Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-Ethyl 5-methyl 3-hydroxythiophene-2,5-dicarboxylate

Simon J. Coles,[a]* Holger Guthmann,[b] Michael B. Hursthouse[a] and Patrick J. Murphy[b]

[a]Department of Chemistry, University of Southampton, Southampton SO17 1BJ, England, and [b]Department of Chemistry, University of Wales, Bangor, Gwynedd LL57 2UW, Wales

Correspondence e-mail: s.j.coles@soton.ac.uk

Received 7 September 2001
Accepted 19 September 2001
Online 29 September 2001

Key indicators

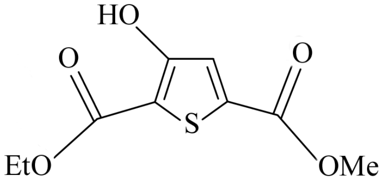
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.006 Å
R factor = 0.084
wR factor = 0.268
Data-to-parameter ratio = 16.6

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

The title compound, $C_9H_{10}O_5S$, crystallizes with two independent molecules in the asymmetric unit. Hydrogen bonding between these planar molecules gives rise to dimeric species, which produce a crystal structure composed of sheets, stacked parallel to the c axis.

Comment

The title compound, (I), crystallizes with two chemically equivalent but crystallographically independent molecules in the asymmetric unit and is shown, along with the atom numbering scheme, in Fig. 1.



(I)

The structure is composed of a thiophene ring substituted in the 2-position by an ethyl ester, in the 3-position by a hydroxyl group and in the 5-position by a methyl ester. This arrangement produces a virtually planar molecule with only the methyl and ethyl H atoms deviating from the plane. Maximum deviations from the heavy-atom plane are 0.124 (C7) and 0.195 Å (O6) for molecules A and B, respectively. All bond lengths and angles fall into accepted ranges reported from an assessment of the Cambridge Structural Database (Allen & Kennard, 1993; Orpen *et al.*, 1992). Pertinent non-bonded interactions are detailed in Table 1. Intramolecular hydrogen bonds exist in molecule A between O3—H3A···O1 and concordantly between O8—H8···O6 in molecule B. These intramolecular interactions are bifurcated, as strong intermolecular hydrogen bonds between O3—H3A···O6 and O8—H8···O1 allow dimeric association of the two independent molecules. These dimer units produce a crystal structure composed of sheets, stacked parallel to the c axis.

Experimental

2-Ethyl 5-methyl 3-hydroxythiophene-2,5-dicarboxylate, (I), was formed from the reaction of ethyl thioglycolate, (II), with dimethylacetylene dicarboxylate (DMAD) in the presence of 2,4-diaminobutanoic acid (DBU). The reaction proceeds *via* a Michael addition of (II) to DMAD, followed by an *in-situ* Dieckmann cyclization. This reaction was originally observed in an attempted non-classical Wittig reaction of (II) with DMAD and PPh_3 (Evans *et al.*, 2001).

DOI: 10.1107/S1600536801015379

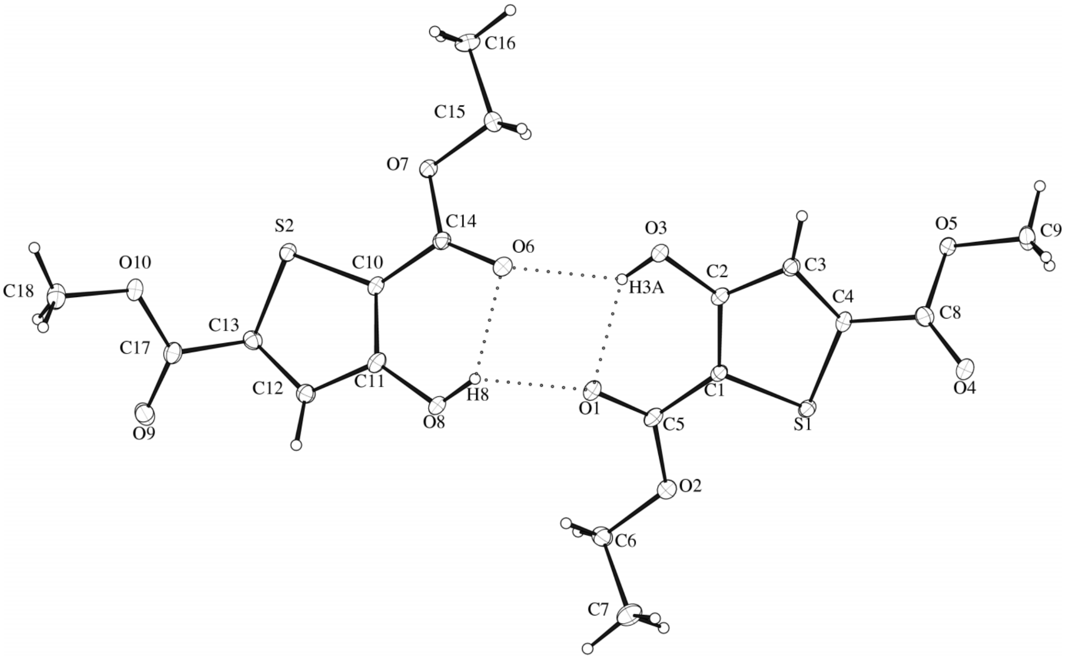


Figure 1
View of (I) (50% probability displacement ellipsoids).

Crystal data

$C_9H_{10}O_5S$
$M_r = 230.23$
Triclinic, $P\overline{1}$
$a = 7.6602\ (15)$ Å
$b = 11.120\ (2)$ Å
$c = 12.572\ (3)$ Å
$\alpha = 94.09\ (3)^\circ$
$\beta = 102.64\ (3)^\circ$
$\gamma = 95.77\ (3)^\circ$
$V = 1034.8\ (4)$ Å^3
$Z = 4$
$D_x = 1.478$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9463 reflections
$\theta = 2.9$–27.5°
$\mu = 0.31$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.20 × 0.18 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.940$, $T_{\max} = 0.982$
11 567 measured reflections
4585 independent reflections
3689 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.067$
$\theta_{\max} = 27.5^\circ$
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.085$
$wR(F^2) = 0.268$
$S = 1.05$
4585 reflections
277 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1293P)^2 + 5.7167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 1.73$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.020 (9)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O1	0.84	2.1	2.791 (5)	140
O3—H3A···O6	0.84	2.19	2.873 (5)	138
O8—H8···O1	0.84	2.16	2.845 (5)	138
O8—H8···O6	0.84	2.11	2.795 (5)	139

The quality of the crystal was poor, showing crazed faces, which produced a diffraction pattern with split reflections. The integration of the reflections was performed with a large box size and small spot size, in order to attempt to measure only one component of the splitting, however, in such a bad situation, some intensities will inevitably be enhanced or reduced. This severely affected the refinement and final structure as demonstrated by the statistics of fit, residual density, *etc*. H atoms were included in idealized positions with coordinates and displacement parameters allowed to ride on those of the parent atom.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993).

The authors would like to thank the EPSRC for funding of crystallographic facilities.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–429.
Evans, L. A., Griffiths, K. E., Guthmann, H. & Murphy, P. J. (2001). *Tetrahedron Lett.* Submitted.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G., & Taylor, R. (1992). *International Tables for Crystallography*, Vol. C, pp. 707–791. Kluwer Academic Publiahers.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter & R. M. Sweet, pp. 307–326. London: Academic Press.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.