Absorption correction:	$\theta_{max} = 27.56°$
ψ scans (North, Phillips & Mathews, 1968)	$h = 0 \rightarrow 7$
$T_{min} = 0.922$, $T_{max} = 1.000$	$k = 0 \rightarrow 14$
4873 measured reflections	$l = -39 \rightarrow 39$
4424 independent reflections	3 standard reflections monitored every 200 reflections
	intensity decay: 0.5%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.03$
$R = 0.0663$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
$wR = 0.0647$	$\Delta\rho_{min} = -0.40$ e Å^{-3}
$S = 2.967$	Extinction correction: none
1488 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
217 parameters	
H atoms riding with C—H and N—H 0.95 Å	
$w = 1/[\sigma^2(F_o) + 0.013(F_o^2)]$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Cl1	0.2812 (5)	−0.0013 (2)	0.04817 (9)	0.093 (1)
Cl2	0.6271 (3)	−0.3527 (2)	0.21221 (7)	0.0534 (7)
N1	0.1854 (10)	−0.5783 (6)	0.1281 (2)	0.045 (2)
N2	0.4311 (12)	−0.6965 (6)	0.1686 (2)	0.049 (2)
N3	0.2775 (10)	−0.5423 (5)	0.2456 (2)	0.041 (2)
C1	0.2441 (13)	−0.6210 (7)	0.1694 (3)	0.042 (3)
C2	0.4955 (13)	−0.7036 (7)	0.1248 (3)	0.044 (3)
C3	0.6789 (14)	−0.7705 (7)	0.1042 (3)	0.057 (3)
C4	0.7048 (15)	−0.7603 (9)	0.0593 (3)	0.066 (3)
C5	0.5535 (16)	−0.6864 (9)	0.0337 (3)	0.064 (3)
C6	0.3694 (14)	−0.6199 (8)	0.0527 (3)	0.061 (3)
C7	0.3438 (12)	−0.6304 (7)	0.0984 (2)	0.039 (2)
C8	−0.0122 (12)	−0.4952 (8)	0.1143 (3)	0.054 (3)
C9	0.0700 (12)	−0.3753 (7)	0.0967 (2)	0.042 (2)
C10	−0.0741 (13)	−0.3125 (8)	0.0669 (3)	0.056 (3)
C11	−0.0141 (15)	−0.1988 (9)	0.0507 (3)	0.061 (3)
C12	0.2004 (15)	−0.1447 (8)	0.0660 (3)	0.055 (3)
C13	0.3494 (14)	−0.2078 (8)	0.0947 (3)	0.060 (3)
C14	0.2856 (14)	−0.3212 (8)	0.1105 (3)	0.051 (3)
C15	0.1109 (13)	−0.5880 (7)	0.2111 (3)	0.048 (3)
C16	0.1440 (15)	−0.4816 (9)	0.2838 (3)	0.070 (3)
C17	0.3169 (19)	−0.4781 (10)	0.3205 (3)	0.102 (5)
C18	0.4935 (15)	−0.5806 (10)	0.3140 (3)	0.073 (4)
C19	0.4305 (13)	−0.6369 (8)	0.2694 (3)	0.062 (3)

Table 2. *Selected geometric parameters (Å, °)*

Cl1—C12	1.722 (9)	N3—C16	1.526 (10)
N1—C1	1.357 (8)	N3—C19	1.509 (9)
N1—C7	1.384 (8)	C1—C15	1.507 (10)
N1—C8	1.475 (9)	C8—C9	1.491 (10)
N2—C1	1.325 (8)	C16—C17	1.45 (1)
N2—C2	1.371 (8)	C17—C18	1.50 (1)
N3—C15	1.462 (9)	C18—C19	1.51 (1)
C1—N1—C7	107.5 (6)	N2—C2—C3	130.8 (8)
C1—N1—C8	129.3 (7)	N2—C2—C7	110.4 (6)
C7—N1—C8	123.2 (7)	N1—C7—C2	104.6 (6)
C1—N2—C2	105.5 (6)	N1—C7—C6	132.8 (8)
C15—N3—C16	112.0 (6)	N1—C8—C9	114.5 (6)
C15—N3—C19	116.0 (6)	N3—C15—C1	111.1 (6)
C16—N3—C19	102.7 (6)	N3—C16—C17	105.1 (7)
N1—C1—N2	112.0 (7)	C16—C17—C18	107.6 (9)
N1—C1—C15	124.2 (7)	C17—C18—C19	106.3 (7)
N2—C1—C15	123.8 (7)	N3—C19—C18	104.8 (7)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1N3···Cl2	0.95	2.08	3.021 (6)	173

The space group, $P2_1/n$, was uniquely determined from the systematic absences: $h0l$, $h + l = 2n + 1$ and $0k0$, $k = 2n + 1$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

The author thanks the Natural Sciences and Engineering Research Council (Canada) for providing the diffractometer through an equipment grant to the University of Calgary, and the University of Calgary for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1128). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Bis(ethylsulfonyl)benzo[*b*]thiophene

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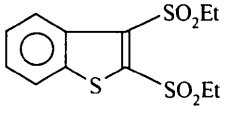
Abstract

The structure of the title compound, diethyl benzo[*b*]-thiophene-2,3-disulfinate, $C_{12}H_{14}O_4S_3$, is composed of an essentially planar benzothiophene moiety contain-

ing two ethylsulfonyl groups which are oriented in opposite directions. The mean values of the important bond distances are S—C_{sp^3} 1.762 (4), endocyclic S—C_{sp^2} 1.725 (1), exocyclic S—C_{sp^2} 1.786 (3) and S=O 1.435 (1) Å.

Comment

The one-step thioalkylation of heteroaromatic compounds is a desirable process as it can lead to the formation of heteroaromatic thiols by reduction of the sulfur. Our initial experiments involving benzo[*b*]thiophene and ethyl disulfide afforded 2,3-bis(ethylthio)benzo[*b*]-thiophene, (1), as a yellow oil (Clark, Mesher & Primak, 1996). As identification of (1) by NMR was ambiguous, it was oxidized to the title compound, (2), which has been characterized by X-ray crystallographic methods and is described herein.



(2)

An *ORTEP*II (Johnson, 1976) drawing of the title compound showing the atomic numbering scheme is presented in Fig. 1. The benzothiophene moiety is essentially planar [maximum deviation 0.024 (2) Å for C4], with atoms S2 and S3 of the ethylsulfonyl groups substituted at the 2 and 3 positions lying 0.169 (1) and 0.095 (1) Å, respectively, above and below the plane. The ethylsulfonyl groups are oriented in opposite directions with almost identical torsion angles of C2—S2—C10—C11 58.3 (2)° and C3—S3—C12—C13 57.3 (2)°. The intramolecular repulsions between the two ethylsulfonyl groups are relieved by widening of the angles at the C_{sp^2} atoms C2 and C3, *i.e.* C3—C2—S2 132.5 (2)° and C2—C3—S3 124.3 (2)°. The mean bond distances are S—C_{sp^3} 1.762 (4), endocyclic S—C_{sp^2} 1.725 (1), exocyclic S—C_{sp^2} 1.786 (3) and S=O 1.435 (1). The crystal structure is composed of discrete molecules of (2), with no unusual interactions.

A search of the Cambridge Structural Database (Allen & Kennard, 1993) did not uncover any structures containing the 2,3-dithio-1-benzothiophene moiety.

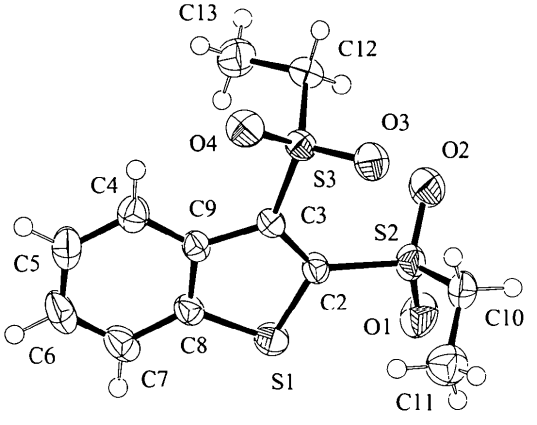


Fig. 1. A view of the title compound with the atomic numbering scheme. The displacement ellipsoids of the non-H atoms are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

Experimental

The title compound was prepared as described previously by Clark, Mesher & Primark (1996) and oxidized to the sulfone employing the method of Aitken, Armstrong & Mesher (1994). Crystals were obtained as colourless prisms by slow evaporation from a $CDCl_3$ solution.

Crystal data

$C_{12}H_{14}O_4S_3$
M_r = 318.42
Triclinic
$P\bar{1}$
a = 8.759 (1) Å
b = 11.745 (3) Å
c = 7.322 (1) Å
α = 91.54 (2)°
β = 111.07 (1)°
γ = 82.00 (2)°
V = 695.8 (2) Å^3
Z = 2
D_x = 1.520 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 12 reflections
θ = 10.0–15.0°
μ = 0.538 mm^{-1}
T = 296 K
Prismatic
0.65 × 0.33 × 0.30 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.968, T_{max} = 1.000
4333 measured reflections
4086 independent reflections
2579 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.0145
θ_{max} = 30.0°
h = 0 → 12
k = −16 → 16
l = −10 → 9
3 standard reflections monitored every 200 reflections
frequency: 180 min
intensity decay: 0.31%

Refinement

Refinement on F
R = 0.0375
wR = 0.0383
S = 2.081
2579 reflections
173 parameters
H atoms riding at geometrically idealized positions with C—H = 0.95 Å
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Extinction correction: Zachariasen (1968) type 2 Gaussian isotropic
Extinction coefficient: 6.05299
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
S1	0.41431 (9)	0.42445 (6)	0.2751 (1)	0.0439 (2)
S2	0.54843 (8)	0.18915 (6)	0.2103 (1)	0.0408 (2)
S3	0.12666 (8)	0.14367 (5)	0.09422 (9)	0.0329 (2)
O1	0.6844 (2)	0.2526 (2)	0.3005 (3)	0.0612 (7)
O2	0.5455 (2)	0.0841 (2)	0.3030 (3)	0.0542 (6)
O3	0.2072 (2)	0.0890 (1)	−0.0312 (2)	0.0446 (5)
O4	−0.0496 (2)	0.1730 (2)	0.0127 (3)	0.0456 (5)
C2	0.3715 (3)	0.2876 (2)	0.2048 (3)	0.0325 (6)
C3	0.2123 (3)	0.2732 (2)	0.1759 (3)	0.0286 (6)
C4	−0.0462 (3)	0.3980 (2)	0.2043 (4)	0.0397 (7)
C5	−0.1066 (3)	0.5056 (3)	0.2445 (4)	0.0484 (8)
C6	−0.0101 (4)	0.5944 (2)	0.2920 (4)	0.0533 (9)
C7	0.1513 (4)	0.5770 (2)	0.3058 (4)	0.0466 (8)
C8	0.2149 (3)	0.4667 (2)	0.2662 (3)	0.0351 (7)
C9	0.1183 (3)	0.3769 (2)	0.2131 (3)	0.0304 (6)
C10	0.5337 (3)	0.1619 (2)	−0.0323 (4)	0.0475 (8)
C11	0.5231 (4)	0.2669 (3)	−0.1518 (5)	0.063 (1)
C12	0.1801 (3)	0.0574 (2)	0.3079 (4)	0.0399 (7)
C13	0.1194 (4)	0.1145 (3)	0.4607 (4)	0.0555 (9)

Table 2. *Selected geometric parameters (Å, °)*

S1—C2	1.724 (2)	S3—O4	1.435 (2)
S1—C8	1.726 (3)	S3—C3	1.783 (2)
S2—O1	1.437 (2)	S3—C12	1.766 (3)
S2—O2	1.432 (2)	C2—C3	1.367 (3)
S2—C2	1.789 (2)	C3—C9	1.450 (3)
S2—C10	1.758 (3)	C10—C11	1.504 (4)
S3—O3	1.435 (2)	C12—C13	1.506 (4)
C2—S1—C8	91.4 (1)	S1—C2—S2	113.9 (1)
O1—S2—O2	118.6 (1)	S1—C2—C3	113.2 (2)
O1—S2—C2	103.8 (1)	S2—C2—C3	132.5 (2)
O1—S2—C10	108.5 (1)	S3—C3—C2	124.3 (2)
O2—S2—C2	108.1 (1)	S3—C3—C9	123.4 (2)
O2—S2—C10	109.1 (1)	C2—C3—C9	112.2 (2)
C2—S2—C10	108.3 (1)	S1—C8—C7	125.7 (2)
O3—S3—O4	117.5 (1)	S1—C8—C9	112.3 (2)
O3—S3—C3	107.5 (1)	C3—C9—C4	130.4 (2)
O3—S3—C12	109.3 (1)	C3—C9—C8	110.9 (2)
O4—S3—C3	107.5 (1)	S2—C10—C11	114.7 (2)
O4—S3—C12	109.0 (1)	S3—C12—C13	113.3 (2)
C3—S3—C12	105.3 (1)		

The space group was determined based on a statistical analysis of intensity distribution and the successful solution and refinement of the structure.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1212). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Fluorene-1-carboxylic Acid

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Abstract

In fluorene-1-carboxylic acid, $C_{14}H_{10}O_2$, the sole hydrogen bond is of the cyclic dimer type about a center of symmetry. The carboxyl H atom is ordered. Distances in the fluorene core are very similar to those in fluorene itself; the fluorene core dihedral angle is, however, larger than in fluorene.

Comment

The structure of fluorene-1-carboxylic acid was of interest both because of the potential for hydrogen bonding and because of the possibility of comparisons of the fluorene core geometry with that of fluorene itself, as determined by Belsky, Zavodnik & Vozzhennikov (1984) at 295 K and Gerkin, Lundstedt & Reppart (1984) at 159 K .

In the present structure (I), hydrogen bonding is of the cyclic dimer type about a center of symmetry. There is only a single hydrogen bond and this has O(1) as donor and O(2^i) as acceptor [symmetry operator: (i) $1-x$, $1-y$, $-z$]; the donor–acceptor distance, 2.632 (4) Å, is well below the average for organic O···O hydro-