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4,5,4′,5′-Tetrakis(2-methyl-thiophen-3-yl)-[2,2′]bi[[1,3]dithiolylidene]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.009 Å
R factor = 0.072
wR factor = 0.138
Data-to-parameter ratio = 11.4

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

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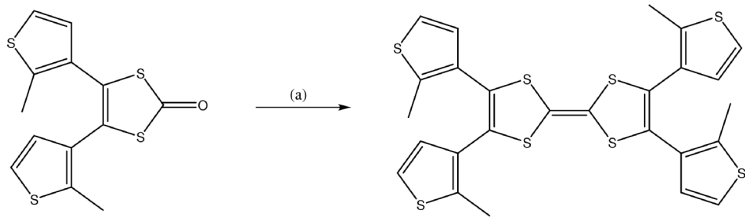
The structure of the title compound, $C_{26}H_{20}S_8$, has been established by X-ray crystallography. In space group $C2/c$, the asymmetric unit contains one molecule.

Comment

The title compound, (I), was prepared as part of a programme investigating the synthesis of thiophene-substituted tetrathiafulvenes and polymers derived from them. We were particularly interested in the preparation of tetrathiafulvenes in which specific positions of the pendant thiophenes are blocked (Roberts-Bleming *et al.*, 2001). In the structure of (I), both C_4S rings adopt envelope conformations, and C1 and C1′ deviate by 0.323 (6) and 0.267 (7) Å from the respective C_2S_2 mean plane. All thiophene rings are essentially planar. The bond lengths and angles show the expected values. The shortest intermolecular S···S distances are S1···S1(0.5−x, 1.5−y, 1−z) = 3.551 (4) Å, S3···S3′(0.5+ x, 1.5−y, 0.5+z) = 3.570 (3) Å and S4···S4′(0.5+x, 0.5−y, 0.5+z) = 3.556 (3) Å, all of which are somewhat shorter than the sum of van der Waals radii (3.70 Å).



(II) (I)

Experimental

4,5-Bis(2-methylthiophen-3-yl)-[1,3]dithiol-2-one, (II), was treated with triethylphosphite at 373 K for 3 h to give, on work-up, the title compound (I) in 50% yield. Crystals of (I) were isolated as colourless platelets by slow evaporation of a diethyl ether/petroleum ether (40/60) mixture.

Crystal data

$C_{26}H_{20}S_8$
M_r = 588.90
Monoclinic, $C2/c$
a = 27.083 (2) Å
b = 10.0911 (10) Å
c = 20.304 (2) Å
β = 110.212 (3)°
V = 5207.5 (6) Å^3
Z = 8
D_x = 1.502 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 446 reflections
θ = 2.9–23.5°
μ = 0.70 mm^{-1}
T = 150 (2) K
Needle cut from platelet, colourless
0.12 × 0.02 × 0.01 mm

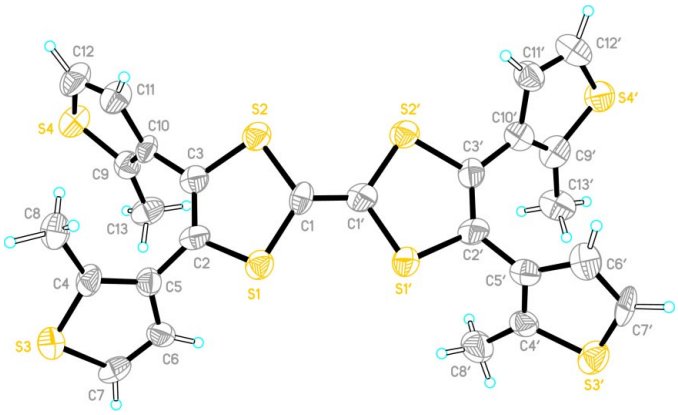


Figure 1
Structure of (I) showing 50% probability displacement ellipsoids.

Data collection

Nonius KappaCCD area detector diffractometer
φ and ω scans to fill Ewald sphere
Absorption correction: multi-scan (Blessing, 1997)
T_{min} = 0.921, T_{max} = 0.997
11 126 measured reflections
3519 independent reflections
1961 reflections with $I > 2\sigma(I)$
R_{int} = 0.117
θ_{max} = 23.5°
$h = -29 \rightarrow 25$
$k = -8 \rightarrow 11$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.138
S = 1.09
3519 reflections
308 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*).

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