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# 7-(Benzylsulfanyl)-5-(2-methoxyphenyl)-1,3-dimethyl-5,6-dihydropyrimido[4,5-*d*]pyrimidine-2,4(1*H*,3*H*)-dione

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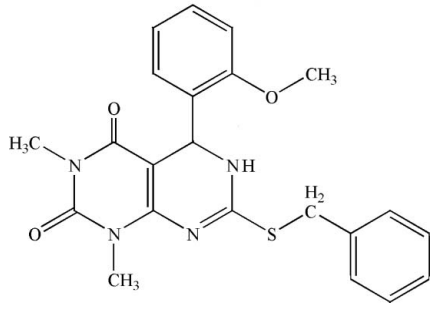
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.056; $wR$ factor = 0.135; data-to-parameter ratio = 19.4.

In the molecule of the title compound, $C_{22}H_{22}N_4O_3S$, the benzene and phenyl rings are oriented at a dihedral angle of 88.72 (4)°. The other two rings have flattened-boat conformations. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules.

## Related literature

For general background, see: Sharma *et al.* (2004); Quiroga *et al.* (2002); Devi *et al.* (2003). For bond-length data, see: Allen *et al.* (1987). For ring conformation puckering parameters, see: Cremer & Pople (1975).



## Experimental

### *Crystal data*

$C_{22}H_{22}N_4O_3S$
$M_r$ = 422.51
Monoclinic, $P2_1/n$
$a$ = 10.9216 (9) Å
$b$ = 8.8528 (5) Å
$c$ = 20.7263 (15) Å
$\beta$ = 90.638 (6)°
$V$ = 2003.8 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 294 (2) K
0.4 × 0.3 × 0.05 mm

### *Data collection*

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min}$ = 0.928, $T_{max}$ = 0.985
23191 measured reflections
5394 independent reflections
4510 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.063

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.135
$S$ = 1.15
5394 reflections
278 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N4—H4$B$···O1$^{i}$ | 0.85 (3) | 2.02 (3) | 2.836 (2) | 161 (2) |

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); 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: HK2501).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o1666 [ **doi:10.1107/S160053680802401X** ]

# 7-(Benzylsulfanyl)-5-(2-methoxyphenyl)-1,3-dimethyl-5,6-dihydropyrimido[4,5-*d*]pyrimidine-2,4(1*H*,3*H*)-dione

**A. Bazgir and F. Faraji**

## Comment

The importance of fused pyrimidines, common source for the development of new potential therapeutic agents (Sharma *et al.*, 2004), is well known. Among them, the pyrimido[2,3-d]pyrimidines are an important class of annulated uracils with biological significance because of their connection with purine pteridine system (Quiroga *et al.*, 2002). Numerous reports delineate the antitumor, antivial, antioxidant, antifungal and hepatoprotective activities of these compounds (Devi *et al.*, 2003). Therefore, for the preparation of these complex molecules large efforts have been directed towards the synthetic manipulation of uracils. We report herein the synthesis and crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C1-C6) and D (C16-C21) are, of course, planar and they are oriented at a dihedral angle of A/D = 88.72 (4)°. Rings B (N2/N3/C9/C11/C13/C14) and C (N1/N4/C8/C9/C14/C15) have flattened-boat [φ = 105.63 (2)°, θ = 100.53 (3)° (for ring B) and φ = 29.58 (3)°, θ = 58.23 (3)° (for ring C)] conformations, having total puckering amplitudes, $Q_T$, of 0.120 (3) and 0.364 (3) Å, respectively (Cremer & Pople, 1975).

In the crystal structure, intermolecular N-H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

## Experimental

For the preparation of the title compound, 6-amino-1,3-dimethyluracil (0.15 g, 1 mmol), 2-methylbenzaldehyde (0.12 g, 1 mmol), 2-benzylthiourea hydrochloride (0.30 g, 1.5 mmol) and p-toluenesulfonic acid (0.1 g) were mixed. The reaction mixture was placed in a screw capped vial and irradiated for 5 min with a power of 700 W microwave irradiation. After cooling, the reaction mixture was washed with water, and then recrystallized from ethyl acetate to afford the title compound (yield; 0.25 g. 65%, m.p. 519-521 K).

## Refinement

H4B atom (for NH) was located in difference syntheses and refined isotropically [N-H = 0.85 (3) Å and $U_{iso}$(H) = 0.049 (6) Å$^2$]. The remaining H atoms were positioned geometrically, with C-H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H, respectively, and constrained to ride on their parent atoms with $U_{iso}$(H) = x$U_{eq}$(C), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

## Figures

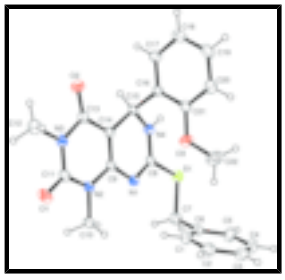

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

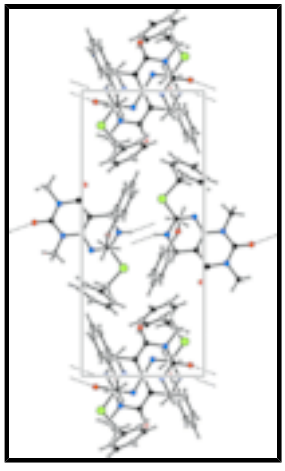

Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

## 7-(Benzylsulfanyl)-5-(2-methoxyphenyl)-1,3-dimethyl-5,6-dihydropyrimido[4,5-\ *d*]pyrimidine-2,4(1*H*,3*H*)-dione

*Crystal data*

| | |
|---|---|
| $C_{22}H_{22}N_4O_3S$ | $F_{000}$ = 888 |
| $M_r$ = 422.51 | $D_x$ = 1.401 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 2175 reflections |
| $a$ = 10.9216 (9) Å | θ = 2.1–29.3º |
| $b$ = 8.8528 (5) Å | μ = 0.20 mm$^{-1}$ |
| $c$ = 20.7263 (15) Å | $T$ = 294 (2) K |
| β = 90.638 (6)º | Plate, colorless |
| $V$ = 2003.8 (2) Å$^3$ | 0.4 × 0.3 × 0.05 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | $R_{int}$ = 0.063 |
| φ and ω scans | $\theta_{max}$ = 29.3º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1998) | $\theta_{min}$ = 2.1º |

$T_{min}$ = 0.928, $T_{max}$ = 0.985
23191 measured reflections
5394 independent reflections
4510 reflections with $I > 2\sigma(I)$

$h = -14 \rightarrow 15$
$k = -11 \rightarrow 12$
$l = -28 \rightarrow 28$

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.135$
$S = 1.15$
5394 reflections
278 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.6815P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.038$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$
Extinction correction: none

*Special details*

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.34470 (5) | 0.84201 (5) | 0.12120 (2) | 0.04173 (13) |
| O1 | 0.34328 (15) | 0.11232 (15) | −0.03830 (7) | 0.0508 (4) |
| O2 | 0.32309 (14) | 0.52289 (16) | −0.17071 (6) | 0.0469 (3) |
| O3 | 0.08733 (11) | 0.71339 (19) | 0.00741 (7) | 0.0502 (4) |
| N1 | 0.33713 (13) | 0.57943 (16) | 0.05837 (7) | 0.0322 (3) |
| N2 | 0.34856 (13) | 0.34559 (15) | 0.00770 (7) | 0.0320 (3) |
| N3 | 0.32991 (14) | 0.31591 (16) | −0.10452 (7) | 0.0349 (3) |
| N4 | 0.33864 (13) | 0.80243 (16) | −0.00370 (7) | 0.0326 (3) |
| H4B | 0.341 (2) | 0.898 (3) | −0.0043 (11) | 0.049 (6)* |
| C1 | 0.15207 (19) | 0.5279 (2) | 0.18010 (10) | 0.0459 (4) |
| H1 | 0.1982 | 0.4632 | 0.1547 | 0.055* |
| C2 | 0.0325 (2) | 0.4896 (3) | 0.19479 (10) | 0.0509 (5) |
| H2 | −0.0011 | 0.4002 | 0.179 | 0.061* |
| C3 | −0.0368 (2) | 0.5839 (3) | 0.23278 (10) | 0.0526 (5) |
| H3 | −0.1169 | 0.5582 | 0.2429 | 0.063* |
| C4 | 0.0136 (2) | 0.7165 (3) | 0.25561 (11) | 0.0592 (6) |
| H4 | −0.0328 | 0.7807 | 0.2811 | 0.071* |
| C5 | 0.1329 (2) | 0.7546 (3) | 0.24080 (10) | 0.0525 (5) |
| H5 | 0.1659 | 0.8443 | 0.2566 | 0.063* |
| C6 | 0.20406 (17) | 0.6607 (2) | 0.20257 (8) | 0.0388 (4) |
| C7 | 0.33376 (18) | 0.7042 (2) | 0.18617 (9) | 0.0432 (4) |

| | | | | |
|---|---|---|---|---|
| H7A | 0.3782 | 0.6139 | 0.1739 | 0.052* |
| H7B | 0.3733 | 0.7453 | 0.2245 | 0.052* |
| C8 | 0.33809 (14) | 0.72697 (18) | 0.05202 (8) | 0.0300 (3) |
| C9 | 0.33441 (13) | 0.50102 (17) | 0.00128 (8) | 0.0288 (3) |
| C10 | 0.37101 (17) | 0.2765 (2) | 0.07119 (9) | 0.0382 (4) |
| H10A | 0.2942 | 0.2532 | 0.0909 | 0.057* |
| H10B | 0.4159 | 0.3458 | 0.0981 | 0.057* |
| H10C | 0.4176 | 0.1854 | 0.066 | 0.057* |
| C11 | 0.34047 (15) | 0.25006 (18) | −0.04476 (9) | 0.0345 (3) |
| C12 | 0.3234 (2) | 0.2154 (2) | −0.16086 (10) | 0.0483 (5) |
| H12A | 0.3785 | 0.1321 | −0.1546 | 0.072* |
| H12B | 0.3461 | 0.2704 | −0.1988 | 0.072* |
| H12C | 0.2414 | 0.178 | −0.166 | 0.072* |
| C13 | 0.32425 (15) | 0.47335 (19) | −0.11530 (8) | 0.0331 (3) |
| C14 | 0.31967 (14) | 0.56353 (17) | −0.05833 (8) | 0.0293 (3) |
| C15 | 0.30427 (14) | 0.73269 (17) | −0.06556 (8) | 0.0287 (3) |
| H15 | 0.3644 | 0.7662 | −0.0973 | 0.034* |
| C16 | 0.17931 (14) | 0.78714 (18) | −0.08936 (8) | 0.0301 (3) |
| C17 | 0.16945 (17) | 0.8519 (2) | −0.15010 (9) | 0.0378 (4) |
| H17 | 0.2381 | 0.8546 | −0.1762 | 0.045* |
| C18 | 0.0604 (2) | 0.9128 (2) | −0.17325 (10) | 0.0469 (5) |
| H18 | 0.0559 | 0.9551 | −0.2143 | 0.056* |
| C19 | −0.0411 (2) | 0.9096 (3) | −0.13438 (11) | 0.0532 (5) |
| H19 | −0.1143 | 0.9515 | −0.1491 | 0.064* |
| C20 | −0.03502 (18) | 0.8450 (3) | −0.07381 (10) | 0.0493 (5) |
| H20 | −0.1041 | 0.8439 | −0.048 | 0.059* |
| C21 | 0.07381 (16) | 0.7812 (2) | −0.05109 (9) | 0.0371 (4) |
| C22 | −0.0178 (2) | 0.7044 (4) | 0.04781 (13) | 0.0771 (9) |
| H22A | −0.0516 | 0.8035 | 0.0535 | 0.116* |
| H22B | 0.0057 | 0.6636 | 0.089 | 0.116* |
| H22C | −0.0781 | 0.64 | 0.028 | 0.116* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0512 (11) | 0.0413 (10) | 0.0454 (10) | 0.0063 (8) | 0.0071 (8) | −0.0019 (8) |
| C2 | 0.0550 (12) | 0.0452 (11) | 0.0528 (11) | −0.0047 (9) | 0.0061 (9) | 0.0045 (9) |
| C3 | 0.0478 (11) | 0.0587 (13) | 0.0515 (11) | 0.0019 (10) | 0.0107 (9) | 0.0136 (10) |
| C4 | 0.0607 (14) | 0.0600 (14) | 0.0573 (13) | 0.0121 (11) | 0.0216 (11) | −0.0024 (11) |
| C5 | 0.0591 (13) | 0.0489 (12) | 0.0495 (11) | −0.0002 (9) | 0.0081 (9) | −0.0100 (9) |
| C6 | 0.0425 (9) | 0.0429 (10) | 0.0311 (8) | 0.0043 (7) | −0.0013 (7) | 0.0027 (7) |
| C7 | 0.0402 (9) | 0.0522 (11) | 0.0372 (9) | 0.0014 (8) | −0.0051 (7) | −0.0013 (8) |
| C8 | 0.0233 (7) | 0.0286 (7) | 0.0383 (8) | 0.0005 (5) | 0.0010 (6) | −0.0006 (6) |
| C9 | 0.0228 (7) | 0.0239 (7) | 0.0398 (8) | 0.0006 (5) | 0.0039 (6) | 0.0030 (6) |
| C10 | 0.0377 (9) | 0.0320 (8) | 0.0447 (9) | 0.0034 (7) | 0.0009 (7) | 0.0115 (7) |
| C11 | 0.0318 (8) | 0.0251 (7) | 0.0467 (9) | −0.0018 (6) | 0.0029 (7) | 0.0016 (7) |
| C12 | 0.0609 (12) | 0.0340 (9) | 0.0501 (11) | −0.0013 (9) | −0.0015 (9) | −0.0085 (8) |
| C13 | 0.0302 (8) | 0.0285 (7) | 0.0407 (8) | −0.0014 (6) | 0.0046 (6) | 0.0017 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0258 (7) | 0.0242 (7) | 0.0381 (8) | −0.0002 (5) | 0.0027 (6) | 0.0029 (6) |
| C15 | 0.0262 (7) | 0.0242 (7) | 0.0356 (8) | −0.0012 (5) | 0.0034 (6) | 0.0052 (6) |
| C16 | 0.0287 (7) | 0.0246 (7) | 0.0369 (8) | −0.0004 (6) | 0.0005 (6) | 0.0034 (6) |
| C17 | 0.0413 (9) | 0.0327 (8) | 0.0395 (9) | −0.0009 (7) | 0.0007 (7) | 0.0054 (7) |
| C18 | 0.0540 (12) | 0.0421 (10) | 0.0444 (10) | 0.0064 (9) | −0.0092 (8) | 0.0101 (8) |
| C19 | 0.0440 (11) | 0.0559 (12) | 0.0593 (12) | 0.0163 (9) | −0.0118 (9) | 0.0057 (10) |
| C20 | 0.0320 (9) | 0.0632 (13) | 0.0525 (11) | 0.0102 (9) | 0.0007 (8) | 0.0039 (10) |
| C21 | 0.0303 (8) | 0.0393 (9) | 0.0415 (9) | 0.0023 (7) | 0.0007 (6) | 0.0042 (7) |
| C22 | 0.0416 (12) | 0.127 (3) | 0.0628 (15) | 0.0041 (14) | 0.0173 (10) | 0.0345 (16) |
| N1 | 0.0338 (7) | 0.0268 (6) | 0.0361 (7) | 0.0024 (5) | 0.0011 (5) | 0.0018 (5) |
| N2 | 0.0324 (7) | 0.0240 (6) | 0.0396 (7) | 0.0015 (5) | 0.0024 (5) | 0.0056 (5) |
| N3 | 0.0379 (7) | 0.0257 (7) | 0.0411 (7) | −0.0017 (5) | 0.0034 (6) | −0.0025 (6) |
| N4 | 0.0340 (7) | 0.0220 (6) | 0.0418 (8) | −0.0020 (5) | −0.0016 (6) | 0.0017 (6) |
| O1 | 0.0688 (10) | 0.0210 (6) | 0.0624 (9) | −0.0018 (6) | −0.0002 (7) | 0.0037 (6) |
| O2 | 0.0654 (9) | 0.0384 (7) | 0.0371 (6) | −0.0033 (6) | 0.0075 (6) | 0.0023 (6) |
| O3 | 0.0279 (6) | 0.0759 (10) | 0.0468 (7) | 0.0022 (6) | 0.0057 (5) | 0.0226 (7) |
| S1 | 0.0468 (3) | 0.0340 (2) | 0.0444 (2) | −0.00253 (18) | 0.00230 (19) | −0.00687 (18) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N4—H4B | 0.85 (3) | C11—N2 | 1.379 (2) |
| C1—C6 | 1.383 (3) | C12—N3 | 1.469 (2) |
| C1—C2 | 1.387 (3) | C12—H12A | 0.96 |
| C1—H1 | 0.93 | C12—H12B | 0.96 |
| C2—C3 | 1.379 (3) | C12—H12C | 0.96 |
| C2—H2 | 0.93 | C13—O2 | 1.229 (2) |
| C3—C4 | 1.378 (4) | C13—N3 | 1.413 (2) |
| C3—H3 | 0.93 | C13—C14 | 1.427 (2) |
| C4—C5 | 1.383 (3) | C14—C15 | 1.514 (2) |
| C4—H4 | 0.93 | C15—N4 | 1.468 (2) |
| C5—C6 | 1.392 (3) | C15—C16 | 1.524 (2) |
| C5—H5 | 0.93 | C15—H15 | 0.98 |
| C6—C7 | 1.510 (3) | C16—C17 | 1.386 (2) |
| C7—S1 | 1.822 (2) | C16—C21 | 1.407 (2) |
| C7—H7A | 0.97 | C17—C18 | 1.388 (3) |
| C7—H7B | 0.97 | C17—H17 | 0.93 |
| C8—N1 | 1.313 (2) | C18—C19 | 1.377 (3) |
| C8—N4 | 1.334 (2) | C18—H18 | 0.93 |
| C8—S1 | 1.7596 (17) | C19—C20 | 1.381 (3) |
| C9—C14 | 1.362 (2) | C19—H19 | 0.93 |
| C9—N1 | 1.372 (2) | C20—C21 | 1.393 (3) |
| C9—N2 | 1.3908 (19) | C20—H20 | 0.93 |
| C10—N2 | 1.469 (2) | C21—O3 | 1.359 (2) |
| C10—H10A | 0.96 | C22—O3 | 1.431 (2) |
| C10—H10B | 0.96 | C22—H22A | 0.96 |
| C10—H10C | 0.96 | C22—H22B | 0.96 |
| C11—O1 | 1.227 (2) | C22—H22C | 0.96 |
| C11—N3 | 1.373 (2) | | |
| C6—C1—C2 | 121.20 (19) | N3—C13—C14 | 115.02 (14) |

| | | | |
|---|---|---|---|
| C6—C1—H1 | 119.4 | C9—C14—C13 | 121.24 (14) |
| C2—C1—H1 | 119.4 | C9—C14—C15 | 120.24 (14) |
| C3—C2—C1 | 120.1 (2) | C13—C14—C15 | 118.45 (14) |
| C3—C2—H2 | 120 | N4—C15—C14 | 107.61 (13) |
| C1—C2—H2 | 120 | N4—C15—C16 | 111.66 (13) |
| C4—C3—C2 | 119.4 (2) | C14—C15—C16 | 116.28 (13) |
| C4—C3—H3 | 120.3 | N4—C15—H15 | 106.9 |
| C2—C3—H3 | 120.3 | C14—C15—H15 | 106.9 |
| C3—C4—C5 | 120.4 (2) | C16—C15—H15 | 106.9 |
| C3—C4—H4 | 119.8 | C17—C16—C21 | 118.14 (15) |
| C5—C4—H4 | 119.8 | C17—C16—C15 | 119.01 (14) |
| C4—C5—C6 | 120.9 (2) | C21—C16—C15 | 122.79 (14) |
| C4—C5—H5 | 119.5 | C16—C17—C18 | 122.16 (17) |
| C6—C5—H5 | 119.5 | C16—C17—H17 | 118.9 |
| C1—C6—C5 | 117.99 (19) | C18—C17—H17 | 118.9 |
| C1—C6—C7 | 121.56 (17) | C19—C18—C17 | 118.85 (18) |
| C5—C6—C7 | 120.45 (18) | C19—C18—H18 | 120.6 |
| C6—C7—S1 | 113.99 (13) | C17—C18—H18 | 120.6 |
| C6—C7—H7A | 108.8 | C18—C19—C20 | 120.64 (18) |
| S1—C7—H7A | 108.8 | C18—C19—H19 | 119.7 |
| C6—C7—H7B | 108.8 | C20—C19—H19 | 119.7 |
| S1—C7—H7B | 108.8 | C19—C20—C21 | 120.48 (19) |
| H7A—C7—H7B | 107.6 | C19—C20—H20 | 119.8 |
| N1—C8—N4 | 125.81 (15) | C21—C20—H20 | 119.8 |
| N1—C8—S1 | 119.63 (13) | O3—C21—C20 | 124.34 (17) |
| N4—C8—S1 | 114.52 (12) | O3—C21—C16 | 115.97 (15) |
| C14—C9—N1 | 125.32 (14) | C20—C21—C16 | 119.68 (17) |
| C14—C9—N2 | 120.04 (15) | O3—C22—H22A | 109.5 |
| N1—C9—N2 | 114.64 (14) | O3—C22—H22B | 109.5 |
| N2—C10—H10A | 109.5 | H22A—C22—H22B | 109.5 |
| N2—C10—H10B | 109.5 | O3—C22—H22C | 109.5 |
| H10A—C10—H10B | 109.5 | H22A—C22—H22C | 109.5 |
| N2—C10—H10C | 109.5 | H22B—C22—H22C | 109.5 |
| H10A—C10—H10C | 109.5 | C8—N1—C9 | 114.65 (14) |
| H10B—C10—H10C | 109.5 | C11—N2—C9 | 121.68 (14) |
| O1—C11—N3 | 121.48 (17) | C11—N2—C10 | 117.34 (14) |
| O1—C11—N2 | 121.47 (17) | C9—N2—C10 | 120.98 (14) |
| N3—C11—N2 | 117.05 (14) | C11—N3—C13 | 124.39 (14) |
| N3—C12—H12A | 109.5 | C11—N3—C12 | 117.56 (15) |
| N3—C12—H12B | 109.5 | C13—N3—C12 | 118.05 (15) |
| H12A—C12—H12B | 109.5 | C8—N4—C15 | 122.81 (14) |
| N3—C12—H12C | 109.5 | C8—N4—H4B | 120.9 (16) |
| H12A—C12—H12C | 109.5 | C15—N4—H4B | 114.5 (16) |
| H12B—C12—H12C | 109.5 | C21—O3—C22 | 117.82 (16) |
| O2—C13—N3 | 119.99 (16) | C8—S1—C7 | 102.25 (9) |
| O2—C13—C14 | 124.99 (16) | | |
| C6—C1—C2—C3 | −0.4 (3) | C17—C16—C21—O3 | 178.46 (16) |
| C1—C2—C3—C4 | 0.3 (3) | C15—C16—C21—O3 | −4.6 (3) |
| C2—C3—C4—C5 | −0.2 (4) | C17—C16—C21—C20 | −2.5 (3) |

| | | | |
|---|---|---|---|
| C3—C4—C5—C6 | 0.2 (4) | C15—C16—C21—C20 | 174.39 (17) |
| C2—C1—C6—C5 | 0.4 (3) | N4—C8—N1—C9 | −1.1 (2) |
| C2—C1—C6—C7 | −179.27 (18) | S1—C8—N1—C9 | −178.71 (11) |
| C4—C5—C6—C1 | −0.2 (3) | C14—C9—N1—C8 | −7.4 (2) |
| C4—C5—C6—C7 | 179.4 (2) | N2—C9—N1—C8 | 172.47 (14) |
| C1—C6—C7—S1 | 100.2 (2) | O1—C11—N2—C9 | −174.13 (16) |
| C5—C6—C7—S1 | −79.5 (2) | N3—C11—N2—C9 | 6.3 (2) |
| N1—C9—C14—C13 | 176.78 (15) | O1—C11—N2—C10 | 5.0 (2) |
| N2—C9—C14—C13 | −3.1 (2) | N3—C11—N2—C10 | −174.62 (14) |
| N1—C9—C14—C15 | −0.1 (2) | C14—C9—N2—C11 | −4.1 (2) |
| N2—C9—C14—C15 | −179.98 (13) | N1—C9—N2—C11 | 175.99 (14) |
| O2—C13—C14—C9 | −172.94 (17) | C14—C9—N2—C10 | 176.78 (15) |
| N3—C13—C14—C9 | 7.4 (2) | N1—C9—N2—C10 | −3.1 (2) |
| O2—C13—C14—C15 | 4.0 (2) | O1—C11—N3—C13 | 178.99 (17) |
| N3—C13—C14—C15 | −175.67 (14) | N2—C11—N3—C13 | −1.4 (2) |
| C9—C14—C15—N4 | 13.62 (19) | O1—C11—N3—C12 | −0.7 (3) |
| C13—C14—C15—N4 | −163.38 (13) | N2—C11—N3—C12 | 178.94 (16) |
| C9—C14—C15—C16 | −112.44 (17) | O2—C13—N3—C11 | 175.12 (16) |
| C13—C14—C15—C16 | 70.56 (19) | C14—C13—N3—C11 | −5.2 (2) |
| N4—C15—C16—C17 | 124.18 (16) | O2—C13—N3—C12 | −5.2 (2) |
| C14—C15—C16—C17 | −111.82 (17) | C14—C13—N3—C12 | 174.47 (15) |
| N4—C15—C16—C21 | −52.7 (2) | N1—C8—N4—C15 | 17.5 (2) |
| C14—C15—C16—C21 | 71.3 (2) | S1—C8—N4—C15 | −164.81 (11) |
| C21—C16—C17—C18 | 1.3 (3) | C14—C15—N4—C8 | −21.9 (2) |
| C15—C16—C17—C18 | −175.73 (17) | C16—C15—N4—C8 | 106.81 (17) |
| C16—C17—C18—C19 | 0.4 (3) | C20—C21—O3—C22 | 1.3 (3) |
| C17—C18—C19—C20 | −1.0 (3) | C16—C21—O3—C22 | −179.8 (2) |
| C18—C19—C20—C21 | −0.3 (4) | N1—C8—S1—C7 | −5.17 (15) |
| C19—C20—C21—O3 | −179.0 (2) | N4—C8—S1—C7 | 176.99 (12) |
| C19—C20—C21—C16 | 2.1 (3) | C6—C7—S1—C8 | −84.81 (15) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4—H4B···O1$^{i}$ | 0.85 (3) | 2.02 (3) | 2.836 (2) | 161 (2) |

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

**Fig. 1**

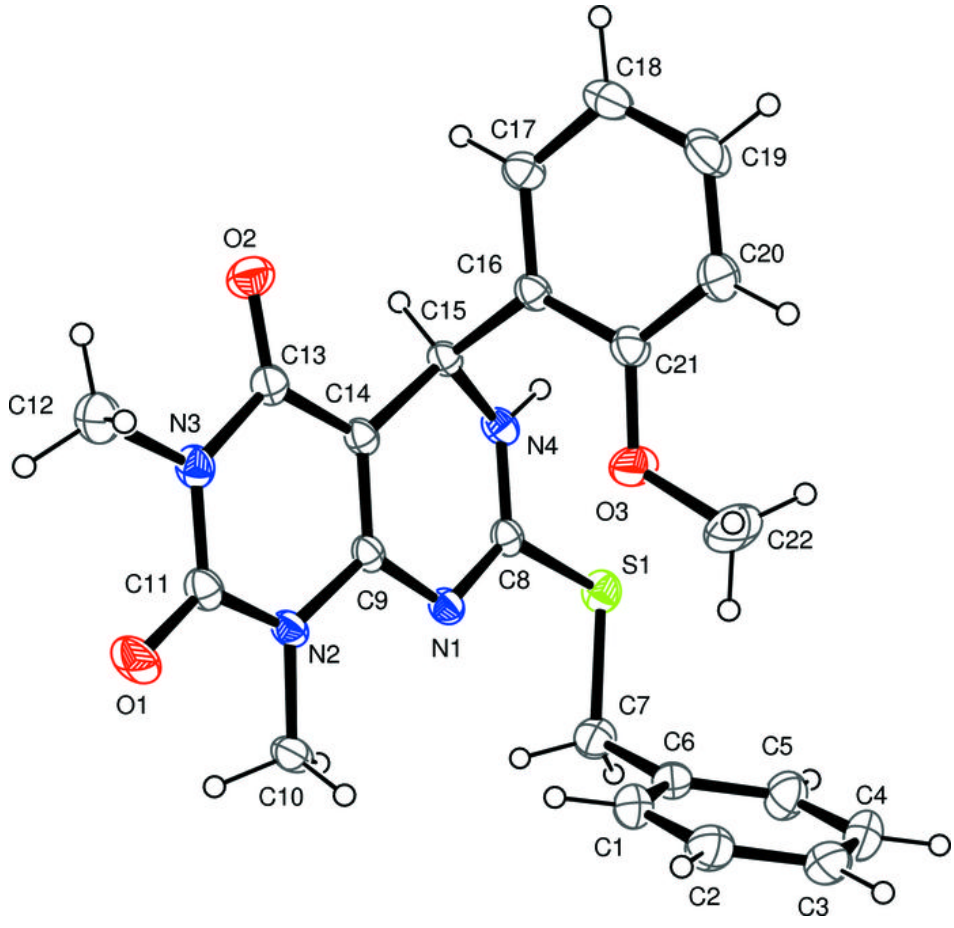
C18
C17
C19
O2
C16
C20
C15
C13
C21
C12
C14
N3
N4
O3
C22
S1
C11
C8
C9
N1
N2
O1
C7
C6
C5
C4
C1
C2
C3
C10

**Fig. 2**