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## Structure Reports

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## Ethyl 5-acetyl-2-amino-4-methylthiophene-3-carboxylate

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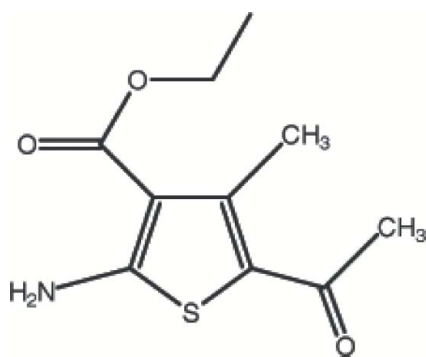
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Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.111; data-to-parameter ratio = 25.0.

In the title compound,  $\text{C}_{10}\text{H}_{13}\text{NO}_3\text{S}$ , prepared in a one-pot reaction, the molecular conformation is stabilized by an intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond. The packing is consolidated by further  $\text{N}-\text{H}\cdots\text{O}$  links. The H atoms of two of the methyl groups are disordered over two sets of sites with equal occupancies.

## Related literature

For related literature, see: Gewald *et al.* (1966); Sabnis *et al.* (1999); Akkurt *et al.* (2008); Allen *et al.* (1987).



## Experimental

## Crystal data

$\text{C}_{10}\text{H}_{13}\text{NO}_3\text{S}$   
 $M_r = 227.28$

Monoclinic,  $P2_1/n$   
 $a = 7.5397$  (3) Å

$b = 8.4514$  (3) Å  
 $c = 16.7058$  (6) Å  
 $\beta = 94.465$  (1)°  
 $V = 1061.28$  (7) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 0.29$  mm<sup>-1</sup>  
 $T = 150$  (2) K  
 $0.29 \times 0.26 \times 0.10$  mm

## Data collection

Bruker APEXII CCD diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)  
 $T_{\min} = 0.920$ ,  $T_{\max} = 0.971$

12338 measured reflections  
3400 independent reflections  
2944 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.111$   
 $S = 1.05$   
3400 reflections

136 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.44$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                                     | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|----------------------------------------------------------|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H}1\text{A}\cdots\text{O2}$             | 0.86         | 2.15               | 2.7404 (14) | 125                  |
| $\text{N1}-\text{H}1\text{A}\cdots\text{O2}^{\text{i}}$  | 0.86         | 2.40               | 3.2077 (15) | 156                  |
| $\text{N1}-\text{H}1\text{B}\cdots\text{O1}^{\text{ii}}$ | 0.86         | 2.24               | 2.9933 (14) | 147                  |
| $\text{C5}-\text{H5A}\cdots\text{O3}$                    | 0.96         | 2.04               | 2.7978 (16) | 135                  |

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

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: APEX2; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (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: HB2733).

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**supplementary materials**

*Acta Cryst.* (2008). E64, o1084 [ doi:10.1107/S1600536808014177 ]

## Ethyl 5-acetyl-2-amino-4-methylthiophene-3-carboxylate

M. Akkurt, S. Ö. Yildirim, A. M. Asiri and V. McKee

### Comment

2-Aminothiophene derivatives are important intermediates in the synthesis of a variety of agrochemicals, dyes and pharmacologically active compounds (Sabnis *et al.*, 1999). The most convergent and well established classical approach for the preparation of 2-aminothiophenes is Gewald's method (Gewald *et al.*, 1966), which involves the multicomponent condensation of a ketone with an activated nitrile and elemental sulfur in the presence of diethylamine as a catalyst.

As a part of an ongoing investigation into the development of anil derivatives, we here report the structure of the title compound, (I).

All bond lengths and angles in (I) (Fig. 1) are within their normal ranges (Akkurt *et al.*, 2008; Allen *et al.*, 1987). The thiophene ring is almost planar, with a maximum deviation of  $-0.009$  (1) Å for C6. The structure is stabilized by weak intramolecular C—H $\cdots$ O and N—H $\cdots$ O, and intermolecular N—H $\cdots$ O hydrogen bonding interactions (Table 1 and Fig. 2).

### Experimental

A mixture of ethyl cyanoacetate (11.3 g, 0.10 mol) and acetyl acetone (10.22 g, 0.10 mol) in absolute ethanol (20 ml) was added to a solution of elemental sulfur (3.2 g, 0.10 mol) and diethylamine (5 ml) in 50 ml absolute ethanol at room temperature. The reaction mixture was refluxed for 3 h and then cooled. The precipitated product was filtered, washed with ethanol, dried and recrystallized from ethanol as orange blocks of (I) [yield: 52%, m.p. 435–437 K]. IR ( $\text{cm}^{-1}$ ) 3408, 3294 (NH), 1666 (CO), 1605, 1586, 1253.  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ): 1.38 (t, 3H,  $\text{CH}_3\text{CH}_2\text{O}$ ), 2.43 (s, 3H,  $\text{COCH}_3$ ), 2.7 (s, 3H,  $\text{CH}_3$ ), 4.32 (q, 2H,  $\text{OCH}_2$ ), 6.67 (broad s, 2H,  $\text{NH}_2$ ).

### Refinement

All the H atoms were positioned geometrically ( $\text{C—H} = 0.96 - 0.97$  Å and  $\text{N—H} = 0.86$  Å) and refined as riding with  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{carrier})$  ( $1.5U_{\text{eq}}$  for methyl C). The methyl H atoms attached to C1 and C5 were refined as disordered over two sets of sites.

### Figures

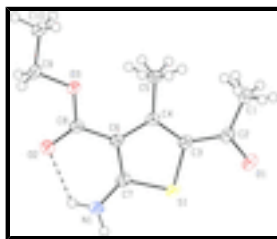


Fig. 1. View of the molecular structure of (I) with displacement ellipsoids for non-H atoms drawn at the 50% probability level. The hydrogen bond is shown as a dashed line.

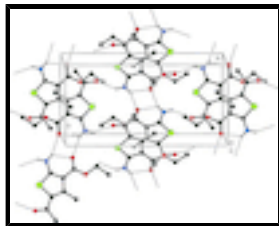


Fig. 2. View of the packing and hydrogen bonding in (I).

## Ethyl 5-acetyl-2-amino-4-methylthiophene-3-carboxylate

### Crystal data

|                                 |                                                         |
|---------------------------------|---------------------------------------------------------|
| $C_{10}H_{13}NO_3S$             | $F(000) = 480$                                          |
| $M_r = 227.28$                  | $D_x = 1.423 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1/n$            | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: $-P 2_1n$          | Cell parameters from 4913 reflections                   |
| $a = 7.5397 (3) \text{ \AA}$    | $\theta = 2.5\text{--}31.1^\circ$                       |
| $b = 8.4514 (3) \text{ \AA}$    | $\mu = 0.29 \text{ mm}^{-1}$                            |
| $c = 16.7058 (6) \text{ \AA}$   | $T = 150 \text{ K}$                                     |
| $\beta = 94.465 (1)^\circ$      | Block, orange                                           |
| $V = 1061.28 (7) \text{ \AA}^3$ | $0.29 \times 0.26 \times 0.10 \text{ mm}$               |
| $Z = 4$                         |                                                         |

### Data collection

|                                                             |                                                                        |
|-------------------------------------------------------------|------------------------------------------------------------------------|
| Bruker APEXII CCD diffractometer                            | 3400 independent reflections                                           |
| Radiation source: sealed tube graphite                      | 2944 reflections with $I > 2\sigma(I)$                                 |
| $\varphi$ and $\omega$ scans                                | $R_{\text{int}} = 0.025$                                               |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2003) | $\theta_{\text{max}} = 31.8^\circ$ , $\theta_{\text{min}} = 2.5^\circ$ |
| $T_{\text{min}} = 0.920$ , $T_{\text{max}} = 0.971$         | $h = -11 \rightarrow 10$                                               |
| 12338 measured reflections                                  | $k = -12 \rightarrow 12$                                               |
|                                                             | $l = -23 \rightarrow 24$                                               |

### Refinement

|                                 |                                                                |
|---------------------------------|----------------------------------------------------------------|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.038$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.111$               | H-atom parameters constrained                                  |
| $S = 1.05$                      | $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.3751P]$                |
| 3400 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 136 parameters                  | $(\Delta/\sigma)_{\text{max}} = 0.001$                         |
| 0 restraints                    | $\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$            |
|                                 | $\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$           |

*Special details*

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement.** Refinement on  $F^2$  for ALL reflections except those flagged by the user for potential systematic errors. Weighted  $R$ -factors  $wR$  and all goodnesses 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 observed criterion of  $F^2 > \sigma(F^2)$  is used only for calculating  $-R$ -factor-obs *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 ( $\text{\AA}^2$ )*

|      | <i>x</i>     | <i>y</i>     | <i>z</i>    | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|--------------|-------------|----------------------------------|-----------|
| S1   | 0.74240 (4)  | 0.44716 (4)  | 0.82882 (2) | 0.0234 (1)                       |           |
| O1   | 0.58575 (14) | 0.74136 (13) | 0.79734 (6) | 0.0324 (3)                       |           |
| O2   | 0.94110 (15) | 0.13922 (12) | 1.03979 (6) | 0.0315 (3)                       |           |
| O3   | 0.83808 (13) | 0.32842 (11) | 1.11776 (5) | 0.0251 (3)                       |           |
| N1   | 0.88307 (16) | 0.17090 (13) | 0.87660 (6) | 0.0263 (3)                       |           |
| C1   | 0.5682 (2)   | 0.86338 (17) | 0.92406 (8) | 0.0307 (4)                       |           |
| C2   | 0.61541 (17) | 0.72854 (15) | 0.87090 (7) | 0.0240 (3)                       |           |
| C3   | 0.69568 (16) | 0.58332 (15) | 0.90307 (7) | 0.0210 (3)                       |           |
| C4   | 0.74019 (15) | 0.52653 (14) | 0.97964 (7) | 0.0191 (3)                       |           |
| C5   | 0.71928 (18) | 0.61996 (15) | 1.05495 (7) | 0.0249 (3)                       |           |
| C6   | 0.81050 (15) | 0.36847 (14) | 0.97838 (7) | 0.0194 (3)                       |           |
| C7   | 0.82188 (16) | 0.31123 (14) | 0.89967 (7) | 0.0208 (3)                       |           |
| C8   | 0.87045 (16) | 0.26784 (14) | 1.04635 (7) | 0.0207 (3)                       |           |
| C9   | 0.90152 (19) | 0.23865 (16) | 1.18821 (7) | 0.0272 (3)                       |           |
| C10  | 0.8610 (2)   | 0.33700 (19) | 1.25956 (8) | 0.0325 (4)                       |           |
| HN1A | 0.92190      | 0.10290      | 0.91200     | 0.0320*                          |           |
| H1A  | 0.51730      | 0.94800      | 0.89160     | 0.0460*                          | 0.500     |
| H1B  | 0.67360      | 0.90040      | 0.95430     | 0.0460*                          | 0.500     |
| H1C  | 0.48370      | 0.82780      | 0.96020     | 0.0460*                          | 0.500     |
| H1D  | 0.59910      | 0.83610      | 0.97920     | 0.0460*                          | 0.500     |
| H1E  | 0.44280      | 0.88380      | 0.91640     | 0.0460*                          | 0.500     |
| H1F  | 0.63260      | 0.95630      | 0.91050     | 0.0460*                          | 0.500     |
| HN1B | 0.88350      | 0.14890      | 0.82640     | 0.0320*                          |           |
| H5A  | 0.75800      | 0.55710      | 1.10090     | 0.0370*                          | 0.500     |
| H5B  | 0.59650      | 0.64800      | 1.05770     | 0.0370*                          | 0.500     |
| H5C  | 0.78990      | 0.71440      | 1.05430     | 0.0370*                          | 0.500     |
| H5D  | 0.67160      | 0.72250      | 1.04100     | 0.0370*                          | 0.500     |
| H5E  | 0.83320      | 0.63170      | 1.08430     | 0.0370*                          | 0.500     |
| H5F  | 0.63970      | 0.56530      | 1.08760     | 0.0370*                          | 0.500     |
| H9A  | 1.02850      | 0.21990      | 1.18830     | 0.0330*                          |           |
| H9B  | 0.84120      | 0.13740      | 1.18920     | 0.0330*                          |           |
| H10A | 0.90050      | 0.28220      | 1.30800     | 0.0490*                          |           |

## supplementary materials

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|      |         |         |         |         |
|------|---------|---------|---------|---------|
| H10B | 0.73510 | 0.35490 | 1.25850 | 0.0490* |
| H10C | 0.92150 | 0.43670 | 1.25770 | 0.0490* |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$   | $U^{23}$    |
|-----|------------|------------|------------|-------------|------------|-------------|
| S1  | 0.0306 (2) | 0.0254 (2) | 0.0142 (1) | -0.0032 (1) | 0.0017 (1) | -0.0009 (1) |
| O1  | 0.0409 (6) | 0.0366 (5) | 0.0198 (4) | 0.0027 (4)  | 0.0031 (4) | 0.0076 (4)  |
| O2  | 0.0468 (6) | 0.0238 (4) | 0.0241 (5) | 0.0083 (4)  | 0.0037 (4) | -0.0003 (3) |
| O3  | 0.0348 (5) | 0.0258 (4) | 0.0147 (4) | 0.0055 (4)  | 0.0015 (3) | 0.0012 (3)  |
| N1  | 0.0373 (6) | 0.0222 (5) | 0.0197 (5) | -0.0009 (4) | 0.0038 (4) | -0.0052 (4) |
| C1  | 0.0386 (7) | 0.0263 (6) | 0.0271 (6) | 0.0064 (5)  | 0.0016 (5) | 0.0038 (5)  |
| C2  | 0.0248 (5) | 0.0262 (6) | 0.0212 (5) | -0.0031 (4) | 0.0027 (4) | 0.0040 (4)  |
| C3  | 0.0245 (5) | 0.0224 (5) | 0.0163 (5) | -0.0032 (4) | 0.0023 (4) | -0.0006 (4) |
| C4  | 0.0200 (5) | 0.0211 (5) | 0.0164 (5) | -0.0030 (4) | 0.0022 (4) | -0.0010 (4) |
| C5  | 0.0327 (6) | 0.0246 (6) | 0.0174 (5) | 0.0024 (5)  | 0.0017 (4) | -0.0026 (4) |
| C6  | 0.0216 (5) | 0.0206 (5) | 0.0161 (5) | -0.0026 (4) | 0.0018 (4) | -0.0013 (4) |
| C7  | 0.0228 (5) | 0.0217 (5) | 0.0180 (5) | -0.0045 (4) | 0.0024 (4) | -0.0017 (4) |
| C8  | 0.0234 (5) | 0.0215 (5) | 0.0173 (5) | -0.0025 (4) | 0.0019 (4) | -0.0008 (4) |
| C9  | 0.0360 (7) | 0.0270 (6) | 0.0185 (5) | 0.0027 (5)  | 0.0010 (5) | 0.0057 (4)  |
| C10 | 0.0407 (7) | 0.0392 (7) | 0.0179 (5) | 0.0024 (6)  | 0.0044 (5) | 0.0023 (5)  |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|              |             |            |        |
|--------------|-------------|------------|--------|
| S1—C3        | 1.7479 (13) | C1—H1A     | 0.9600 |
| S1—C7        | 1.7232 (12) | C1—H1B     | 0.9600 |
| O1—C2        | 1.2366 (15) | C1—H1C     | 0.9600 |
| O2—C8        | 1.2192 (16) | C1—H1D     | 0.9600 |
| O3—C8        | 1.3380 (15) | C1—H1E     | 0.9600 |
| O3—C9        | 1.4498 (15) | C1—H1F     | 0.9600 |
| N1—C7        | 1.3400 (16) | C5—H5A     | 0.9600 |
| N1—HN1A      | 0.8600      | C5—H5B     | 0.9600 |
| N1—HN1B      | 0.8600      | C5—H5C     | 0.9600 |
| C1—C2        | 1.5044 (19) | C5—H5D     | 0.9600 |
| C2—C3        | 1.4529 (18) | C5—H5E     | 0.9600 |
| C3—C4        | 1.3829 (17) | C5—H5F     | 0.9600 |
| C4—C6        | 1.4379 (17) | C9—H9A     | 0.9700 |
| C4—C5        | 1.5040 (17) | C9—H9B     | 0.9700 |
| C6—C7        | 1.4100 (17) | C10—H10A   | 0.9600 |
| C6—C8        | 1.4619 (17) | C10—H10B   | 0.9600 |
| C9—C10       | 1.5039 (19) | C10—H10C   | 0.9600 |
| C3—S1—C7     | 91.72 (6)   | H1C—C1—H1D | 56.00  |
| C8—O3—C9     | 116.87 (10) | H1C—C1—H1E | 56.00  |
| HN1A—N1—HN1B | 120.00      | H1C—C1—H1F | 141.00 |
| C7—N1—HN1A   | 120.00      | H1D—C1—H1E | 110.00 |
| C7—N1—HN1B   | 120.00      | H1D—C1—H1F | 109.00 |
| O1—C2—C1     | 119.18 (12) | H1E—C1—H1F | 109.00 |
| O1—C2—C3     | 118.66 (11) | C4—C5—H5A  | 109.00 |

|              |              |               |              |
|--------------|--------------|---------------|--------------|
| C1—C2—C3     | 122.17 (11)  | C4—C5—H5B     | 110.00       |
| S1—C3—C2     | 113.26 (9)   | C4—C5—H5C     | 109.00       |
| C2—C3—C4     | 134.38 (11)  | C4—C5—H5D     | 109.00       |
| S1—C3—C4     | 112.34 (9)   | C4—C5—H5E     | 109.00       |
| C5—C4—C6     | 124.25 (10)  | C4—C5—H5F     | 109.00       |
| C3—C4—C6     | 111.84 (10)  | H5A—C5—H5B    | 109.00       |
| C3—C4—C5     | 123.91 (11)  | H5A—C5—H5C    | 110.00       |
| C4—C6—C7     | 112.44 (10)  | H5A—C5—H5D    | 141.00       |
| C4—C6—C8     | 128.40 (11)  | H5A—C5—H5E    | 56.00        |
| C7—C6—C8     | 119.16 (10)  | H5A—C5—H5F    | 56.00        |
| N1—C7—C6     | 128.26 (11)  | H5B—C5—H5C    | 109.00       |
| S1—C7—N1     | 120.11 (9)   | H5B—C5—H5D    | 56.00        |
| S1—C7—C6     | 111.64 (9)   | H5B—C5—H5E    | 141.00       |
| O2—C8—O3     | 122.18 (11)  | H5B—C5—H5F    | 56.00        |
| O2—C8—C6     | 124.04 (11)  | H5C—C5—H5D    | 56.00        |
| O3—C8—C6     | 113.77 (10)  | H5C—C5—H5E    | 56.00        |
| O3—C9—C10    | 106.24 (11)  | H5C—C5—H5F    | 141.00       |
| C2—C1—H1A    | 109.00       | H5D—C5—H5E    | 109.00       |
| C2—C1—H1B    | 109.00       | H5D—C5—H5F    | 109.00       |
| C2—C1—H1C    | 109.00       | H5E—C5—H5F    | 109.00       |
| C2—C1—H1D    | 109.00       | O3—C9—H9A     | 110.00       |
| C2—C1—H1E    | 109.00       | O3—C9—H9B     | 110.00       |
| C2—C1—H1F    | 109.00       | C10—C9—H9A    | 111.00       |
| H1A—C1—H1B   | 109.00       | C10—C9—H9B    | 110.00       |
| H1A—C1—H1C   | 109.00       | H9A—C9—H9B    | 109.00       |
| H1A—C1—H1D   | 141.00       | C9—C10—H10A   | 109.00       |
| H1A—C1—H1E   | 56.00        | C9—C10—H10B   | 110.00       |
| H1A—C1—H1F   | 56.00        | C9—C10—H10C   | 109.00       |
| H1B—C1—H1C   | 110.00       | H10A—C10—H10B | 109.00       |
| H1B—C1—H1D   | 56.00        | H10A—C10—H10C | 110.00       |
| H1B—C1—H1E   | 141.00       | H10B—C10—H10C | 109.00       |
| H1B—C1—H1F   | 56.00        |               |              |
| C7—S1—C3—C2  | -178.44 (10) | C2—C3—C4—C6   | 177.16 (13)  |
| C7—S1—C3—C4  | 0.21 (10)    | C3—C4—C6—C7   | 1.69 (15)    |
| C3—S1—C7—N1  | -179.68 (11) | C3—C4—C6—C8   | -179.30 (12) |
| C3—S1—C7—C6  | 0.76 (10)    | C5—C4—C6—C7   | -177.67 (11) |
| C9—O3—C8—O2  | 3.93 (18)    | C5—C4—C6—C8   | 1.35 (19)    |
| C9—O3—C8—C6  | -177.14 (10) | C4—C6—C7—S1   | -1.52 (13)   |
| C8—O3—C9—C10 | 175.85 (11)  | C4—C6—C7—N1   | 178.96 (12)  |
| O1—C2—C3—S1  | 1.18 (16)    | C8—C6—C7—S1   | 179.36 (9)   |
| O1—C2—C3—C4  | -177.07 (13) | C8—C6—C7—N1   | -0.2 (2)     |
| C1—C2—C3—S1  | -178.56 (10) | C4—C6—C8—O2   | -174.17 (12) |
| C1—C2—C3—C4  | 3.2 (2)      | C4—C6—C8—O3   | 6.93 (18)    |
| S1—C3—C4—C5  | 178.25 (10)  | C7—C6—C8—O2   | 4.79 (19)    |
| S1—C3—C4—C6  | -1.10 (13)   | C7—C6—C8—O3   | -174.12 (11) |
| C2—C3—C4—C5  | -3.5 (2)     |               |              |

## supplementary materials

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### Hydrogen-bond geometry (Å, °)

| <i>D</i> —H··· <i>A</i>    | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—HN1A···O2               | 0.86        | 2.15          | 2.7404 (14)           | 125                     |
| N1—HN1A···O2 <sup>i</sup>  | 0.86        | 2.40          | 3.2077 (15)           | 156                     |
| N1—HN1B···O1 <sup>ii</sup> | 0.86        | 2.24          | 2.9933 (14)           | 147                     |
| C5—H5A···O3                | 0.96        | 2.04          | 2.7978 (16)           | 135                     |

Symmetry codes: (i)  $-x+2, -y, -z+2$ ; (ii)  $-x+3/2, y-1/2, -z+3/2$ .

Fig. 1

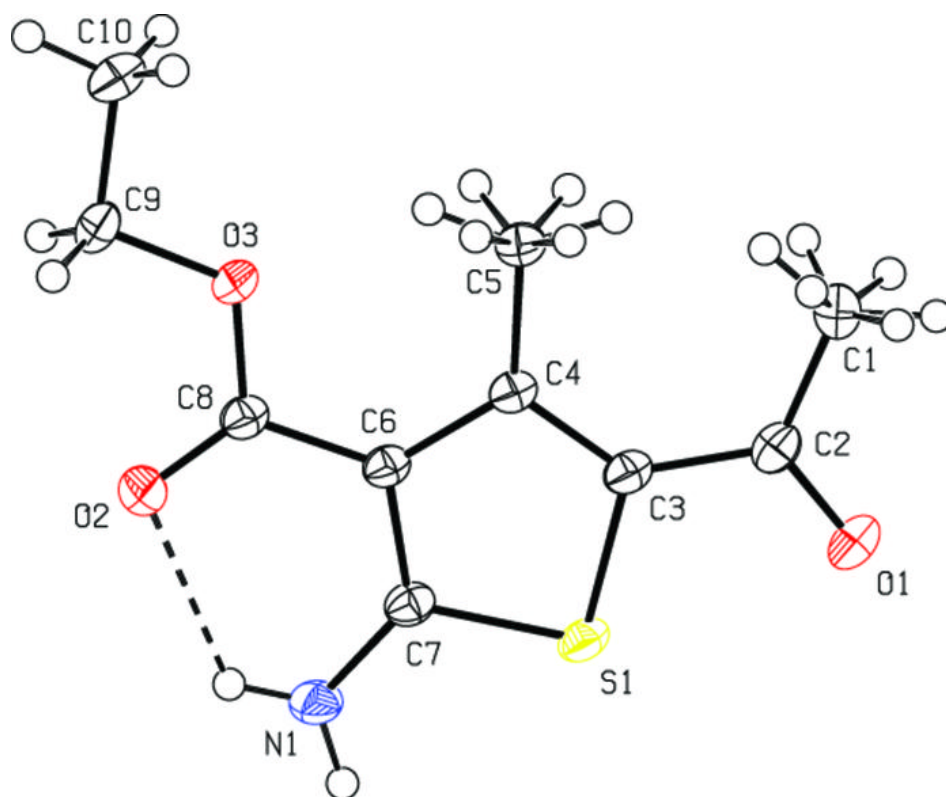


Fig. 2

