0557 (12)	0.0510 (11)	0.0833 (15)	−0.0028 (9)	−0.0277 (10)	−0.0268 (11)
O21	0.0531 (9)	0.0676 (10)	0.1593 (18)	0.0065 (7)	−0.0400 (10)	−0.0650 (12)

Geometric parameters (Å, °)

S—C4	1.665 (2)	O16—C17	1.463 (2)
N1—C2	1.365 (2)	C17—C18	1.478 (3)
N1—C6	1.384 (2)	C19—C20	1.512 (3)
N1—C19	1.480 (2)	C20—O21	1.412 (3)
N1—O15^{i}	2.858 (2)	O21—H21	0.82
C2—O15^{i}	3.182 (3)	C8—H8	0.93

C6—O15^{i}	3.117 (2)	C9—H9	0.93
C2—N3	1.302 (2)	C10—H10	0.93
C2—C7	1.494 (2)	C11—H11	0.93
N3—C4	1.377 (2)	C12—H12	0.93
C4—C5	1.420 (3)	C13—H13A	0.96
C5—C6	1.358 (3)	C13—H13B	0.96
C5—C14	1.503 (2)	C13—H13C	0.96
C6—C13	1.496 (3)	C17—H17A	0.97
C7—C12	1.372 (3)	C17—H17B	0.97
C7—C8	1.377 (3)	C18—H18A	0.96
C8—C9	1.384 (3)	C18—H18B	0.96
C9—C10	1.367 (4)	C18—H18C	0.96
C10—C11	1.354 (4)	C19—H19A	0.97
C11—C12	1.388 (3)	C19—H19B	0.97
C14—O15	1.195 (3)	C20—H20A	0.97
C14—O16	1.313 (2)	C20—H20B	0.97
C2—N1—C6	118.59 (15)	C6—C13—H13B	109.5
C2—N1—C19	121.19 (14)	H13A—C13—H13B	109.5
C6—N1—C19	120.19 (15)	C6—C13—H13C	109.5
N3—C2—N1	124.07 (15)	H13A—C13—H13C	109.5
N3—C2—C7	117.36 (15)	H13B—C13—H13C	109.5
N1—C2—C7	118.55 (15)	O15—C14—O16	124.43 (17)
C2—N3—C4	119.96 (16)	O15—C14—C5	123.69 (18)
N3—C4—C5	117.43 (17)	O16—C14—C5	111.86 (15)
N3—C4—S	119.70 (15)	C14—O16—C17	116.71 (15)
C5—C4—S	122.87 (15)	O16—C17—C18	106.96 (17)
C6—C5—C4	121.37 (16)	O16—C17—H17A	110.3
C6—C5—C14	120.09 (17)	C18—C17—H17A	110.3
C4—C5—C14	118.46 (17)	O16—C17—H17B	110.3
C5—C6—N1	118.38 (16)	C18—C17—H17B	110.3
C5—C6—C13	122.62 (17)	H17A—C17—H17B	108.6
N1—C6—C13	118.97 (17)	C17—C18—H18A	109.5
C12—C7—C8	120.04 (18)	C17—C18—H18B	109.5
C12—C7—C2	120.84 (17)	H18A—C18—H18B	109.5
C8—C7—C2	119.11 (17)	C17—C18—H18C	109.5
C7—C8—C9	119.5 (2)	H18A—C18—H18C	109.5
C7—C8—H8	120.2	H18B—C18—H18C	109.5
C9—C8—H8	120.2	N1—C19—C20	111.69 (17)
C10—C9—C8	120.2 (2)	N1—C19—H19A	109.3
C10—C9—H9	119.9	C20—C19—H19A	109.3
C8—C9—H9	119.9	N1—C19—H19B	109.3
C11—C10—C9	120.2 (2)	C20—C19—H19B	109.3
C11—C10—H10	119.9	H19A—C19—H19B	107.9
C9—C10—H10	119.9	O21—C20—C19	108.4 (2)
C10—C11—C12	120.6 (2)	O21—C20—H20A	110
C10—C11—H11	119.7	C19—C20—H20A	110
C12—C11—H11	119.7	O21—C20—H20B	110
C7—C12—C11	119.4 (2)	C19—C20—H20B	110

C7—C12—H12	120.3	H20A—C20—H20B	108.4
C11—C12—H12	120.3	C20—O21—H21	109.5
C6—C13—H13A	109.5		
C6—N1—C2—N3	4.2 (3)	N1—C2—C7—C12	−92.5 (2)
C19—N1—C2—N3	−178.05 (16)	N3—C2—C7—C8	−90.3 (2)
C6—N1—C2—C7	−174.66 (15)	N1—C2—C7—C8	88.7 (2)
C19—N1—C2—C7	3.1 (2)	C12—C7—C8—C9	2.2 (3)
N1—C2—N3—C4	−0.5 (3)	C2—C7—C8—C9	−179.0 (2)
C7—C2—N3—C4	178.33 (15)	C7—C8—C9—C10	−0.8 (4)
C2—N3—C4—C5	−2.0 (3)	C8—C9—C10—C11	−0.7 (4)
C2—N3—C4—S	178.69 (14)	C9—C10—C11—C12	0.7 (5)
N3—C4—C5—C6	0.9 (3)	C8—C7—C12—C11	−2.1 (4)
S—C4—C5—C6	−179.84 (13)	C2—C7—C12—C11	179.0 (2)
N3—C4—C5—C14	−175.92 (15)	C10—C11—C12—C7	0.7 (4)
S—C4—C5—C14	3.4 (2)	C6—C5—C14—O15	−85.8 (3)
C4—C5—C6—N1	2.7 (3)	C4—C5—C14—O15	91.1 (3)
C14—C5—C6—N1	179.42 (15)	C6—C5—C14—O16	92.9 (2)
C4—C5—C6—C13	−175.09 (17)	C4—C5—C14—O16	−90.3 (2)
C14—C5—C6—C13	1.7 (3)	O15—C14—O16—C17	−2.3 (3)
C2—N1—C6—C5	−5.1 (2)	C5—C14—O16—C17	179.06 (17)
C19—N1—C6—C5	177.14 (15)	C14—O16—C17—C18	−179.1 (2)
C2—N1—C6—C13	172.76 (16)	C2—N1—C19—C20	93.1 (2)
C19—N1—C6—C13	−5.0 (2)	C6—N1—C19—C20	−89.1 (2)
N3—C2—C7—C12	88.6 (2)	N1—C19—C20—O21	177.98 (15)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O21—H21···N3^{ii}	0.82	2.21	2.986 (2)	159
C13—H13B···S^{ii}	0.96	2.73	3.640 (2)	160

Symmetry codes: (ii) *x*−1, *y*, *z*.

Fig. 1

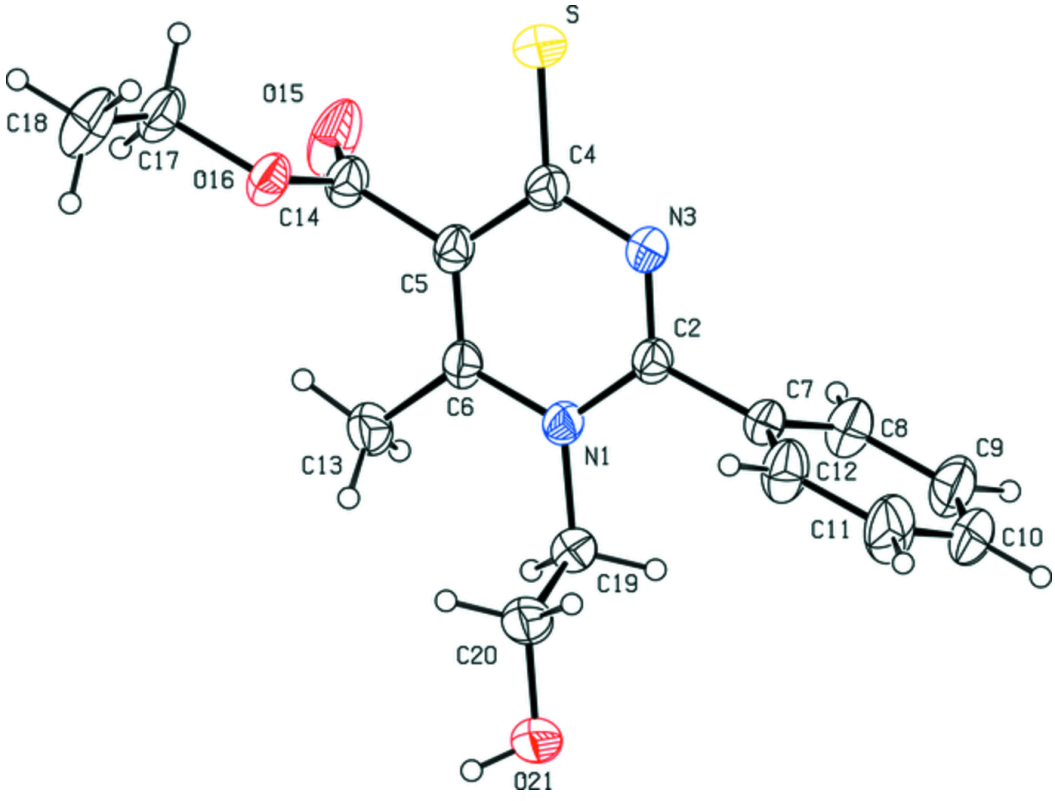
S
O15
C18
C17
O16
C14
C4
N3
C5
C2
C7
C6
C8
C9
C13
N1
C12
C11
C10
C19
C20
O21

Fig. 2