Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Ethyl 4-(4,5-dimethoxy-2-nitrophenyl)-6-methyl-1-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate

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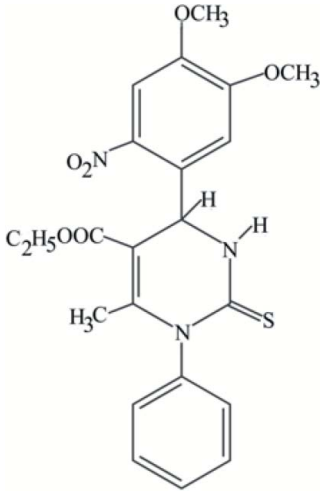
Received 21 May 2007; accepted 27 June 2007

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.044; $wR$ factor = 0.130; data-to-parameter ratio = 17.3.

In the title compound, $C_{22}H_{23}N_3O_6S$, the pyridimine ring adopts a screw boat conformation, with one C and one N atom displaced from the plane of the remaining atoms. In the crystal structure, centrosymmetric dimers are formed *via* pairs of N—H···S hydrogen bonds. The keto O atom of the ester group is disordered equally over two positions.

## Related literature

For related literature, see: Kumaradhas *et al.* (2007).




## Experimental

### Crystal data

$C_{22}H_{23}N_3O_6S$
$M_r$ = 457.49
Triclinic, $P\overline{1}$
$a$ = 8.1475 (8) Å
$b$ = 10.3588 (10) Å
$c$ = 14.8263 (15) Å
$\alpha$ = 86.620 (2)°
$\beta$ = 78.262 (2)°
$\gamma$ = 68.395 (2)°
$V$ = 1138.90 (19) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.19 mm$^{-1}$
$T$ = 296 (2) K
0.21 × 0.11 × 0.08 mm

### Data collection

Bruker APEX II CCD diffractometer
Absorption correction: none
13201 measured reflections
5229 independent reflections
4549 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
$S$ = 1.09
5229 reflections
302 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···S$^{i}$ | 0.86 | 2.61 | 3.4505 (11) | 167 |
| C8—H8$B$···O3$^{ii}$ | 0.96 | 2.56 | 3.400 (2) | 146 |

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x, y-1, z$.

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2430).

## References

Bruker (2000). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Kumaradhas, P., David Stephen, A., Satheesh Rai, N., Kalluraya, B. & Sridhar, B. (2007). *Acta Cryst.* E**63**, o3402.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

 doi:10.1107/S1600536807031534  

# supplementary materials

*Acta Cryst.* (2007). E**63**, o3403 [ **doi:10.1107/S1600536807031534** ]

# Ethyl 4-(4,5-dimethoxy-2-nitrophenyl)-6-methyl-1-phenyl-2-thioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate

**P. Kumaradhas, A. David Stephen, N. Satheesh Rai, B. Kalluraya and B. Sridar**

## Comment

As part of our ongoing strudies of biologically active molecules (Kumaradhas *et al.*, 2007), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

Within the 1,4–pyridimine ring, the C10—N2 and C10—N3 bonds are somewhat short, and the C1—C2 bond of the substituent 2–nitro–4,5–dimethoxy phenyl ring is lengthened, probably due to steric factors. The pyridimine ring shows a distorted envelope conformation in which C9 and N3 lie above the mean plane of the other ring atoms, having deviations of 0.303 (1)Å and 0.144 (1)Å respectively.

The dihedral angle of the pendant phenyl and aromatic rings with the central ring are 85.9 (1)° and 86.4 (1)° respectively, which marks the near-perpendicularity of both the rings.

In the crystal, the molecules of (I) form inversion dimers (Table 1, Fig. 2) *via* pairs of N—H···S hydrogen bonds thus generating $R_2^2(8)$ loops. A weak C—H···O interaction also occurs.

## Experimental

A mixture of 2-nitroveratraldehyde (0.01 mol), ethylaceto acetate (0.015 mole), thiourea (0.01 mol) and concentrated sulfuric acid (1–2 drops) in ethanol (10 ml) taken in a borosil beaker (100 ml capacity) was zapped inside a microwave oven for 3–4 minutes (power = 160 watt). The reaction mixture was then allowed to stand at room temperature and poured into water. The product separated was filtered, washed with water followed by ethanol, dried and finally recrystallized from ethanol by slow evaporation at room temperature to yield pale yellow rectangular blocks of (I).

## Refinement

Atom O5 is disordered over two sites with equal occupancies. All the H atoms were placed geometrically (C—H = 0.93–0.97 Å, N—H = 0.86 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(carrier) or 1.5$U_{eq}$(methyl C).

## Figures

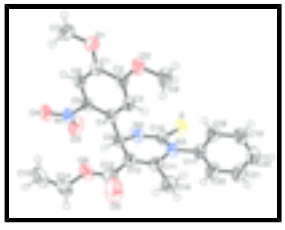

Fig. 1. The molecular structure of (I), showing 50% displacement ellipsoids and arbitrary spheres for the H atoms. Only one orientation of the disordered atom O5 is shown.

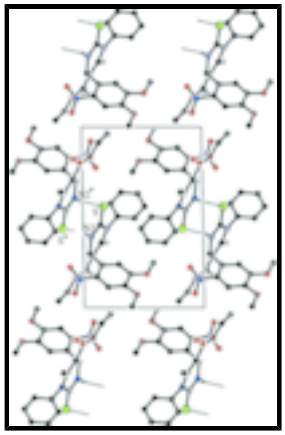

Fig. 2. Part of the crystal structure of (I), showing the formation of an $R_2^2(8)$ dimer. Atoms marked with an asterisk (*) are at the symmetry positions $(1-x, 2-y, 1-z)$. The H atoms not involved in these motifs shown have been omitted.

**Ethyl 4-(4,5-dimethoxy-2-nitrophenyl)-6-methyl-1-phenyl-2-thioxo-1,2,3,4-\ tetrahydropyrimidine-5-carboxylate**

*Crystal data*

| | |
|---|---|
| $C_{22}H_{23}N_3O_6S$ | $Z = 2$ |
| $M_r = 457.49$ | $F_{000} = 480$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.334$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 8.1475$ (8) Å | Cell parameters from 25 reflections |
| $b = 10.3588$ (10) Å | $\theta = 5$–11º |
| $c = 14.8263$ (15) Å | $\mu = 0.19$ mm$^{-1}$ |
| $\alpha = 86.620$ (2)º | $T = 296$ (2) K |
| $\beta = 78.262$ (2)º | Block, pale yellow |
| $\gamma = 68.395$ (2)º | 0.21 × 0.11 × 0.08 mm |
| $V = 1138.90$ (19) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker APEX II CCD diffractometer | $R_{int} = 0.017$ |
| ω scans | $\theta_{max} = 28.0$º |
| Absorption correction: none | $\theta_{min} = 2.1$º |
| 13201 measured reflections | $h = -10 \rightarrow 10$ |
| 5229 independent reflections | $k = -13 \rightarrow 13$ |
| 4549 reflections with $I > 2\sigma(I)$ | $l = -19 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.188P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | $(\Delta/\sigma)_{max} = 0.001$ |

| | |
|---|---|
| $wR(F^2) = 0.130$ | $\Delta\rho_{max} = 0.32$ e Å$^{-3}$ |
| $S = 1.09$ | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| 5229 reflections | Extinction correction: none |
| 302 parameters | |

*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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S | 0.45829 (6) | 0.83000 (4) | 0.43847 (3) | 0.05704 (14) | |
| O1 | 0.66977 (18) | 0.55834 (12) | 0.92626 (9) | 0.0652 (3) | |
| O2 | 0.52874 (18) | 0.47350 (11) | 0.81277 (8) | 0.0621 (3) | |
| O3 | 0.3863 (2) | 1.11302 (13) | 0.77587 (9) | 0.0747 (4) | |
| O4 | 0.38192 (18) | 1.07621 (12) | 0.92019 (8) | 0.0638 (3) | |
| O5 | −0.2304 (13) | 0.9803 (10) | 0.8041 (8) | 0.090 (2) | 0.5 |
| O5' | −0.1923 (13) | 0.9289 (10) | 0.8221 (8) | 0.089 (2) | 0.5 |
| O6 | −0.04308 (15) | 1.06751 (13) | 0.83484 (8) | 0.0618 (3) | |
| N1 | 0.39604 (17) | 1.03669 (12) | 0.84201 (9) | 0.0473 (3) | |
| N2 | 0.32962 (15) | 0.93324 (12) | 0.60670 (8) | 0.0424 (3) | |
| H2 | 0.3986 | 0.9801 | 0.5912 | 0.051* | |
| N3 | 0.18386 (16) | 0.80205 (12) | 0.56207 (8) | 0.0447 (3) | |
| C1 | 0.56626 (19) | 0.65847 (15) | 0.87698 (10) | 0.0461 (3) | |
| C2 | 0.4914 (2) | 0.61169 (14) | 0.81356 (10) | 0.0457 (3) | |
| C3 | 0.38765 (19) | 0.70637 (14) | 0.75925 (9) | 0.0434 (3) | |
| H3 | 0.3384 | 0.6746 | 0.718 | 0.052* | |
| C4 | 0.35371 (17) | 0.84891 (13) | 0.76403 (8) | 0.0372 (3) | |
| C5 | 0.42686 (17) | 0.89020 (13) | 0.82832 (9) | 0.0381 (3) | |
| C6 | 0.53023 (18) | 0.79808 (14) | 0.88500 (9) | 0.0429 (3) | |
| H6 | 0.5748 | 0.8307 | 0.9281 | 0.052* | |
| C7 | 0.7410 (3) | 0.6026 (2) | 0.99266 (16) | 0.0827 (6) | |
| H7A | 0.8186 | 0.6497 | 0.9626 | 0.124* | |
| H7B | 0.8086 | 0.5235 | 1.0242 | 0.124* | |
| H7C | 0.6444 | 0.6649 | 1.0362 | 0.124* | |
| C8 | 0.4525 (3) | 0.41968 (18) | 0.75192 (14) | 0.0709 (5) | |
| H8A | 0.3236 | 0.4612 | 0.7673 | 0.106* | |
| H8B | 0.4892 | 0.3208 | 0.7582 | 0.106* | |
| H8C | 0.4936 | 0.441 | 0.6895 | 0.106* | |
| C9 | 0.23486 (17) | 0.94602 (13) | 0.70240 (9) | 0.0384 (3) | |
| H9 | 0.2029 | 1.0416 | 0.7236 | 0.046* | |
| C10 | 0.31935 (18) | 0.85618 (13) | 0.54131 (9) | 0.0411 (3) | |
| C11 | 0.04369 (18) | 0.84849 (14) | 0.64043 (10) | 0.0437 (3) | |
| C12 | 0.06332 (18) | 0.92001 (14) | 0.70715 (10) | 0.0421 (3) | |

| | | | | |
|---|---|---|---|---|
| C13 | −0.0804 (2) | 0.97947 (18) | 0.78865 (11) | 0.0547 (4) |
| C14 | −0.1783 (3) | 1.1408 (2) | 0.91359 (14) | 0.0761 (5) |
| H14A | −0.2157 | 1.0754 | 0.954 | 0.091* |
| H14B | −0.2831 | 1.2066 | 0.8929 | 0.091* |
| C15 | −0.0993 (3) | 1.2135 (3) | 0.96261 (17) | 0.0924 (7) |
| H15A | 0.0002 | 1.1472 | 0.9857 | 0.139* |
| H15B | −0.1884 | 1.2664 | 1.0131 | 0.139* |
| H15C | −0.0577 | 1.2748 | 0.9213 | 0.139* |
| C16 | −0.1218 (2) | 0.8164 (2) | 0.63980 (14) | 0.0648 (4) |
| H16A | −0.2048 | 0.8462 | 0.6973 | 0.097* |
| H16B | −0.0885 | 0.7182 | 0.6316 | 0.097* |
| H16C | −0.1776 | 0.8643 | 0.5902 | 0.097* |
| C17 | 0.1732 (2) | 0.71097 (15) | 0.49452 (10) | 0.0456 (3) |
| C18 | 0.2479 (2) | 0.56988 (15) | 0.50319 (11) | 0.0495 (3) |
| H18 | 0.3075 | 0.5323 | 0.5513 | 0.059* |
| C19 | 0.2334 (2) | 0.48365 (17) | 0.43937 (13) | 0.0602 (4) |
| H19 | 0.2839 | 0.3878 | 0.4446 | 0.072* |
| C20 | 0.1453 (3) | 0.53926 (19) | 0.36889 (13) | 0.0667 (5) |
| H20 | 0.1357 | 0.4812 | 0.3264 | 0.08* |
| C21 | 0.0711 (3) | 0.6803 (2) | 0.36084 (14) | 0.0750 (5) |
| H21 | 0.0114 | 0.7178 | 0.3127 | 0.09* |
| C22 | 0.0843 (3) | 0.76721 (18) | 0.42366 (13) | 0.0653 (5) |
| H22 | 0.0336 | 0.863 | 0.4182 | 0.078* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S | 0.0731 (3) | 0.0625 (3) | 0.0431 (2) | −0.0406 (2) | 0.00476 (17) | −0.00769 (16) |
| O1 | 0.0813 (8) | 0.0456 (6) | 0.0700 (7) | −0.0122 (6) | −0.0376 (6) | −0.0021 (5) |
| O2 | 0.0946 (9) | 0.0369 (5) | 0.0626 (7) | −0.0250 (6) | −0.0302 (6) | −0.0007 (5) |
| O3 | 0.1192 (11) | 0.0593 (7) | 0.0756 (8) | −0.0612 (8) | −0.0346 (8) | 0.0162 (6) |
| O4 | 0.0815 (8) | 0.0499 (6) | 0.0617 (7) | −0.0215 (6) | −0.0166 (6) | −0.0194 (5) |
| O5 | 0.050 (2) | 0.132 (7) | 0.093 (5) | −0.047 (4) | 0.014 (2) | −0.040 (4) |
| O5' | 0.080 (5) | 0.122 (6) | 0.079 (4) | −0.071 (5) | 0.026 (3) | −0.019 (3) |
| O6 | 0.0557 (6) | 0.0628 (7) | 0.0611 (7) | −0.0246 (5) | 0.0126 (5) | −0.0179 (5) |
| N1 | 0.0533 (7) | 0.0435 (6) | 0.0530 (7) | −0.0261 (5) | −0.0100 (5) | −0.0051 (5) |
| N2 | 0.0493 (6) | 0.0475 (6) | 0.0393 (6) | −0.0309 (5) | −0.0028 (5) | 0.0008 (4) |
| N3 | 0.0509 (6) | 0.0445 (6) | 0.0470 (6) | −0.0272 (5) | −0.0085 (5) | −0.0019 (5) |
| C1 | 0.0484 (7) | 0.0447 (7) | 0.0446 (7) | −0.0150 (6) | −0.0096 (6) | −0.0055 (6) |
| C2 | 0.0574 (8) | 0.0368 (7) | 0.0449 (7) | −0.0193 (6) | −0.0089 (6) | −0.0048 (5) |
| C3 | 0.0526 (7) | 0.0403 (7) | 0.0425 (7) | −0.0216 (6) | −0.0102 (6) | −0.0056 (5) |
| C4 | 0.0393 (6) | 0.0387 (6) | 0.0356 (6) | −0.0195 (5) | −0.0004 (5) | −0.0044 (5) |
| C5 | 0.0388 (6) | 0.0379 (6) | 0.0398 (6) | −0.0199 (5) | 0.0007 (5) | −0.0074 (5) |
| C6 | 0.0435 (7) | 0.0470 (7) | 0.0417 (7) | −0.0202 (6) | −0.0061 (5) | −0.0081 (5) |
| C7 | 0.1015 (15) | 0.0638 (11) | 0.0877 (14) | −0.0146 (10) | −0.0580 (13) | −0.0021 (10) |
| C8 | 0.1120 (15) | 0.0466 (9) | 0.0721 (11) | −0.0417 (10) | −0.0328 (11) | −0.0006 (8) |
| C9 | 0.0419 (6) | 0.0363 (6) | 0.0395 (6) | −0.0198 (5) | −0.0023 (5) | −0.0018 (5) |
| C10 | 0.0479 (7) | 0.0380 (6) | 0.0419 (7) | −0.0216 (6) | −0.0079 (5) | 0.0035 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.0423 (7) | 0.0399 (7) | 0.0529 (8) | −0.0205 (6) | −0.0086 (6) | 0.0044 (5) |
| C12 | 0.0402 (7) | 0.0411 (7) | 0.0471 (7) | −0.0193 (5) | −0.0057 (5) | 0.0038 (5) |
| C13 | 0.0459 (8) | 0.0655 (9) | 0.0538 (8) | −0.0257 (7) | −0.0007 (6) | −0.0023 (7) |
| C14 | 0.0600 (10) | 0.0847 (13) | 0.0672 (11) | −0.0180 (10) | 0.0148 (9) | −0.0229 (10) |
| C15 | 0.0887 (15) | 0.0849 (15) | 0.0886 (15) | −0.0272 (12) | 0.0184 (12) | −0.0394 (12) |
| C16 | 0.0544 (9) | 0.0724 (11) | 0.0808 (12) | −0.0386 (9) | −0.0101 (8) | −0.0065 (9) |
| C17 | 0.0536 (8) | 0.0428 (7) | 0.0486 (7) | −0.0256 (6) | −0.0124 (6) | 0.0004 (6) |
| C18 | 0.0551 (8) | 0.0448 (7) | 0.0536 (8) | −0.0226 (6) | −0.0144 (6) | 0.0044 (6) |
| C19 | 0.0711 (10) | 0.0429 (8) | 0.0718 (11) | −0.0268 (7) | −0.0126 (8) | −0.0043 (7) |
| C20 | 0.0886 (13) | 0.0644 (10) | 0.0639 (10) | −0.0435 (10) | −0.0193 (9) | −0.0084 (8) |
| C21 | 0.1055 (15) | 0.0693 (11) | 0.0692 (11) | −0.0398 (11) | −0.0457 (11) | 0.0091 (9) |
| C22 | 0.0893 (13) | 0.0454 (8) | 0.0726 (11) | −0.0266 (8) | −0.0392 (10) | 0.0089 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S—C10 | 1.6733 (14) | C7—H7C | 0.96 |
| O1—C1 | 1.3558 (18) | C8—H8A | 0.96 |
| O1—C7 | 1.414 (2) | C8—H8B | 0.96 |
| O2—C2 | 1.3496 (16) | C8—H8C | 0.96 |
| O2—C8 | 1.433 (2) | C9—C12 | 1.5053 (17) |
| O3—N1 | 1.2195 (17) | C9—H9 | 0.98 |
| O4—N1 | 1.2212 (16) | C11—C12 | 1.3383 (19) |
| O5—C13 | 1.194 (10) | C11—C16 | 1.505 (2) |
| O5'—C13 | 1.221 (10) | C12—C13 | 1.480 (2) |
| O6—C13 | 1.325 (2) | C14—C15 | 1.457 (3) |
| O6—C14 | 1.456 (2) | C14—H14A | 0.97 |
| N1—C5 | 1.4635 (16) | C14—H14B | 0.97 |
| N2—C10 | 1.3257 (17) | C15—H15A | 0.96 |
| N2—C9 | 1.4588 (16) | C15—H15B | 0.96 |
| N2—H2 | 0.86 | C15—H15C | 0.96 |
| N3—C10 | 1.3874 (17) | C16—H16A | 0.96 |
| N3—C11 | 1.4166 (18) | C16—H16B | 0.96 |
| N3—C17 | 1.4518 (17) | C16—H16C | 0.96 |
| C1—C6 | 1.3730 (19) | C17—C18 | 1.370 (2) |
| C1—C2 | 1.4128 (19) | C17—C22 | 1.376 (2) |
| C2—C3 | 1.378 (2) | C18—C19 | 1.389 (2) |
| C3—C4 | 1.4020 (18) | C18—H18 | 0.93 |
| C3—H3 | 0.93 | C19—C20 | 1.367 (3) |
| C4—C5 | 1.3833 (17) | C19—H19 | 0.93 |
| C4—C9 | 1.5228 (18) | C20—C21 | 1.369 (3) |
| C5—C6 | 1.388 (2) | C20—H20 | 0.93 |
| C6—H6 | 0.93 | C21—C22 | 1.379 (2) |
| C7—H7A | 0.96 | C21—H21 | 0.93 |
| C7—H7B | 0.96 | C22—H22 | 0.93 |
| C1—O1—C7 | 116.94 (13) | N3—C10—S | 122.04 (10) |
| C2—O2—C8 | 117.96 (12) | C12—C11—N3 | 119.26 (12) |
| C13—O6—C14 | 117.29 (14) | C12—C11—C16 | 124.83 (14) |
| O3—N1—O4 | 123.17 (13) | N3—C11—C16 | 115.88 (13) |
| O3—N1—C5 | 119.24 (12) | C11—C12—C13 | 122.83 (13) |

| | | | |
|---|---|---|---|
| O4—N1—C5 | 117.60 (12) | C11—C12—C9 | 121.15 (12) |
| C10—N2—C9 | 126.12 (11) | C13—C12—C9 | 116.02 (12) |
| C10—N2—H2 | 116.9 | O5—C13—O5' | 28.2 (6) |
| C9—N2—H2 | 116.9 | O5—C13—O6 | 118.7 (6) |
| C10—N3—C11 | 121.49 (11) | O5'—C13—O6 | 122.6 (6) |
| C10—N3—C17 | 118.25 (12) | O5—C13—C12 | 127.9 (6) |
| C11—N3—C17 | 119.66 (11) | O5'—C13—C12 | 123.7 (6) |
| O1—C1—C6 | 124.99 (13) | O6—C13—C12 | 111.26 (12) |
| O1—C1—C2 | 115.74 (12) | O6—C14—C15 | 108.14 (16) |
| C6—C1—C2 | 119.27 (13) | O6—C14—H14A | 110.1 |
| O2—C2—C3 | 125.40 (12) | C15—C14—H14A | 110.1 |
| O2—C2—C1 | 115.11 (13) | O6—C14—H14B | 110.1 |
| C3—C2—C1 | 119.49 (12) | C15—C14—H14B | 110.1 |
| C2—C3—C4 | 122.31 (12) | H14A—C14—H14B | 108.4 |
| C2—C3—H3 | 118.8 | C14—C15—H15A | 109.5 |
| C4—C3—H3 | 118.8 | C14—C15—H15B | 109.5 |
| C5—C4—C3 | 116.15 (12) | H15A—C15—H15B | 109.5 |
| C5—C4—C9 | 125.22 (11) | C14—C15—H15C | 109.5 |
| C3—C4—C9 | 118.58 (11) | H15A—C15—H15C | 109.5 |
| C4—C5—C6 | 123.17 (12) | H15B—C15—H15C | 109.5 |
| C4—C5—N1 | 121.33 (12) | C11—C16—H16A | 109.5 |
| C6—C5—N1 | 115.50 (11) | C11—C16—H16B | 109.5 |
| C1—C6—C5 | 119.56 (12) | H16A—C16—H16B | 109.5 |
| C1—C6—H6 | 120.2 | C11—C16—H16C | 109.5 |
| C5—C6—H6 | 120.2 | H16A—C16—H16C | 109.5 |
| O1—C7—H7A | 109.5 | H16B—C16—H16C | 109.5 |
| O1—C7—H7B | 109.5 | C18—C17—C22 | 120.62 (14) |
| H7A—C7—H7B | 109.5 | C18—C17—N3 | 119.71 (13) |
| O1—C7—H7C | 109.5 | C22—C17—N3 | 119.65 (13) |
| H7A—C7—H7C | 109.5 | C17—C18—C19 | 119.26 (14) |
| H7B—C7—H7C | 109.5 | C17—C18—H18 | 120.4 |
| O2—C8—H8A | 109.5 | C19—C18—H18 | 120.4 |
| O2—C8—H8B | 109.5 | C20—C19—C18 | 120.23 (15) |
| H8A—C8—H8B | 109.5 | C20—C19—H19 | 119.9 |
| O2—C8—H8C | 109.5 | C18—C19—H19 | 119.9 |
| H8A—C8—H8C | 109.5 | C19—C20—C21 | 120.08 (16) |
| H8B—C8—H8C | 109.5 | C19—C20—H20 | 120 |
| N2—C9—C12 | 109.01 (10) | C21—C20—H20 | 120 |
| N2—C9—C4 | 111.33 (11) | C20—C21—C22 | 120.35 (16) |
| C12—C9—C4 | 112.06 (10) | C20—C21—H21 | 119.8 |
| N2—C9—H9 | 108.1 | C22—C21—H21 | 119.8 |
| C12—C9—H9 | 108.1 | C17—C22—C21 | 119.46 (16) |
| C4—C9—H9 | 108.1 | C17—C22—H22 | 120.3 |
| N2—C10—N3 | 116.24 (12) | C21—C22—H22 | 120.3 |
| N2—C10—S | 121.69 (10) | | |
| C7—O1—C1—C6 | 1.9 (2) | C17—N3—C10—S | 5.02 (18) |
| C7—O1—C1—C2 | −177.74 (17) | C10—N3—C11—C12 | −16.1 (2) |
| C8—O2—C2—C3 | −1.2 (2) | C17—N3—C11—C12 | 172.90 (12) |
| C8—O2—C2—C1 | 178.20 (15) | C10—N3—C11—C16 | 161.79 (14) |

| | | | |
|---|---|---|---|
| O1—C1—C2—O2 | 2.0 (2) | C17—N3—C11—C16 | −9.19 (19) |
| C6—C1—C2—O2 | −177.72 (13) | N3—C11—C12—C13 | 176.31 (13) |
| O1—C1—C2—C3 | −178.59 (14) | C16—C11—C12—C13 | −1.4 (2) |
| C6—C1—C2—C3 | 1.7 (2) | N3—C11—C12—C9 | −2.9 (2) |
| O2—C2—C3—C4 | 179.72 (14) | C16—C11—C12—C9 | 179.39 (14) |
| C1—C2—C3—C4 | 0.3 (2) | N2—C9—C12—C11 | 21.97 (17) |
| C2—C3—C4—C5 | −1.5 (2) | C4—C9—C12—C11 | −101.72 (14) |
| C2—C3—C4—C9 | −178.99 (13) | N2—C9—C12—C13 | −157.29 (12) |
| C3—C4—C5—C6 | 0.75 (19) | C4—C9—C12—C13 | 79.02 (15) |
| C9—C4—C5—C6 | 178.01 (12) | C14—O6—C13—O5 | 11.5 (5) |
| C3—C4—C5—N1 | −177.98 (12) | C14—O6—C13—O5' | −21.1 (5) |
| C9—C4—C5—N1 | −0.71 (19) | C14—O6—C13—C12 | 176.21 (15) |
| O3—N1—C5—C4 | −35.34 (19) | C11—C12—C13—O5 | −4.6 (5) |
| O4—N1—C5—C4 | 144.93 (13) | C9—C12—C13—O5 | 174.6 (5) |
| O3—N1—C5—C6 | 145.85 (14) | C11—C12—C13—O5' | 29.9 (6) |
| O4—N1—C5—C6 | −33.89 (17) | C9—C12—C13—O5' | −150.8 (5) |
| O1—C1—C6—C5 | 177.86 (14) | C11—C12—C13—O6 | −167.62 (14) |
| C2—C1—C6—C5 | −2.5 (2) | C9—C12—C13—O6 | 11.62 (19) |
| C4—C5—C6—C1 | 1.3 (2) | C13—O6—C14—C15 | 170.88 (19) |
| N1—C5—C6—C1 | −179.94 (12) | C10—N3—C17—C18 | 97.45 (16) |
| C10—N2—C9—C12 | −27.44 (18) | C11—N3—C17—C18 | −91.28 (17) |
| C10—N2—C9—C4 | 96.69 (15) | C10—N3—C17—C22 | −84.14 (19) |
| C5—C4—C9—N2 | 108.95 (13) | C11—N3—C17—C22 | 87.13 (18) |
| C3—C4—C9—N2 | −73.85 (14) | C22—C17—C18—C19 | 0.2 (2) |
| C5—C4—C9—C12 | −128.66 (13) | N3—C17—C18—C19 | 178.56 (14) |
| C3—C4—C9—C12 | 48.54 (16) | C17—C18—C19—C20 | −0.1 (3) |
| C9—N2—C10—N3 | 11.6 (2) | C18—C19—C20—C21 | 0.1 (3) |
| C9—N2—C10—S | −170.21 (11) | C19—C20—C21—C22 | −0.1 (3) |
| C11—N3—C10—N2 | 12.12 (19) | C18—C17—C22—C21 | −0.2 (3) |
| C17—N3—C10—N2 | −176.78 (12) | N3—C17—C22—C21 | −178.56 (17) |
| C11—N3—C10—S | −166.08 (10) | C20—C21—C22—C17 | 0.1 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···S$^{i}$ | 0.86 | 2.61 | 3.4505 (11) | 167 |
| C8—H8B···O3$^{ii}$ | 0.96 | 2.56 | 3.400 (2) | 146 |

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

**Fig. 1**

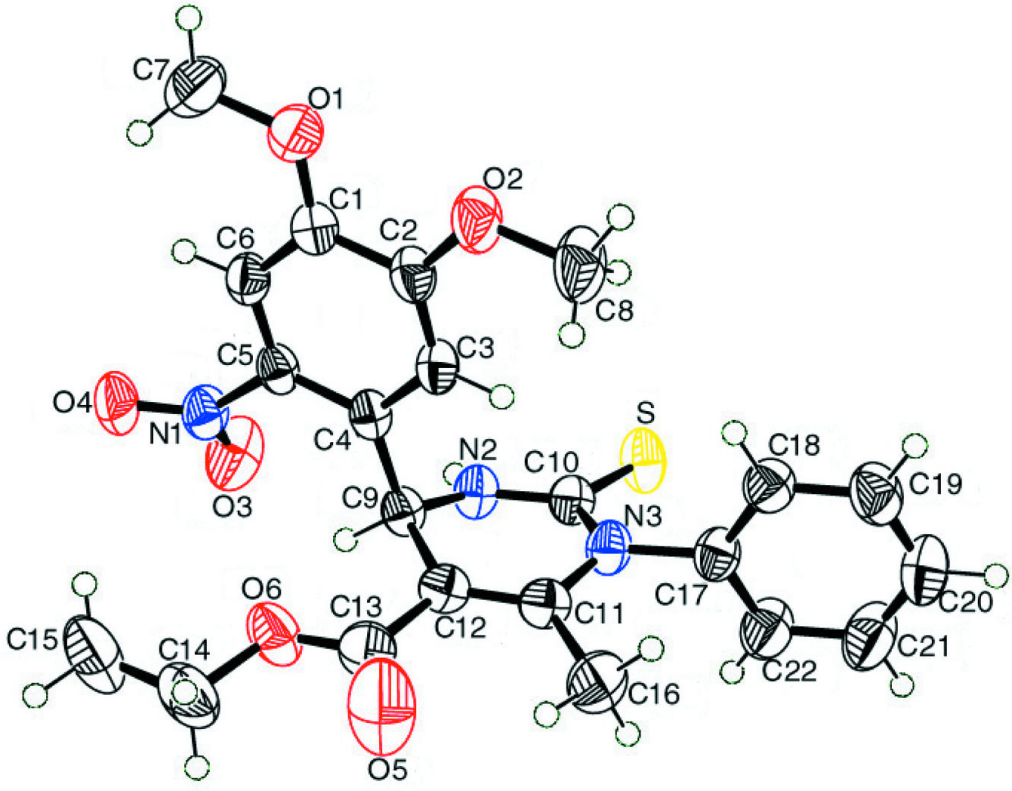
C7
O1
C1
O2
C6
C2
C8
C5
C3
O4
N1
C4
S
N2
C10
C18
C19
O3
C9
N3
O6
C13
C12
C11
C17
C20
C15
C14
C21
C22
C16
O5

Fig. 2