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

# Diethyl 2-{[(5-oxo-5*H*-thiochromeno-[2,3-*b*]pyridin-7-yl)amino]methylidene}-propanedioate

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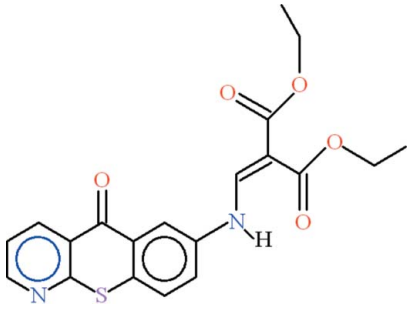
Received 17 April 2011; accepted 29 April 2011

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.054; $wR$ factor = 0.130; data-to-parameter ratio = 12.3.

In the title compound, $C_{15}H_{14}O_2S$, the three fused rings are roughly coplanar, the largest deviation from the mean plane being 0.1285 (13) Å for the S atom. An intramolecular N—H···O hydrogen bond generates an *S*6 ring. In the crystal, intermolecular C—H···O hydrogen bonds form $R_2^2(14)$, $R_2^2(13)$ and $R_3^2(17)$ ring motifs, building a layer parallel to (100).

## Related literature

For related structures, see: Khan *et al.* (2008*a*,*b*); Lokaj *et al.* (1994); Lynch & McClenaghan (2003); For graph-set notation, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



## Experimental

### Crystal data

$C_{20}H_{18}N_2O_5S$
$M_r$ = 398.42
Monoclinic, $C2/c$
$a$ = 13.8013 (7) Å
$b$ = 7.5180 (3) Å
$c$ = 36.2743 (15) Å
$\beta$ = 94.330 (3)°
$V$ = 3753.0 (3) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.21 mm$^{-1}$
$T$ = 296 K
0.35 × 0.18 × 0.15 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.968, $T_{max}$ = 0.985
13274 measured reflections
3314 independent reflections
1776 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.077

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.130
$S$ = 0.97
3314 reflections
269 parameters
10 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O3 | 0.86 | 2.06 | 2.683 (3) | 129 |
| C11—H11···O5$^{i}$ | 0.93 | 2.49 | 3.403 (4) | 167 |
| C12—H12···O1$^{i}$ | 0.93 | 2.45 | 3.322 (4) | 157 |
| C17*A*—H17*C*···O3$^{ii}$ | 0.96 | 2.58 | 3.516 (8) | 165 |
| C19*A*—H19*B*···O3$^{iii}$ | 0.97 | 2.47 | 3.231 (11) | 135 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009)'; software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Ex-Vice Chancellor, University of Sargodha, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2678).

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# supporting information

*Acta Cryst.* (2011). E**67**, o1348 [doi:10.1107/S1600536811016291]

# Diethyl 2-{[(5-oxo-5*H*-thiochromeno[2,3-*b*]pyridin-7-yl)amino]methylidene}propanedioate

**Muhammad Naeem Khan, M. Nawaz Tahir, Misbahul Ain Khan, Munawar Ali Munawar and Abdul Qayyum Ather**

## S1. Comment

We reported the crystal structures of (II) *i.e.* 7-nitro-5*H*-thiochromeno[2,3-*b*]pyridin-5-one (Khan *et al.*, 2008*a*) and (III) *i.e.* 5*H*-thiochromeno[2,3-*b*]pyridin-5-one (Khan *et al.*, 2008*b*). The title compound (Fig. 1) is in continuation of our work to synthesize the derivatives of 5*H*-thiochromeno[2,3-*b*]pyridin-5-one. The crystal structures of (IV) *i.e.*, diethyl 2-(2,3-diphenylquinoxalin-6-ylaminomethylene)malonate (Lokaj *et al.*, 1994) and (V) *i.e.*, diethyl (4-*tert*-butyl-1,3-thiazol-2-ylaminomethylene)malonate (Lynch & McClenaghan, 2003) have been published. Both contain the diethyl (aminomethylidene)propanedioate moiety which is also present in (I).

In (I), the central heterocyclic ring A (C1/C2/C6/S1/C7/C8), the pyridinic group B (C2/C3/C4/C5/N1/C6) and benzene ring C (C7—C12) are planar with r. m. s. deviation of 0.0388, 0.0053 and 0.0049 Å, respectively. The carbonyl O-atom is at a distance of 0.1532 (35) Å from its parent ring A. The dihedral angle between A/B, A/C and B/C is 3.68 (12), 3.50 (9) and 7.19 (12)°, respectively. A strong intramolecular H-bonding of N—H···O type completes an S(6) (Etter *et al.*, 1990; Bernstein *et al.*, 1995) ring motif. Intermolecular C—H···O hydrogen bonds complete $R_2^2(14)$, $R_2^2(13)$ and $R_3^2(17)$ ring motifs building a layer parallel to the (1 0 0) plane (Table 1, Fig 2).

## S2. Experimental

Diethyl (ethoxymethylene)malonate (0.473 g, 2.1 mmol), 7-amino-5*H*-thiochromeno[2,3-*b*]pyridin-5-one (500 mg, 2.1 mmol) and 8.0 ml of ethyl alcohol was heated under reflux on water bath for 3 h. Completion of reaction was monitored by TLC. The product precipitated by the addition of n-hexane was filtered, washed, dried and recrystallized from chloroform to give the yellow needles of (I).

## S3. Refinement

The diethyl groups are disordered over two set of sites with occupancy ratio of 0.75:0.25 and 0.53:0.47. The disordered poitions were refined using O-C and C-C bond restraints to maintain chemically reasonable geometry. The disordered groups were refined anistropically with however equal thermal parameters for the C-atoms.

The H-atoms were positioned geometrically (N—H = 0.86, C–H = 0.93–0.97 Å) and treated as riding with $U_{iso}$(H) = $xU_{eq}$(C, O), where $x$ = 1.5 for methyl and $x$ = 1.2 for aryl H-atoms.

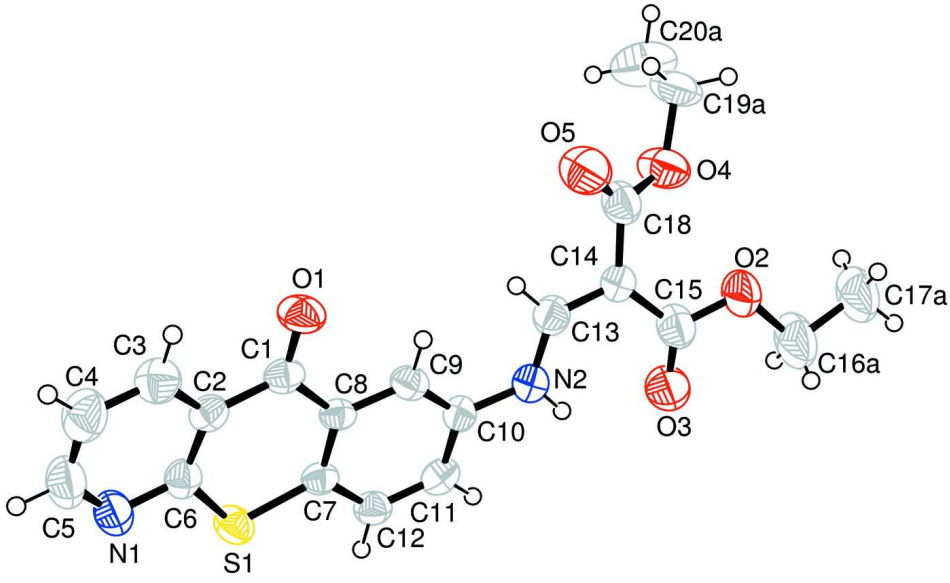


**Figure 1**

View of the title compound with the atom numbering scheme. The thermal displacements are drawn at the 50% probability level. H-atoms are shown as small spheres of arbitrary radii. The disordered miniority ethyl groups are omitted for clarity.

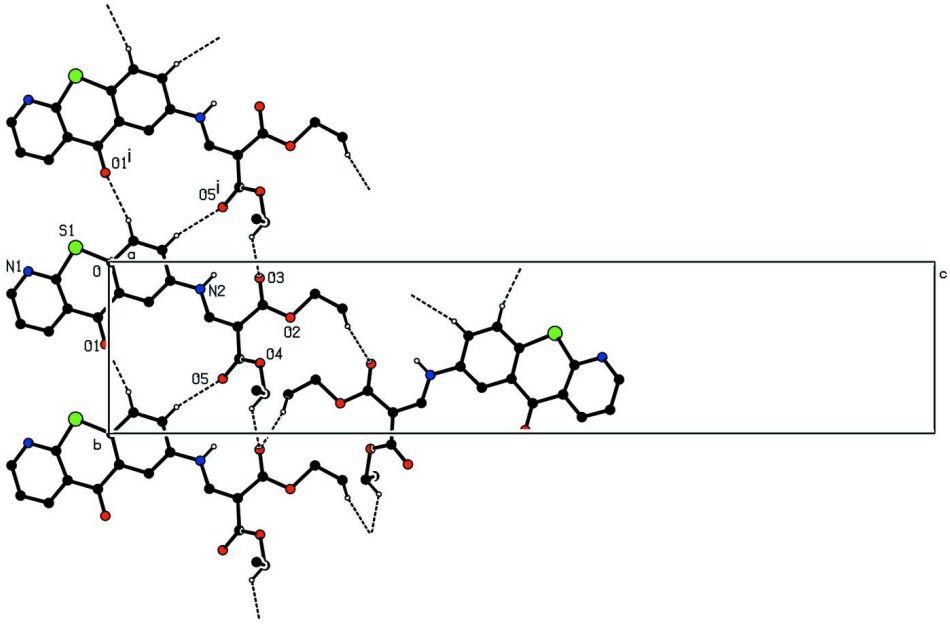


**Figure 2**

Partial packing view showing the formation of different ring motifs through C-H···O hydrogen bonds. Only the major component of the disordered moieties are represented and H atoms not involved in hydrogen bondings have been omitted for clarity. [Symmetry code: (i) *x*, *y*-1, *z*.]

**Diethyl 2-{[(5-oxo-5*H*-thiochromeno[2,3-*b*]pyridin-7- yl)amino]methylidene}propanedioate**

*Crystal data*

$C_{20}H_{18}N_2O_5S$
$M_r$ = 398.42
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 13.8013 (7) Å
*b* = 7.5180 (3) Å
*c* = 36.2743 (15) Å
*β* = 94.330 (3)°
*V* = 3753.0 (3) Å$^3$
*Z* = 8
*F*(000) = 1664
$D_x$ = 1.410 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1789 reflections
*θ* = 2.3–25.3°
*μ* = 0.21 mm$^{-1}$
*T* = 296 K
Needle, yellow
0.35 × 0.18 × 0.15 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm$^{-1}$
*ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.968, $T_{max}$ = 0.985
13274 measured reflections
3314 independent reflections
1776 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.077
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.3°
*h* = −16→16
*k* = 0→8
*l* = 0→42

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.130
*S* = 0.97
3314 reflections
269 parameters
10 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

*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.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.10364 (6) | −0.08366 (11) | −0.04024 (2) | 0.0529 (3) | |
| O1 | 0.11162 (17) | 0.4807 (3) | −0.00428 (6) | 0.0604 (7) | |
| O2 | 0.3194 (2) | 0.3243 (3) | 0.21973 (6) | 0.0911 (9) | |
| C16A | 0.3217 (7) | 0.1867 (9) | 0.24963 (15) | 0.1062 (19) | 0.75 |

| | | | | | |
|---|---|---|---|---|---|
| H16A | 0.2563 | 0.1492 | 0.2540 | 0.127* | 0.75 |
| H16B | 0.3585 | 0.0834 | 0.2428 | 0.127* | 0.75 |
| C17A | 0.3688 (6) | 0.2726 (9) | 0.28278 (13) | 0.1062 (19) | 0.75 |
| H17A | 0.4333 | 0.3086 | 0.2780 | 0.159* | 0.75 |
| H17B | 0.3718 | 0.1901 | 0.3030 | 0.159* | 0.75 |
| H17C | 0.3318 | 0.3751 | 0.2890 | 0.159* | 0.75 |
| C16B | 0.2928 (14) | 0.259 (3) | 0.2569 (5) | 0.122 (7) | 0.25 |
| H16C | 0.2441 | 0.1659 | 0.2556 | 0.147* | 0.25 |
| H16D | 0.2762 | 0.3534 | 0.2736 | 0.147* | 0.25 |
| C17B | 0.3965 (14) | 0.192 (3) | 0.2636 (8) | 0.122 (7) | 0.25 |
| H17D | 0.4131 | 0.1221 | 0.2429 | 0.184* | 0.25 |
| H17E | 0.4019 | 0.1198 | 0.2855 | 0.184* | 0.25 |
| H17F | 0.4401 | 0.2912 | 0.2668 | 0.184* | 0.25 |
| O3 | 0.2852 (2) | 0.0944 (4) | 0.18250 (6) | 0.0853 (9) | |
| O4 | 0.40074 (19) | 0.5902 (3) | 0.18304 (7) | 0.0793 (8) | |
| C19A | 0.4388 (7) | 0.7695 (14) | 0.1889 (4) | 0.105 (3) | 0.53 |
| H19A | 0.4366 | 0.8060 | 0.2145 | 0.126* | 0.53 |
| H19B | 0.4031 | 0.8548 | 0.1730 | 0.126* | 0.53 |
| C20A | 0.5401 (7) | 0.7499 (13) | 0.1787 (3) | 0.105 (3) | 0.53 |
| H20A | 0.5726 | 0.6611 | 0.1941 | 0.157* | 0.53 |
| H20B | 0.5734 | 0.8614 | 0.1822 | 0.157* | 0.53 |
| H20C | 0.5399 | 0.7146 | 0.1533 | 0.157* | 0.53 |
| C19B | 0.4500 (8) | 0.7644 (17) | 0.1791 (5) | 0.108 (4) | 0.47 |
| H19C | 0.4098 | 0.8599 | 0.1874 | 0.130* | 0.47 |
| H19D | 0.4615 | 0.7854 | 0.1534 | 0.130* | 0.47 |
| C20B | 0.5450 (7) | 0.7572 (16) | 0.2024 (3) | 0.108 (4) | 0.47 |
| H20D | 0.5325 | 0.7451 | 0.2280 | 0.163* | 0.47 |
| H20E | 0.5808 | 0.8647 | 0.1990 | 0.163* | 0.47 |
| H20F | 0.5822 | 0.6571 | 0.1950 | 0.163* | 0.47 |
| O5 | 0.2939 (2) | 0.6807 (4) | 0.13788 (7) | 0.0874 (9) | |
| N1 | 0.0155 (2) | 0.0557 (4) | −0.09743 (7) | 0.0591 (8) | |
| N2 | 0.23819 (19) | 0.1593 (4) | 0.11064 (7) | 0.0544 (8) | |
| H2 | 0.2432 | 0.0765 | 0.1270 | 0.065* | |
| C1 | 0.1051 (2) | 0.3242 (5) | −0.01327 (8) | 0.0440 (8) | |
| C2 | 0.0601 (2) | 0.2734 (4) | −0.05003 (8) | 0.0446 (8) | |
| C3 | 0.0182 (2) | 0.4052 (5) | −0.07279 (9) | 0.0618 (10) | |
| H3 | 0.0195 | 0.5229 | −0.0649 | 0.074* | |
| C4 | −0.0251 (3) | 0.3623 (6) | −0.10695 (10) | 0.0737 (12) | |
| H4 | −0.0542 | 0.4491 | −0.1223 | 0.088* | |
| C5 | −0.0242 (3) | 0.1861 (6) | −0.11782 (9) | 0.0719 (11) | |
| H5 | −0.0533 | 0.1574 | −0.1410 | 0.086* | |
| C6 | 0.0558 (2) | 0.1015 (4) | −0.06379 (8) | 0.0460 (8) | |
| C7 | 0.1410 (2) | 0.0021 (4) | 0.00313 (8) | 0.0407 (8) | |
| C8 | 0.1395 (2) | 0.1820 (4) | 0.01247 (8) | 0.0390 (8) | |
| C9 | 0.1711 (2) | 0.2333 (4) | 0.04837 (8) | 0.0452 (8) | |
| H9 | 0.1691 | 0.3528 | 0.0549 | 0.054* | |
| C10 | 0.2052 (2) | 0.1102 (5) | 0.07425 (8) | 0.0448 (8) | |
| C11 | 0.2065 (2) | −0.0693 (4) | 0.06457 (8) | 0.0507 (9) | |

| | | | | |
|---|---|---|---|---|
| H11 | 0.2294 | −0.1534 | 0.0819 | 0.061* |
| C12 | 0.1743 (2) | −0.1219 (4) | 0.02973 (8) | 0.0468 (8) |
| H12 | 0.1746 | −0.2421 | 0.0237 | 0.056* |
| C13 | 0.2620 (2) | 0.3227 (5) | 0.12156 (8) | 0.0525 (9) |
| H13 | 0.2555 | 0.4106 | 0.1035 | 0.063* |
| C14 | 0.2949 (2) | 0.3776 (4) | 0.15601 (8) | 0.0503 (9) |
| C15 | 0.3008 (3) | 0.2515 (6) | 0.18640 (9) | 0.0634 (10) |
| C18 | 0.3268 (3) | 0.5646 (5) | 0.15800 (9) | 0.0608 (10) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0609 (6) | 0.0445 (6) | 0.0529 (5) | 0.0014 (5) | 0.0003 (4) | −0.0085 (4) |
| O1 | 0.0728 (18) | 0.0357 (15) | 0.0714 (15) | 0.0011 (13) | −0.0036 (12) | −0.0025 (12) |
| O2 | 0.150 (3) | 0.077 (2) | 0.0444 (14) | −0.0195 (18) | −0.0106 (15) | 0.0048 (13) |
| C16A | 0.175 (6) | 0.088 (4) | 0.050 (2) | 0.010 (4) | −0.023 (3) | 0.004 (2) |
| C17A | 0.175 (6) | 0.088 (4) | 0.050 (2) | 0.010 (4) | −0.023 (3) | 0.004 (2) |
| C16B | 0.20 (2) | 0.056 (13) | 0.107 (13) | −0.034 (13) | 0.004 (15) | 0.009 (9) |
| C17B | 0.20 (2) | 0.056 (13) | 0.107 (13) | −0.034 (13) | 0.004 (15) | 0.009 (9) |
| O3 | 0.135 (3) | 0.0529 (19) | 0.0666 (16) | −0.0019 (18) | 0.0001 (15) | 0.0044 (14) |
| O4 | 0.085 (2) | 0.0608 (19) | 0.0880 (18) | −0.0109 (15) | −0.0233 (15) | −0.0001 (14) |
| C19A | 0.127 (9) | 0.055 (5) | 0.129 (7) | −0.022 (5) | −0.010 (6) | 0.006 (4) |
| C20A | 0.127 (9) | 0.055 (5) | 0.129 (7) | −0.022 (5) | −0.010 (6) | 0.006 (4) |
| C19B | 0.080 (8) | 0.102 (8) | 0.138 (8) | −0.009 (5) | −0.026 (5) | −0.044 (6) |
| C20B | 0.080 (8) | 0.102 (8) | 0.138 (8) | −0.009 (5) | −0.026 (5) | −0.044 (6) |
| O5 | 0.115 (2) | 0.063 (2) | 0.0792 (18) | 0.0021 (16) | −0.0280 (16) | 0.0106 (15) |
| N1 | 0.058 (2) | 0.072 (2) | 0.0474 (17) | −0.0035 (16) | 0.0008 (14) | −0.0064 (15) |
| N2 | 0.065 (2) | 0.053 (2) | 0.0452 (16) | −0.0068 (16) | 0.0044 (13) | 0.0012 (13) |
| C1 | 0.034 (2) | 0.044 (2) | 0.054 (2) | −0.0031 (17) | 0.0088 (15) | 0.0006 (17) |
| C2 | 0.038 (2) | 0.046 (2) | 0.0496 (19) | 0.0001 (16) | 0.0032 (15) | 0.0073 (16) |
| C3 | 0.060 (2) | 0.059 (3) | 0.065 (2) | 0.003 (2) | 0.0019 (19) | 0.003 (2) |
| C4 | 0.075 (3) | 0.081 (3) | 0.063 (3) | 0.007 (2) | −0.008 (2) | 0.016 (2) |
| C5 | 0.075 (3) | 0.091 (4) | 0.049 (2) | −0.001 (3) | −0.0056 (19) | 0.000 (2) |
| C6 | 0.039 (2) | 0.054 (2) | 0.0446 (18) | −0.0004 (17) | 0.0057 (15) | 0.0009 (17) |
| C7 | 0.035 (2) | 0.039 (2) | 0.0479 (18) | −0.0044 (16) | 0.0062 (15) | 0.0002 (15) |
| C8 | 0.0351 (19) | 0.037 (2) | 0.0452 (18) | −0.0044 (15) | 0.0061 (14) | −0.0006 (15) |
| C9 | 0.044 (2) | 0.042 (2) | 0.0507 (19) | −0.0033 (16) | 0.0073 (16) | −0.0075 (16) |
| C10 | 0.047 (2) | 0.050 (2) | 0.0386 (17) | −0.0050 (17) | 0.0066 (14) | 0.0010 (16) |
| C11 | 0.053 (2) | 0.046 (2) | 0.053 (2) | −0.0014 (18) | 0.0071 (17) | 0.0101 (17) |
| C12 | 0.051 (2) | 0.037 (2) | 0.052 (2) | −0.0022 (16) | 0.0077 (16) | −0.0007 (15) |
| C13 | 0.051 (2) | 0.060 (3) | 0.047 (2) | −0.0038 (19) | 0.0060 (16) | 0.0018 (17) |
| C14 | 0.059 (2) | 0.049 (2) | 0.0430 (18) | −0.0047 (18) | 0.0061 (16) | 0.0007 (16) |
| C15 | 0.072 (3) | 0.066 (3) | 0.051 (2) | 0.003 (2) | −0.0017 (19) | −0.005 (2) |
| C18 | 0.067 (3) | 0.064 (3) | 0.050 (2) | 0.001 (2) | −0.0044 (19) | −0.0032 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C6 | 1.737 (3) | C20B—H20D | 0.9600 |
| S1—C7 | 1.742 (3) | C20B—H20E | 0.9600 |
| O1—C1 | 1.222 (3) | C20B—H20F | 0.9600 |
| O2—C15 | 1.334 (4) | O5—C18 | 1.204 (4) |
| O2—C16A | 1.497 (6) | N1—C5 | 1.321 (4) |
| O2—C16B | 1.506 (14) | N1—C6 | 1.347 (4) |
| C16A—C17A | 1.472 (7) | N2—C13 | 1.324 (4) |
| C16A—H16A | 0.9700 | N2—C10 | 1.413 (4) |
| C16A—H16B | 0.9700 | N2—H2 | 0.8600 |
| C17A—H17A | 0.9600 | C1—C8 | 1.474 (4) |
| C17A—H17B | 0.9600 | C1—C2 | 1.478 (4) |
| C17A—H17C | 0.9600 | C2—C6 | 1.385 (4) |
| C16B—C17B | 1.520 (10) | C2—C3 | 1.388 (4) |
| C16B—H16C | 0.9700 | C3—C4 | 1.373 (4) |
| C16B—H16D | 0.9700 | C3—H3 | 0.9300 |
| C17B—H17D | 0.9600 | C4—C5 | 1.382 (5) |
| C17B—H17E | 0.9600 | C4—H4 | 0.9300 |
| C17B—H17F | 0.9600 | C5—H5 | 0.9300 |
| O3—C15 | 1.206 (4) | C7—C12 | 1.395 (4) |
| O4—C18 | 1.328 (4) | C7—C8 | 1.395 (4) |
| O4—C19A | 1.457 (10) | C8—C9 | 1.396 (4) |
| O4—C19B | 1.487 (12) | C9—C10 | 1.375 (4) |
| C19A—C20A | 1.480 (9) | C9—H9 | 0.9300 |
| C19A—H19A | 0.9700 | C10—C11 | 1.395 (4) |
| C19A—H19B | 0.9700 | C11—C12 | 1.366 (4) |
| C20A—H20A | 0.9600 | C11—H11 | 0.9300 |
| C20A—H20B | 0.9600 | C12—H12 | 0.9300 |
| C20A—H20C | 0.9600 | C13—C14 | 1.361 (4) |
| C19B—C20B | 1.506 (10) | C13—H13 | 0.9300 |
| C19B—H19C | 0.9700 | C14—C15 | 1.452 (4) |
| C19B—H19D | 0.9700 | C14—C18 | 1.473 (4) |
| | | | |
| C6—S1—C7 | 102.90 (15) | C6—C2—C1 | 124.7 (3) |
| C15—O2—C16A | 111.4 (4) | C3—C2—C1 | 118.7 (3) |
| C15—O2—C16B | 129.4 (11) | C4—C3—C2 | 120.2 (3) |
| C16A—O2—C16B | 28.4 (8) | C4—C3—H3 | 119.9 |
| C17A—C16A—O2 | 105.8 (5) | C2—C3—H3 | 119.9 |
| C17A—C16A—H16A | 110.6 | C3—C4—C5 | 118.0 (4) |
| O2—C16A—H16A | 110.6 | C3—C4—H4 | 121.0 |
| C17A—C16A—H16B | 110.6 | C5—C4—H4 | 121.0 |
| O2—C16A—H16B | 110.6 | N1—C5—C4 | 124.3 (4) |
| H16A—C16A—H16B | 108.7 | N1—C5—H5 | 117.9 |
| O2—C16B—C17B | 87.8 (14) | C4—C5—H5 | 117.9 |
| O2—C16B—H16C | 114.0 | N1—C6—C2 | 124.6 (3) |
| C17B—C16B—H16C | 114.0 | N1—C6—S1 | 110.9 (3) |
| O2—C16B—H16D | 114.0 | C2—C6—S1 | 124.5 (2) |

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|---|---|---|---|
| C17B—C16B—H16D | 114.0 | C12—C7—C8 | 119.4 (3) |
| H16C—C16B—H16D | 111.2 | C12—C7—S1 | 115.9 (2) |
| C16B—C17B—H17D | 109.5 | C8—C7—S1 | 124.7 (2) |
| C16B—C17B—H17E | 109.5 | C7—C8—C9 | 119.0 (3) |
| H17D—C17B—H17E | 109.5 | C7—C8—C1 | 124.0 (3) |
| C16B—C17B—H17F | 109.5 | C9—C8—C1 | 117.0 (3) |
| H17D—C17B—H17F | 109.5 | C10—C9—C8 | 121.2 (3) |
| H17E—C17B—H17F | 109.5 | C10—C9—H9 | 119.4 |
| C18—O4—C19A | 118.9 (6) | C8—C9—H9 | 119.4 |
| C18—O4—C19B | 113.3 (6) | C9—C10—C11 | 119.3 (3) |
| C19A—O4—C19B | 15.6 (11) | C9—C10—N2 | 122.1 (3) |
| O4—C19A—C20A | 102.0 (8) | C11—C10—N2 | 118.6 (3) |
| O4—C19A—H19A | 111.4 | C12—C11—C10 | 120.2 (3) |
| C20A—C19A—H19A | 111.4 | C12—C11—H11 | 119.9 |
| O4—C19A—H19B | 111.4 | C10—C11—H11 | 119.9 |
| C20A—C19A—H19B | 111.4 | C11—C12—C7 | 120.9 (3) |
| H19A—C19A—H19B | 109.2 | C11—C12—H12 | 119.5 |
| O4—C19B—C20B | 107.4 (10) | C7—C12—H12 | 119.5 |
| O4—C19B—H19C | 110.2 | N2—C13—C14 | 127.9 (3) |
| C20B—C19B—H19C | 110.2 | N2—C13—H13 | 116.1 |
| O4—C19B—H19D | 110.2 | C14—C13—H13 | 116.1 |
| C20B—C19B—H19D | 110.2 | C13—C14—C15 | 119.7 (3) |
| H19C—C19B—H19D | 108.5 | C13—C14—C18 | 114.3 (3) |
| C5—N1—C6 | 116.3 (3) | C15—C14—C18 | 125.9 (3) |
| C13—N2—C10 | 125.3 (3) | O3—C15—O2 | 121.9 (3) |
| C13—N2—H2 | 117.4 | O3—C15—C14 | 123.4 (3) |
| C10—N2—H2 | 117.4 | O2—C15—C14 | 114.6 (4) |
| O1—C1—C8 | 121.0 (3) | O5—C18—O4 | 123.0 (4) |
| O1—C1—C2 | 120.4 (3) | O5—C18—C14 | 124.4 (4) |
| C8—C1—C2 | 118.5 (3) | O4—C18—C14 | 112.5 (3) |
| C6—C2—C3 | 116.6 (3) | | |

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| C15—O2—C16A—C17A | 164.6 (6) | O1—C1—C8—C9 | 5.8 (4) |
| C16B—O2—C16A—C17A | −60.2 (19) | C2—C1—C8—C9 | −172.7 (2) |
| C15—O2—C16B—C17B | 101.0 (17) | C7—C8—C9—C10 | 1.2 (4) |
| C16A—O2—C16B—C17B | 42.9 (14) | C1—C8—C9—C10 | −179.5 (3) |
| C18—O4—C19A—C20A | −118.4 (9) | C8—C9—C10—C11 | −1.1 (4) |
| C19B—O4—C19A—C20A | −46 (3) | C8—C9—C10—N2 | 179.5 (3) |
| C18—O4—C19B—C20B | −166.4 (9) | C13—N2—C10—C9 | −16.8 (5) |
| C19A—O4—C19B—C20B | 79 (3) | C13—N2—C10—C11 | 163.8 (3) |
| O1—C1—C2—C6 | 174.9 (3) | C9—C10—C11—C12 | 0.0 (4) |
| C8—C1—C2—C6 | −6.5 (4) | N2—C10—C11—C12 | 179.4 (3) |
| O1—C1—C2—C3 | −5.4 (4) | C10—C11—C12—C7 | 1.0 (4) |
| C8—C1—C2—C3 | 173.2 (3) | C8—C7—C12—C11 | −0.9 (4) |
| C6—C2—C3—C4 | 0.4 (5) | S1—C7—C12—C11 | 179.2 (2) |
| C1—C2—C3—C4 | −179.3 (3) | C10—N2—C13—C14 | −179.4 (3) |
| C2—C3—C4—C5 | −0.9 (5) | N2—C13—C14—C15 | −4.2 (5) |
| C6—N1—C5—C4 | 0.9 (5) | N2—C13—C14—C18 | 172.5 (3) |

| | | | |
|---|---|---|---|
| C3—C4—C5—N1 | 0.2 (6) | C16A—O2—C15—O3 | 2.2 (6) |
| C5—N1—C6—C2 | −1.5 (5) | C16B—O2—C15—O3 | −23.5 (11) |
| C5—N1—C6—S1 | 179.4 (2) | C16A—O2—C15—C14 | 178.4 (5) |
| C3—C2—C6—N1 | 0.9 (4) | C16B—O2—C15—C14 | 152.7 (10) |
| C1—C2—C6—N1 | −179.4 (3) | C13—C14—C15—O3 | 6.0 (5) |
| C3—C2—C6—S1 | 179.8 (2) | C18—C14—C15—O3 | −170.3 (4) |
| C1—C2—C6—S1 | −0.5 (4) | C13—C14—C15—O2 | −170.2 (3) |
| C7—S1—C6—N1 | −174.8 (2) | C18—C14—C15—O2 | 13.5 (5) |
| C7—S1—C6—C2 | 6.2 (3) | C19A—O4—C18—O5 | 5.6 (8) |
| C6—S1—C7—C12 | 173.8 (2) | C19B—O4—C18—O5 | −10.6 (8) |
| C6—S1—C7—C8 | −6.1 (3) | C19A—O4—C18—C14 | −178.4 (6) |
| C12—C7—C8—C9 | −0.2 (4) | C19B—O4—C18—C14 | 165.4 (7) |
| S1—C7—C8—C9 | 179.7 (2) | C13—C14—C18—O5 | 29.7 (5) |
| C12—C7—C8—C1 | −179.4 (3) | C15—C14—C18—O5 | −153.8 (4) |
| S1—C7—C8—C1 | 0.5 (4) | C13—C14—C18—O4 | −146.2 (3) |
| O1—C1—C8—C7 | −175.0 (3) | C15—C14—C18—O4 | 30.3 (5) |
| C2—C1—C8—C7 | 6.5 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O3 | 0.86 | 2.06 | 2.683 (3) | 129 |
| C11—H11···O5$^{i}$ | 0.93 | 2.49 | 3.403 (4) | 167 |
| C12—H12···O1$^{i}$ | 0.93 | 2.45 | 3.322 (4) | 157 |
| C17*A*—H17*C*···O3$^{ii}$ | 0.96 | 2.58 | 3.516 (8) | 165 |
| C19*A*—H19*B*···O3$^{iii}$ | 0.97 | 2.47 | 3.231 (11) | 135 |

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