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# Ethyl 2-acetylamino-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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**Key indicators**

Single-crystal X-ray study
$T$ = 120 K
Mean $\sigma$(C–C) = 0.002 Å
$R$ factor = 0.036
$wR$ factor = 0.091
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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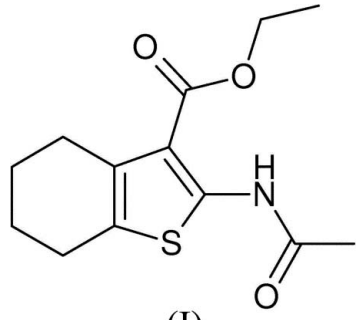
The geometrical parameters for the title compound, $C_{13}H_{17}NO_3S$, are normal. The planar molecular conformation is reinforced by an intramolecular N—H···O interaction.

## Comment

Thiophene derivatives are known to exhibit an array of biological effects, including analgesic and anti-inflammatory activities (Ramanathan & Namboothiri, 1978; Cannito *et al.*, 1990). As part of our own research in this area, the structure of the title compound, (I) (Fig. 1), is presented.



(I)

The geometric parameters for (I) are normal. The five-membered C1/C2/C6/C7/S1 ring is almost planar (r.m.s. deviation from the mean plane = 0.013 Å). The C1–C6 ring is in a half-chair conformation (Table 1), with atoms C1, C2, C5 and C6 almost co-planar (r.m.s. deviation = 0.003 Å) and atoms C3 and C4 displaced from this plane by −0.480 (3) and 0.277 (3) Å, respectively. An intramolecular N—H···O bond (Table 2) helps to establish the molecular conformation. Overall, the molecule of (I) is approximately planar.

The molecular packing for (I) comprises undulating sheets lying parallel to the $(10\bar{1})$ plane (Fig. 2). Within these sheets, the shortest intermolecular contacts are C—H···O interactions (Table 2).

## Experimental

Ethyl-2-amino-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate, (II), was prepared from cyclohexanone, sulfur and ethyl cyanoacetate by a one-pot thiolation-heterocyclization reaction (Gewald *et al.*, 1966). A mixture of (II) (3.5 g, 0.015 mol), acetic anhydride (14 ml) and zinc dust (0.883 g, 0.015 mol) was refluxed for 2 h. The reaction mixture was cooled to room temperature and the solid product was recovered. The crude product was dissolved in warm (318 K) methanol (35 ml) and filtered. The product was recrystallized from acetone to yield colourless crystals of (I) (yield 84.3%; m.p. 388 K). IR (KBr, cm$^{-1}$): 3436 and 3244 (—NH—), 2931 and 2873 (—CH—),

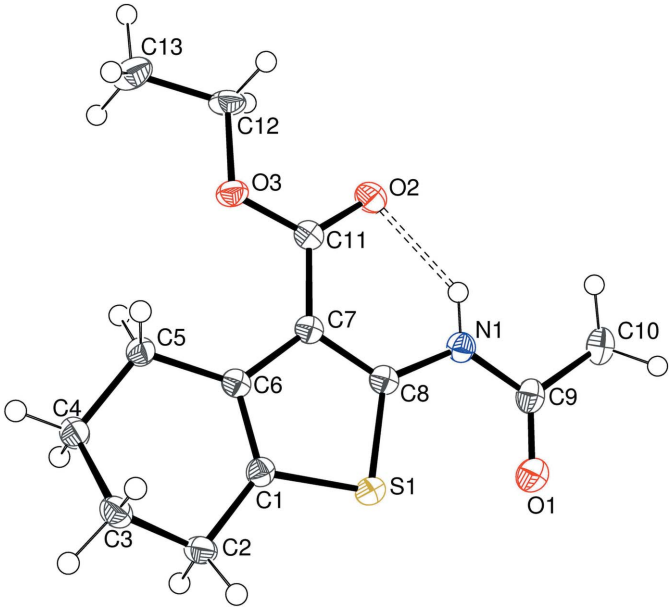



**Figure 1**
The molecular structure of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms. The intramolecular hydrogen bond is indicated by a dashed line.

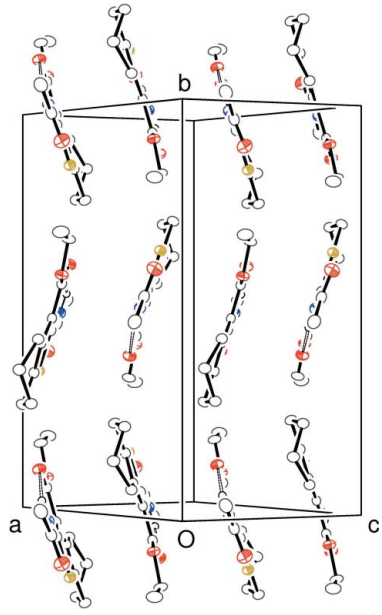



**Figure 2**
A view of the unit-cell contents of (I), with H atoms (except H1) omitted for clarity.

1666 and 1546 (C═O) and 1250 (C—O). Elemental analysis, found: C 58.18, H 6.32, N 5.16%; calculated: C58.40, H 6.41, N 5.24%.

*Crystal data*

$C_{13}H_{17}NO_3S$
$M_r$ = 267.34
Monoclinic, $P2_1/c$
$a$ = 10.2987 (4) Å
$b$ = 16.6174 (5) Å
$c$ = 7.8510 (3) Å
$\beta$ = 108.4381 (18)°
$V$ = 1274.63 (8) Å$^3$
$Z$ = 4
$D_x$ = 1.393 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.25 mm$^{-1}$
$T$ = 120 (2) K
Cut block, colourless
0.44 × 0.30 × 0.18 mm

*Data collection*

Nonius KappaCCD diffractometer
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min}$ = 0.897, $T_{max}$ = 0.958
16538 measured reflections
2917 independent reflections
2338 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.091
$S$ = 1.05
2917 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

**Table 1**
Selected torsion angles (°).

| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | 48.83 (16) | C4—C5—C6—C1 | −10.54 (19) |
| C2—C3—C4—C5 | −63.48 (17) | C5—C6—C1—C2 | −0.9 (2) |
| C3—C4—C5—C6 | 42.28 (18) | C6—C1—C2—C3 | −18.7 (2) |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O2 | 0.858 (18) | 1.979 (18) | 2.6798 (17) | 138.1 (16) |
| C2—H2$A$···O2$^{i}$ | 0.99 | 2.44 | 3.370 (2) | 157 |

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

The N-bound H atom was located in a difference map and its position was refined freely, with $U_{iso}$(H) = 1.2$U_{eq}$(N). The C-bound H atoms were placed in idealised locations (C—H = 0.98–0.99 Å) and refined as riding, with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C). The methyl groups were allowed to rotate about their local threefold axes to fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997) and *SORTAV* (Blessing, 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); software used to prepare material for publication: *SHELXL97*.

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# supporting information

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*Crystal data*

$C_{13}H_{17}NO_3S$
$M_r = 267.34$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.2987$ (4) Å
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$c = 7.8510$ (3) Å
$\beta = 108.4381$ (18)°
$V = 1274.63$ (8) Å$^3$
$Z = 4$
$F(000) = 568$
$D_x = 1.393$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3015 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.25$ mm$^{-1}$
$T = 120$ K
Cut block, colourless
0.44 × 0.30 × 0.18 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
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$T_{min} = 0.897$, $T_{max} = 0.958$
16538 measured reflections
2917 independent reflections
2338 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -13 \rightarrow 13$
$k = -21 \rightarrow 20$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.091$
$S = 1.05$
2917 reflections
168 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*Special details*

**Experimental**. IR (KBr, cm$^{-1}$): 3436 and 3244 (—NH—), 2931 and 2873 (—CH—), 1666 and 1546 (C═O) and 1250 (C—O). Elemental analysis, found: C 58.18, H 6.32, N 5.16%; calculated: C58.40, H 6.41, N 5.24%.

**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.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.42025 (15) | 0.34938 (9) | 0.5719 (2) | 0.0190 (3) |
| C2 | 0.36910 (16) | 0.27403 (9) | 0.4663 (2) | 0.0232 (3) |
| H2A | 0.3934 | 0.2266 | 0.5464 | 0.028* |
| H2B | 0.4126 | 0.2679 | 0.3712 | 0.028* |
| C3 | 0.21396 (16) | 0.27903 (9) | 0.3819 (2) | 0.0232 (3) |
| H3A | 0.1817 | 0.2347 | 0.2944 | 0.028* |
| H3B | 0.1696 | 0.2731 | 0.4762 | 0.028* |
| C4 | 0.17450 (16) | 0.35977 (9) | 0.2872 (2) | 0.0229 (3) |
| H4A | 0.0746 | 0.3605 | 0.2246 | 0.028* |
| H4B | 0.2214 | 0.3658 | 0.1956 | 0.028* |
| C5 | 0.21240 (15) | 0.43102 (9) | 0.41786 (19) | 0.0194 (3) |
| H5A | 0.2108 | 0.4812 | 0.3493 | 0.023* |
| H5B | 0.1433 | 0.4362 | 0.4808 | 0.023* |
| C6 | 0.35228 (15) | 0.42071 (9) | 0.55453 (19) | 0.0178 (3) |
| C7 | 0.43107 (14) | 0.48049 (9) | 0.67846 (19) | 0.0175 (3) |
| C8 | 0.55668 (15) | 0.45085 (9) | 0.78253 (19) | 0.0189 (3) |
| C9 | 0.78203 (16) | 0.46767 (10) | 1.0084 (2) | 0.0232 (3) |
| C10 | 0.86975 (16) | 0.52769 (10) | 1.1382 (2) | 0.0276 (4) |
| H10A | 0.8119 | 0.5710 | 1.1592 | 0.041* |
| H10B | 0.9375 | 0.5504 | 1.0878 | 0.041* |
| H10C | 0.9169 | 0.5008 | 1.2521 | 0.041* |
| C11 | 0.39415 (15) | 0.56423 (9) | 0.6996 (2) | 0.0191 (3) |
| C12 | 0.23372 (17) | 0.66948 (9) | 0.5971 (2) | 0.0261 (4) |
| H12A | 0.3078 | 0.7041 | 0.5829 | 0.031* |
| H12B | 0.2198 | 0.6825 | 0.7131 | 0.031* |
| C13 | 0.10391 (17) | 0.68309 (10) | 0.4450 (3) | 0.0323 (4) |
| H13A | 0.0775 | 0.7398 | 0.4423 | 0.048* |
| H13B | 0.0309 | 0.6494 | 0.4621 | 0.048* |
| H13C | 0.1186 | 0.6689 | 0.3313 | 0.048* |
| N1 | 0.65371 (13) | 0.49412 (8) | 0.91396 (17) | 0.0208 (3) |
| H1 | 0.6261 (17) | 0.5419 (11) | 0.925 (2) | 0.025* |
| O1 | 0.82149 (11) | 0.40043 (7) | 0.98601 (16) | 0.0315 (3) |
| O2 | 0.46870 (11) | 0.61218 (6) | 0.80471 (15) | 0.0253 (3) |

| | | | | |
|---|---|---|---|---|
| O3 | 0.26963 (11) | 0.58460 (6) | 0.59197 (14) | 0.0220 (2) |
| S1 | 0.58051 (4) | 0.35234 (2) | 0.73354 (5) | 0.02108 (12) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0201 (7) | 0.0197 (7) | 0.0179 (7) | 0.0006 (6) | 0.0067 (6) | 0.0014 (6) |
| C2 | 0.0301 (8) | 0.0172 (8) | 0.0221 (8) | 0.0028 (6) | 0.0077 (6) | −0.0015 (6) |
| C3 | 0.0293 (8) | 0.0183 (8) | 0.0211 (7) | −0.0028 (6) | 0.0068 (6) | −0.0020 (6) |
| C4 | 0.0257 (8) | 0.0202 (8) | 0.0205 (8) | −0.0008 (6) | 0.0038 (6) | −0.0008 (6) |
| C5 | 0.0199 (7) | 0.0177 (7) | 0.0195 (7) | 0.0006 (6) | 0.0048 (6) | 0.0009 (6) |
| C6 | 0.0213 (7) | 0.0178 (7) | 0.0161 (7) | −0.0009 (6) | 0.0087 (6) | 0.0012 (6) |
| C7 | 0.0187 (7) | 0.0185 (7) | 0.0170 (7) | −0.0008 (6) | 0.0082 (6) | 0.0010 (6) |
| C8 | 0.0214 (7) | 0.0193 (7) | 0.0180 (7) | −0.0010 (6) | 0.0092 (6) | 0.0016 (6) |
| C9 | 0.0212 (8) | 0.0291 (9) | 0.0199 (7) | −0.0011 (6) | 0.0072 (6) | 0.0039 (7) |
| C10 | 0.0228 (8) | 0.0335 (9) | 0.0232 (8) | −0.0022 (7) | 0.0025 (6) | 0.0028 (7) |
| C11 | 0.0200 (7) | 0.0196 (7) | 0.0194 (7) | −0.0020 (6) | 0.0088 (6) | 0.0003 (6) |
| C12 | 0.0299 (9) | 0.0138 (7) | 0.0353 (9) | 0.0028 (6) | 0.0114 (7) | −0.0025 (7) |
| C13 | 0.0251 (9) | 0.0219 (8) | 0.0483 (11) | 0.0033 (7) | 0.0094 (8) | 0.0038 (8) |
| N1 | 0.0194 (6) | 0.0211 (7) | 0.0213 (6) | −0.0016 (5) | 0.0056 (5) | −0.0002 (5) |
| O1 | 0.0251 (6) | 0.0326 (7) | 0.0322 (7) | 0.0046 (5) | 0.0024 (5) | −0.0019 (5) |
| O2 | 0.0264 (6) | 0.0203 (6) | 0.0274 (6) | −0.0029 (5) | 0.0057 (5) | −0.0048 (5) |
| O3 | 0.0213 (5) | 0.0155 (5) | 0.0279 (6) | 0.0020 (4) | 0.0061 (4) | −0.0008 (4) |
| S1 | 0.0205 (2) | 0.0197 (2) | 0.0226 (2) | 0.00291 (15) | 0.00611 (14) | 0.00109 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.361 (2) | C8—N1 | 1.3884 (19) |
| C1—C2 | 1.502 (2) | C8—S1 | 1.7170 (15) |
| C1—S1 | 1.7356 (15) | C9—O1 | 1.221 (2) |
| C2—C3 | 1.526 (2) | C9—N1 | 1.3674 (19) |
| C2—H2A | 0.990 | C9—C10 | 1.505 (2) |
| C2—H2B | 0.990 | C10—H10A | 0.980 |
| C3—C4 | 1.525 (2) | C10—H10B | 0.980 |
| C3—H3A | 0.990 | C10—H10C | 0.980 |
| C3—H3B | 0.990 | C11—O2 | 1.2266 (18) |
| C4—C5 | 1.534 (2) | C11—O3 | 1.3370 (18) |
| C4—H4A | 0.990 | C12—O3 | 1.4619 (17) |
| C4—H4B | 0.990 | C12—C13 | 1.502 (2) |
| C5—C6 | 1.509 (2) | C12—H12A | 0.990 |
| C5—H5A | 0.990 | C12—H12B | 0.990 |
| C5—H5B | 0.990 | C13—H13A | 0.980 |
| C6—C7 | 1.446 (2) | C13—H13B | 0.980 |
| C7—C8 | 1.384 (2) | C13—H13C | 0.980 |
| C7—C11 | 1.466 (2) | N1—H1 | 0.858 (18) |
| | | | |
| C6—C1—C2 | 126.24 (14) | C7—C8—N1 | 124.98 (14) |
| C6—C1—S1 | 112.86 (11) | C7—C8—S1 | 112.34 (11) |

| | | | |
|---|---|---|---|
| C2—C1—S1 | 120.90 (11) | N1—C8—S1 | 122.68 (11) |
| C1—C2—C3 | 109.31 (12) | O1—C9—N1 | 121.65 (15) |
| C1—C2—H2A | 109.8 | O1—C9—C10 | 123.24 (14) |
| C3—C2—H2A | 109.8 | N1—C9—C10 | 115.11 (14) |
| C1—C2—H2B | 109.8 | C9—C10—H10A | 109.5 |
| C3—C2—H2B | 109.8 | C9—C10—H10B | 109.5 |
| H2A—C2—H2B | 108.3 | H10A—C10—H10B | 109.5 |
| C4—C3—C2 | 109.91 (13) | C9—C10—H10C | 109.5 |
| C4—C3—H3A | 109.7 | H10A—C10—H10C | 109.5 |
| C2—C3—H3A | 109.7 | H10B—C10—H10C | 109.5 |
| C4—C3—H3B | 109.7 | O2—C11—O3 | 122.13 (14) |
| C2—C3—H3B | 109.7 | O2—C11—C7 | 124.28 (14) |
| H3A—C3—H3B | 108.2 | O3—C11—C7 | 113.59 (12) |
| C3—C4—C5 | 112.40 (12) | O3—C12—C13 | 107.03 (13) |
| C3—C4—H4A | 109.1 | O3—C12—H12A | 110.3 |
| C5—C4—H4A | 109.1 | C13—C12—H12A | 110.3 |
| C3—C4—H4B | 109.1 | O3—C12—H12B | 110.3 |
| C5—C4—H4B | 109.1 | C13—C12—H12B | 110.3 |
| H4A—C4—H4B | 107.9 | H12A—C12—H12B | 108.6 |
| C6—C5—C4 | 111.81 (12) | C12—C13—H13A | 109.5 |
| C6—C5—H5A | 109.3 | C12—C13—H13B | 109.5 |
| C4—C5—H5A | 109.3 | H13A—C13—H13B | 109.5 |
| C6—C5—H5B | 109.3 | C12—C13—H13C | 109.5 |
| C4—C5—H5B | 109.3 | H13A—C13—H13C | 109.5 |
| H5A—C5—H5B | 107.9 | H13B—C13—H13C | 109.5 |
| C1—C6—C7 | 111.67 (13) | C9—N1—C8 | 125.56 (14) |
| C1—C6—C5 | 120.99 (13) | C9—N1—H1 | 122.4 (12) |
| C7—C6—C5 | 127.33 (13) | C8—N1—H1 | 111.9 (12) |
| C8—C7—C6 | 111.94 (13) | C11—O3—C12 | 115.31 (12) |
| C8—C7—C11 | 119.90 (13) | C8—S1—C1 | 91.18 (7) |
| C6—C7—C11 | 128.15 (13) | | |

| | | | |
|---|---|---|---|
| C1—C2—C3—C4 | 48.83 (16) | C6—C7—C8—S1 | −0.19 (16) |
| C2—C3—C4—C5 | −63.48 (17) | C11—C7—C8—S1 | −178.84 (11) |
| C3—C4—C5—C6 | 42.28 (18) | C8—C7—C11—O2 | 0.7 (2) |
| C4—C5—C6—C1 | −10.54 (19) | C6—C7—C11—O2 | −177.70 (14) |
| C5—C6—C1—C2 | −0.9 (2) | C8—C7—C11—O3 | −179.44 (12) |
| C6—C1—C2—C3 | −18.7 (2) | C6—C7—C11—O3 | 2.1 (2) |
| S1—C1—C2—C3 | 161.76 (11) | O1—C9—N1—C8 | −1.7 (2) |
| C2—C1—C6—C7 | 179.52 (14) | C10—C9—N1—C8 | 177.50 (13) |
| S1—C1—C6—C7 | −0.95 (16) | C7—C8—N1—C9 | −174.86 (14) |
| S1—C1—C6—C5 | 178.59 (11) | S1—C8—N1—C9 | 5.9 (2) |
| C4—C5—C6—C7 | 168.93 (14) | O2—C11—O3—C12 | 5.2 (2) |
| C1—C6—C7—C8 | 0.73 (18) | C7—C11—O3—C12 | −174.69 (12) |
| C5—C6—C7—C8 | −178.78 (13) | C13—C12—O3—C11 | 169.63 (13) |
| C1—C6—C7—C11 | 179.25 (14) | C7—C8—S1—C1 | −0.29 (12) |
| C5—C6—C7—C11 | −0.3 (2) | N1—C8—S1—C1 | 179.02 (13) |
| C6—C7—C8—N1 | −179.48 (13) | C6—C1—S1—C8 | 0.73 (12) |

| | | | |
|---|---|---|---|
| C11—C7—C8—N1 | 1.9 (2) | C2—C1—S1—C8 | −179.71 (13) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O2 | 0.858 (18) | 1.979 (18) | 2.6798 (17) | 138.1 (16) |
| C2—H2*A*···O2$^{i}$ | 0.99 | 2.44 | 3.370 (2) | 157 |

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