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## Structure Reports

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## 5-[(2-Hydroxyethyl)(methyl)amino]thiophene-2-carbaldehyde

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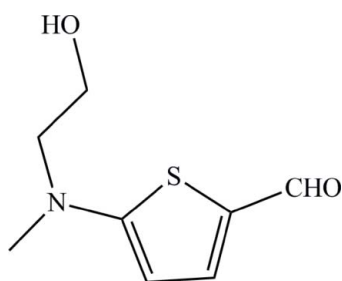
Received 12 April 2014; accepted 23 May 2014

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.024;  $wR$  factor = 0.067; data-to-parameter ratio = 14.1.

In the title compound,  $\text{C}_8\text{H}_{11}\text{NO}_2\text{S}$ , the aldehyde group is approximately coplanar with the thiophene ring [maximum deviation =  $0.023$  (2) Å]. In the crystal, molecules are linked by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds into supramolecular chains propagating along the  $a$ -axis direction.

## Related literature

For potential applications of thiophene derivatives, see: Encinas (2002). For a related thiophene derivative, see: Perašínová *et al.* (2006).



## Experimental

## Crystal data

 $\text{C}_8\text{H}_{11}\text{NO}_2\text{S}$   
 $M_r = 185.24$ 

 Orthorhombic,  $Pca2_1$   
 $a = 15.764$  (5) Å

 $b = 5.136$  (5) Å  
 $c = 11.028$  (5) Å  
 $V = 892.9$  (10) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.32$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.30 \times 0.20 \times 0.20$  mm

## Data collection

 Bruker SMART 1000 CCD area-  
 detector diffractometer  
 5828 measured reflections

 1564 independent reflections  
 1514 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.067$   
 $S = 1.08$   
 1564 reflections  
 111 parameters  
 1 restraint  
 H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.14$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.13$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983),  
 756 Friedel pairs  
 Absolute structure parameter:  
 $-0.03$  (7)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.93	2.751 (2)	174

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: XU5786).

## References

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## supporting information

*Acta Cryst.* (2014). E70, o724 [doi:10.1107/S1600536814012021]

**5-[(2-Hydroxyethyl)(methyl)amino]thiophene-2-carbaldehyde**

Xian-Shun Sun, Nan-Qi Shao and Dan-Dan Li

**S1. Comment**

The introduction about the highpolarizability of sulfur atoms in thiophene rings leads to a stabilization of the conjugated chain and to excellent charge transport properties. Functional thiophene derivatives have attracted comprehensive interest among researchers all over the world and have actually been advanced to be among the most frequently used  $\pi$ -conjugated materials, in particular as active components in organic electronic devices and molecular electronics (Encinas *et al.*, 2002). In the title compound (I) (Fig. 1), the S1—C4 bond length of 1.7398 (18) Å is longer than the corresponding S1—C15 bond length of 1.708 (2) Å in related thiophene derivative (Perašínová *et al.* 2006), which is due to the fact that there is a higher  $\pi$ -electron delocalized system in the molecule 5-(fluoren-9-ylidenemethyl)thiophene-2-carbaldehyde. In the crystal structure of (I), the molecules are interconnected, *via* hydrogen bonds (Table 1) [O1—H1 $\cdots$ O2<sup>i</sup>; symmetry codes: (i)  $x - 1/2, -y, z$ ], forming a one-dimensional structure (Fig. 2).

**S2. Experimental**

A 0.86 g (4.5 mmol) amount of 5-bromothiophene-2-carbaldehyde, 1.13 g (15 mmol) of 2-(methylamino)ethanol, and 0.1 g of toluene-4-sulfonic acid were mixed and stirred at a bath temperature of 373 K for 20 h. The mixture was cooled, 25 mL of water was added. The organic layer and dichloromethane extracts were combined, washed with water, and dried over MgSO<sub>4</sub>. Evaporation of the solvent, purification by column chromatography. <sup>1</sup>H NMR: (400 Hz, DMSO-d<sub>6</sub>), d(p.p.m.):9.40 (s, 1H), 7.65 (d, 1H), 6.12 (d, 1H), 4.87 (t, 1H), 3.62 (q, 2H), 3.47 (t, 2H), 3.09 (s, 3H).

**S3. Refinement**

All H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å and O—H = 0.82 Å) and allowed to ride on their parent atoms with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  or  $1.5 U_{\text{eq}}(\text{C})$  and  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$

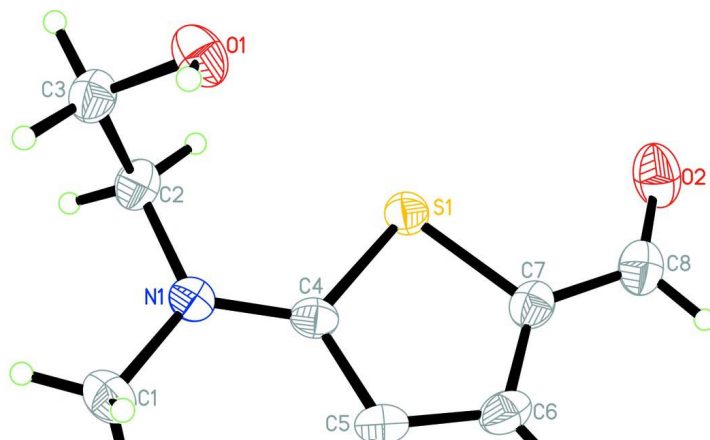


Figure 1

The molecular structure of the title compound (I) showing 30% probability displacement ellipsoids.

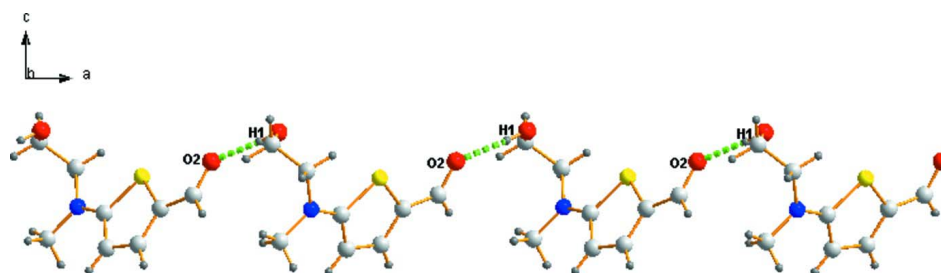


Figure 2

The crystal packing of (I), showing O—H...O hydrogen-bonding interactions as dashed lines.

### 5-[(2-Hydroxyethyl)(methyl)amino]thiophene-2-carbaldehyde

#### Crystal data

$C_8H_{11}NO_2S$

$M_r = 185.24$

Orthorhombic,  $Pca2_1$

Hall symbol:  $P\ 2c\ -2ac$

$a = 15.764\ (5)\ \text{\AA}$

$b = 5.136\ (5)\ \text{\AA}$

$c = 11.028\ (5)\ \text{\AA}$

$V = 892.9\ (10)\ \text{\AA}^3$

$Z = 4$

$F(000) = 392$

$D_x = 1.378\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71069\ \text{\AA}$

Cell parameters from 3223 reflections

$\theta = 2.6\text{--}26.8^\circ$

$\mu = 0.32\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, yellow

$0.30 \times 0.20 \times 0.20\ \text{mm}$

#### Data collection

Bruker SMART 1000 CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

phi and  $\omega$  scans

5828 measured reflections

1564 independent reflections

1514 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 25.0^\circ$ ,  $\theta_{\text{min}} = 2.6^\circ$

$h = -17 \rightarrow 18$

$k = -5 \rightarrow 6$

$l = -13 \rightarrow 13$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.024$  $wR(F^2) = 0.067$  $S = 1.08$ 

1564 reflections

111 parameters

1 restraint

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.0567P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$ Absolute structure: Flack (1983), 756 Friedel  
pairsAbsolute structure parameter:  $-0.03$  (7)*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.

**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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.53676 (3)	0.28672 (8)	0.22080 (5)	0.04276 (14)
O1	0.32880 (9)	0.3310 (3)	0.35492 (15)	0.0575 (4)
H1	0.2888	0.2479	0.3274	0.086*
N1	0.40014 (10)	0.5376 (3)	0.13076 (14)	0.0446 (4)
C4	0.45817 (10)	0.3498 (4)	0.11400 (16)	0.0385 (4)
C5	0.46620 (12)	0.1849 (4)	0.01313 (18)	0.0459 (5)
H5	0.4301	0.1889	-0.0535	0.055*
C6	0.53411 (13)	0.0157 (4)	0.02439 (17)	0.0501 (5)
H6	0.5479	-0.1056	-0.0351	0.060*
C7	0.57958 (11)	0.0402 (4)	0.12977 (19)	0.0457 (4)
O2	0.68678 (9)	-0.0828 (4)	0.26743 (18)	0.0723 (5)
C2	0.39359 (13)	0.6822 (3)	0.24362 (18)	0.0460 (5)
H2A	0.3870	0.8658	0.2251	0.055*
H2B	0.4459	0.6617	0.2888	0.055*
C1	0.33673 (14)	0.5845 (5)	0.0376 (2)	0.0583 (6)
H1A	0.3644	0.6127	-0.0389	0.087*
H1B	0.3040	0.7356	0.0585	0.087*
H1C	0.2999	0.4362	0.0315	0.087*
C8	0.64980 (13)	-0.1077 (4)	0.1692 (2)	0.0553 (5)
H8	0.6700	-0.2351	0.1167	0.066*
C3	0.32038 (12)	0.5959 (4)	0.32204 (19)	0.0490 (5)
H3A	0.3184	0.7022	0.3947	0.059*
H3B	0.2676	0.6204	0.2783	0.059*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0454 (2)	0.0426 (2)	0.0403 (2)	0.00002 (17)	-0.0065 (2)	-0.0035 (3)
O1	0.0509 (8)	0.0526 (8)	0.0689 (10)	-0.0042 (7)	-0.0065 (8)	0.0180 (7)
N1	0.0451 (8)	0.0484 (9)	0.0403 (7)	0.0028 (7)	-0.0043 (7)	0.0008 (7)
C4	0.0413 (10)	0.0403 (9)	0.0338 (9)	-0.0082 (7)	0.0010 (7)	0.0035 (8)
C5	0.0524 (12)	0.0537 (12)	0.0317 (9)	-0.0072 (9)	0.0002 (8)	-0.0023 (8)
C6	0.0592 (13)	0.0510 (12)	0.0400 (10)	-0.0036 (9)	0.0120 (9)	-0.0060 (9)
C7	0.0433 (10)	0.0437 (10)	0.0500 (9)	-0.0024 (8)	0.0106 (8)	0.0003 (8)
O2	0.0587 (9)	0.0613 (10)	0.0969 (13)	0.0069 (8)	-0.0212 (9)	0.0000 (9)
C2	0.0446 (9)	0.0380 (9)	0.0555 (14)	-0.0031 (7)	0.0037 (8)	-0.0050 (8)
C1	0.0518 (12)	0.0673 (15)	0.0558 (12)	0.0036 (11)	-0.0106 (9)	0.0090 (10)
C8	0.0484 (11)	0.0469 (12)	0.0705 (13)	0.0010 (10)	0.0064 (12)	0.0012 (10)
C3	0.0494 (11)	0.0427 (10)	0.0550 (12)	0.0026 (9)	0.0057 (9)	-0.0005 (9)

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

S1—C4	1.7398 (18)	C7—C8	1.411 (3)
S1—C7	1.751 (2)	O2—C8	1.237 (3)
O1—C3	1.414 (3)	C2—C3	1.509 (3)
O1—H1	0.8200	C2—H2A	0.9700
N1—C4	1.342 (3)	C2—H2B	0.9700
N1—C2	1.453 (2)	C1—H1A	0.9600
N1—C1	1.454 (3)	C1—H1B	0.9600
C4—C5	1.404 (3)	C1—H1C	0.9600
C5—C6	1.384 (3)	C8—H8	0.9300
C5—H5	0.9300	C3—H3A	0.9700
C6—C7	1.371 (3)	C3—H3B	0.9700
C6—H6	0.9300		
C4—S1—C7	91.21 (10)	C3—C2—H2A	108.9
C3—O1—H1	109.5	N1—C2—H2B	108.9
C4—N1—C2	122.26 (16)	C3—C2—H2B	108.9
C4—N1—C1	119.38 (17)	H2A—C2—H2B	107.7
C2—N1—C1	118.15 (17)	N1—C1—H1A	109.5
N1—C4—C5	127.17 (17)	N1—C1—H1B	109.5
N1—C4—S1	121.74 (14)	H1A—C1—H1B	109.5
C5—C4—S1	111.08 (15)	N1—C1—H1C	109.5
C6—C5—C4	112.17 (18)	H1A—C1—H1C	109.5
C6—C5—H5	123.9	H1B—C1—H1C	109.5
C4—C5—H5	123.9	O2—C8—C7	125.7 (2)
C7—C6—C5	114.99 (18)	O2—C8—H8	117.1
C7—C6—H6	122.5	C7—C8—H8	117.1
C5—C6—H6	122.5	O1—C3—C2	110.98 (15)
C6—C7—C8	128.45 (19)	O1—C3—H3A	109.4
C6—C7—S1	110.53 (15)	C2—C3—H3A	109.4
C8—C7—S1	120.99 (17)	O1—C3—H3B	109.4

N1—C2—C3	113.27 (16)	C2—C3—H3B	109.4
N1—C2—H2A	108.9	H3A—C3—H3B	108.0
C2—N1—C4—C5	174.75 (18)	C5—C6—C7—C8	177.8 (2)
C1—N1—C4—C5	0.2 (3)	C5—C6—C7—S1	-0.2 (2)
C2—N1—C4—S1	-6.4 (2)	C4—S1—C7—C6	0.14 (15)
C1—N1—C4—S1	179.03 (15)	C4—S1—C7—C8	-178.05 (16)
C7—S1—C4—N1	-179.10 (15)	C4—N1—C2—C3	-103.3 (2)
C7—S1—C4—C5	-0.06 (15)	C1—N1—C2—C3	71.4 (2)
N1—C4—C5—C6	178.94 (18)	C6—C7—C8—O2	-177.4 (2)
S1—C4—C5—C6	0.0 (2)	S1—C7—C8—O2	0.5 (3)
C4—C5—C6—C7	0.1 (2)	N1—C2—C3—O1	60.7 (2)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O1—H1 $\cdots$ O2 <sup>i</sup>	0.82	1.93	2.751 (2)	174

Symmetry code: (i)  $x-1/2, -y, z$ .