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Dispiro(fluorene-9,4′-[1,3]dithiolane-5′,9″-fluorene)

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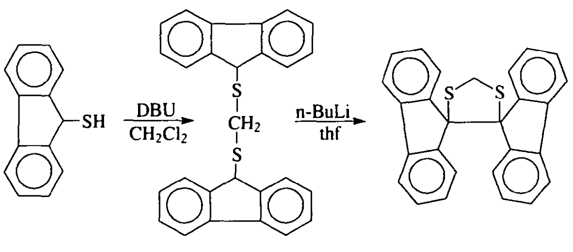
Abstract

The crystal structure of dispiro(fluorene-9,4′-[1,3]dithiolane-5′,9″-fluorene), $C_{27}H_{18}S_2$, is reported. The C4—C5 bond connecting the two fluorenyl groups is 1.580 (4) Å, which is slightly longer than the C—C bond lengths [1.36–1.49 Å] in other 1,3-dithiolanes.

Comment

Only a few X-ray crystal structure determinations of compounds containing the 1,3-dithiolane moiety have been carried out. The structure of the title compound, (2), with atomic numbering scheme is illustrated in Fig. 1.



(1) (2)

The S1—C2, S1—C5, S3—C2 and S3—C4 bond lengths, and the S1—C2—S3, C2—S3—C4 and C5—S1—C2 bond angles are similar to those reported for other dithiolanes (Lynch, Lee, Martin & Davis, 1992; Mentzafors, Polisiou & Georgiadis, 1988; Noordik, Smits, Sicherer-Roetman, Jansen & De Groot, 1985; Dubourg *et al.*, 1984; Barrière, Chiaroni, Cléophax, Géro & Riche, 1984). The C4—C5 bond, however, is slightly longer compared with those of the related 1,3-dithiolanes [1.580 (4) *versus* 1.36–1.49 Å], causing a decrease in the corresponding S3—C4—C5 and S1—C5—C4 bond angles [106.8 (2) and 105.5 (3) compared with 107.5–113.6°]. This may be due to steric effects caused by the large fluorenyl groups attached to C4 and C5. The fluorenyl groups show minimal distortion from ideal geometry, with the C—C distances very close to the expected values for aromatic rings.

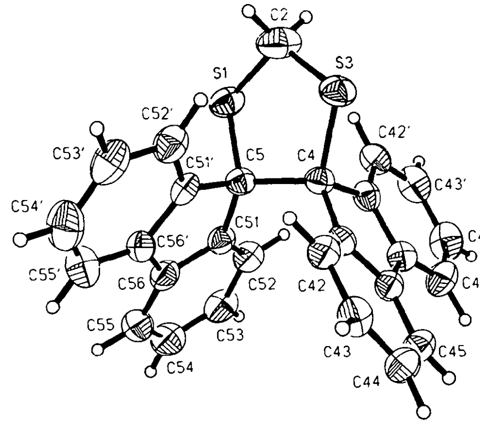


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Bis(fluorenyl-9-thio)methane, (1) (see the scheme above), was prepared by reacting fluorene-9-thiol with 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in dichloromethane. Addition of *n*-butyllithium (2.5 M in hexanes) to (1) in thf gave the title compound (2). Colourless crystals were obtained by crystallization from toluene. The synthesis of (2) from thiofluorenone and diazomethane has been published (Schoenberg, Koenig & Singer, 1967). The NMR spectra of (1) and (2), along with other sulfur-containing 9-substituted fluorenes, are discussed elsewhere (Leino, Luttikhedde, Näsman & Mattinen, 1995).

Crystal data

$C_{27}H_{18}S_2$
$M_r = 406.56$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Triclinic
$P\bar{1}$
$a = 9.299\ (3)$ Å
$b = 9.592\ (4)$ Å
$c = 13.263\ (5)$ Å
$\alpha = 79.42\ (3)°$
$\beta = 69.85\ (3)°$
$\gamma = 62.55\ (3)°$
$V = 985.1\ (6)$ Å^3
$Z = 2$
$D_x = 1.371$ Mg m^{-3}

Cell parameters from 26 reflections
$\theta = 8.8–14.5°$
$\mu = 0.27$ mm^{-1}
$T = 293$ K
Arrowhead
0.4 × 0.25 × 0.1 mm
Colourless

Data collection

Nicolet *R*3*m* diffractometer
ω scans
Absorption correction: none
3499 measured reflections
3499 independent reflections
2070 observed reflections [$F > 4\sigma(F)$]

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 11$
$k = -10 \rightarrow 11$
$l = -14 \rightarrow 15$
2 standard reflections monitored every 98 reflections
intensity decay: 4%

Refinement

Refinement on F
$R = 0.047$
$wR = 0.047$
$S = 1.153$
2070 reflections
262 parameters
$w = 1/[\sigma^2(F) + 0.0005F^2]$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	$U_{\rm eq}$
S1	0.0574 (1)	0.3091 (1)	0.1121 (1)	0.043 (1)
S3	0.4245 (1)	0.2395 (1)	0.0205 (1)	0.042 (1)
C2	0.2393 (6)	0.2565 (6)	−0.0069 (3)	0.060 (3)
C4	0.3367 (4)	0.2536 (4)	0.1668 (3)	0.032 (2)
C5	0.1429 (4)	0.3731 (4)	0.1915 (3)	0.031 (2)
C41	0.4388 (4)	0.3036 (4)	0.2098 (3)	0.031 (2)
C42	0.4824 (4)	0.4286 (5)	0.1782 (3)	0.037 (2)
C43	0.5814 (5)	0.4464 (5)	0.2283 (3)	0.041 (2)
C44	0.6354 (5)	0.3423 (5)	0.3094 (3)	0.047 (2)
C45	0.5952 (5)	0.2158 (5)	0.3395 (3)	0.043 (2)
C46	0.4987 (4)	0.1951 (4)	0.2886 (3)	0.033 (2)
C41′	0.3671 (4)	0.0924 (4)	0.2224 (3)	0.033 (2)
C42′	0.3226 (5)	−0.0203 (5)	0.2072 (3)	0.042 (2)
C43′	0.3582 (5)	−0.1558 (5)	0.2711 (3)	0.049 (2)
C44′	0.4392 (5)	−0.1818 (5)	0.3477 (4)	0.055 (2)
C45′	0.4883 (5)	−0.0703 (5)	0.3605 (3)	0.048 (2)
C46′	0.4530 (4)	0.0655 (4)	0.2969 (3)	0.036 (2)
C51	0.0366 (4)	0.3753 (5)	0.3096 (3)	0.033 (2)
C52	0.0127 (5)	0.2550 (5)	0.3778 (3)	0.042 (2)
C53	−0.0969 (5)	0.2900 (6)	0.4824 (3)	0.050 (2)
C54	−0.1831 (5)	0.4425 (6)	0.5169 (3)	0.053 (2)
C55	−0.1618 (5)	0.5635 (5)	0.4495 (3)	0.051 (2)
C56	−0.0491 (5)	0.5291 (5)	0.3459 (3)	0.039 (2)
C51′	0.1065 (4)	0.5463 (4)	0.1676 (3)	0.034 (2)
C52′	0.1603 (5)	0.6219 (5)	0.0720 (3)	0.044 (2)
C53′	0.1086 (6)	0.7825 (5)	0.0704 (4)	0.054 (3)
C54′	0.0038 (6)	0.8673 (5)	0.1616 (4)	0.061 (3)
C55′	−0.0537 (6)	0.7947 (5)	0.2565 (4)	0.057 (2)
C56′	−0.0032 (5)	0.6350 (5)	0.2583 (3)	0.038 (2)

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

S1—C2	1.809 (4)	S3—C2	1.810 (6)
S1—C5	1.841 (5)	S3—C4	1.832 (3)
C4—C41	1.533 (7)	C5—C51	1.535 (4)
C4—C41′	1.528 (5)	C5—C51′	1.529 (6)
C41—C46	1.397 (5)	C51—C56	1.399 (6)
C41′—C46′	1.393 (6)	C51′—C56′	1.393 (5)
C46—C46′	1.463 (7)	C56—C56′	1.479 (6)
C4—C5	1.580 (4)		
C2—S1—C5	97.4 (2)	C2—S3—C4	97.7 (2)
S1—C5—C4	105.5 (3)	S3—C4—C5	106.8 (2)
S1—C5—C51	107.6 (3)	S3—C4—C41	109.5 (3)
S1—C5—C51′	111.0 (3)	S3—C4—C41′	111.8 (3)
C4—C5—C51	115.0 (3)	C5—C4—C41	113.6 (3)
C4—C5—C51′	115.6 (3)	C5—C4—C41′	113.7 (3)
C51—C5—C51′	102.0 (3)	C41—C4—C41′	101.5 (3)
C5—C51—C56	109.7 (3)	C4—C41—C46	109.9 (4)
C5—C51′—C56′	110.6 (3)	C4—C41′—C46′	110.1 (4)
C51—C56—C56′	109.2 (3)	C41—C46—C46′	109.0 (4)
C56—C56′—C51′	108.4 (4)	C46—C46′—C41′	109.0 (3)
S1—C2—S3	111.2 (3)		

The structure was solved by direct methods and refined using the *SHELXTL-Plus* (Sheldrick, 1990) program package on a MicroVAX computer. Non-H atoms were treated as anisotropic. H atoms were placed in idealized positions and constrained to C—H = 0.96 Å with isotropic displacement parameters $U = 0.06$ Å^2.

Data collection: *P3/V Data Collection Software* (Siemens, 1989). Cell refinement: *P3/V Data Collection Software*. Data reduction: *SHELXTL-Plus*. Structure solution: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Preparation of material for publication: *SHELXTL-Plus*.

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Lists of structure factors, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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