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Dimethyl 6b,9a-dihydroxyacenaphtho[1,2-*c*]thiophene-7,9-dicarboxylate

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In the title compound, $C_{18}H_{16}O_6S$, two hydroxy groups, having a *syn* regiochemistry, act as both hydrogen-bond donors and acceptors, resulting in the formation of an infinite molecular chain.

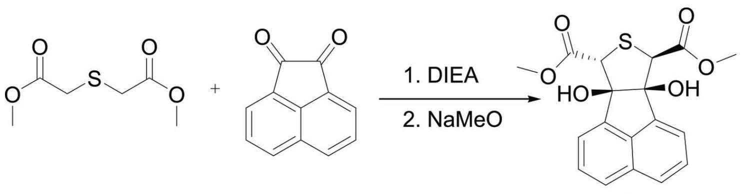
Key indicators

Single-crystal X-ray study
T = 173 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.109
Data-to-parameter ratio = 16.1

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

Comment

We have investigated the title compound, (I), as a dihydroxythiophene derivative *en route* to the synthesis of aromatic thiophenes.



(I)

Bond lengths and angles in (I) are within their normal ranges (Allen *et al.*, 1987). The five-membered ring containing the S atom adopts an envelope conformation with the S atom as the flap, and the mean plane of the four C atoms of the ring makes a dihedral angle of 58.4 (1)° with the naphthalene ring system.

An intermolecular O—H···O hydrogen bond is established between one of the *cis*-diols and and an ester carbonyl O atom of an adjacent molecule (Table 1), forming a zigzag molecular chain along the *c* axis. The other hydroxy group of the *cis*-diol is intramolecularly hydrogen-bonded to a neighbouring hydroxy group.

Experimental

A mixture of *N*,*N*-diisopropylethylamine (DIEA) (0.5 ml, 2.9 mmol), diacetyl sulfide (0.51 g, 2.9 mmol) and acenapthaquinone (0.46 g, 2.5 mmol) in methanol (7.5 ml) was stirred at 303 K. After an hour, tetrahydrofuran (30 ml) was added and the solution was then stirred overnight at 328 K. Sodium methoxide (5 ml of 0.5 *M* NaOMe in MeOH) was then added to the mixture. After an hour, the reaction mixture was added to saturated NH_4Cl (50 ml), extracted with diethyl ether and concentrated under reduced pressure. The crude product was separated by column chromatography on silica gel with hexane–ethyl acetate (15:1, *v*/*v*) as eluant, followed by hexane–ethyl acetate (7:1, *v*/*v*). Appropriate fractions were then combined and concentrated under reduced pressure to afford a product (0.062 g). The product was then recrystallized from a water–acetone (1:1, *v*/*v*) solution (2 ml) over 3 d (yield 0.051 g, 6%).

Crystal data

$C_{18}H_{16}O_6S$	$Z = 4$
$M_r = 360.37$	$D_x = 1.479$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.992$ (2) Å	$\mu = 0.23$ mm^{-1}
$b = 18.501$ (8) Å	$T = 173$ (2) K
$c = 11.165$ (5) Å	Cubic, colourless
$\beta = 101.34$ (2)°	0.20 × 0.10 × 0.08 mm
$V = 1618.6$ (11) Å^3	

Data collection

Nonius KappaCCD area-detector diffractometer	6842 measured reflections
ω and φ scans	3692 independent reflections
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	2649 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.955$, $T_{\max} = 0.982$	$R_{\rm int} = 0.041$
	$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.65P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$(\Delta/\sigma)_{\max} < 0.001$
$wR(F^2) = 0.109$	$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{\min} = -0.34$ e Å^{-3}
3692 reflections	
229 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H05···O2$^{\rm i}$	0.84	1.95	2.754 (2)	161
O6—H06···O5	0.84	2.16	2.633 (2)	115

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

H atoms were located in a difference Fourier map and were included in the refinement at geometrically idealized positions in the riding-model approximation, with O—H = 0.86 Å and C—H = 0.96 or 0.98 Å, and with $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(O) and $1.2U_{\rm eq}$(C).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

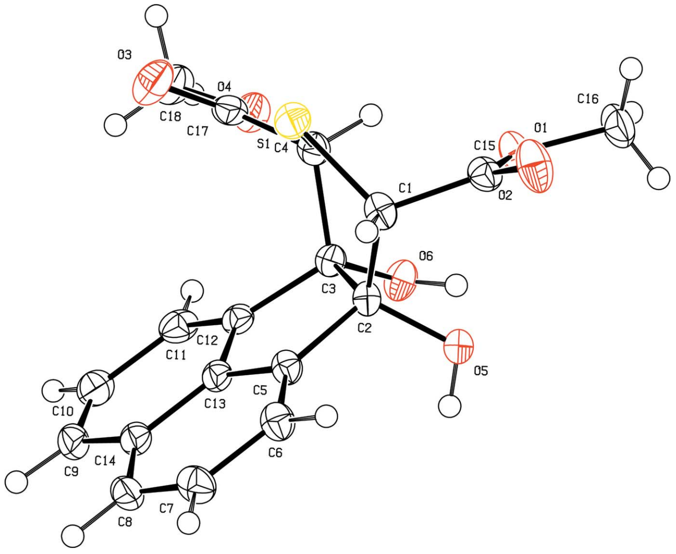


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

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