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The π-donor 4,5;4′,5′-bis(dioxane-1,4-diyl-2,3-dithio)tetrathiafulvalene

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The title molecule, $C_{14}H_{12}O_4S_8$, has a U-shaped conformation and its crystal structure feaures short intermolecular S$\cdots$S contacts.

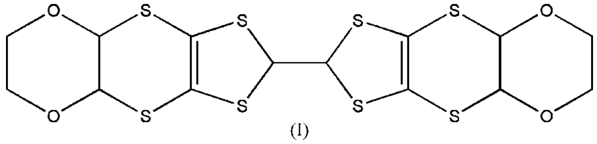
Key indicators

Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.004 Å
R factor = 0.026
wR factor = 0.060
Data-to-parameter ratio = 11.6

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

Comment

Radical cation salts based on bis(ethylenedithio)tetrathiafulvalene (BEDT–TTF) are a major class of organic quasi-two-dimensional organic metals and superconductors (Williams *et al.*, 1992; Ishiguro *et al.*, 1998). In a search for new systems with high conductivity, we have synthesized the BEDT–TTF derivative containing two 1,4-dioxane rings [BDDT–TTF; (I)] and their salts (Kotov *et al.*, 1994; Faulmann *et al.*, 1995). Subsequently, improved syntheses of (I) have been reported (Kini *et al.*, 1995; Yamada *et al.*, 1998). The crystal structure of the (BDDT–TTF)$_2$I$_3$ salt has been investigated (Kini *et al.*, 1995), but that of (I) has not yet been solved owing to difficulties in obtaining single crystals.



(I)

Neutral (I) (Fig. 1) has a U-shaped configuration, as shown in Fig. 2. By contrast, the donor in the aforementioned (BDDT–TTF)$_2$I$_3$ salt adopts a chair conformation, with the dioxane substituents oriented on opposite sides of the plane through the TTF portion of the molecule (Kini *et al.*, 1995). It is possible that molecular rearrangement occurs during oxidation of (I) to form (BDDT–TTF)$_2$I$_3$. The packing as shown in Figs. 3 and 4 is made of pairs of I because of short van der Waals S$\cdots$S contacts (<3.7 Å), partly due to the U-shaped conformation of the neutral molecule.

Experimental

Compound (I) was prepared according to the reported methods of Kotov *et al.* (1994, 1999) and purified by column chromatography.

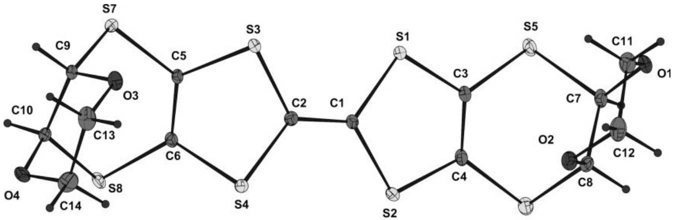


Figure 1
View of (I) showing the atomic labelling. Displacement ellipsoids are drawn at the 30% probability level.

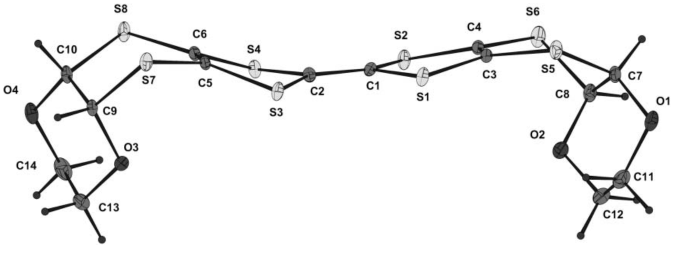


Figure 2
Side view of (I) showing the atomic labelling. Displacement ellipsoids are drawn at the 30% probability level.

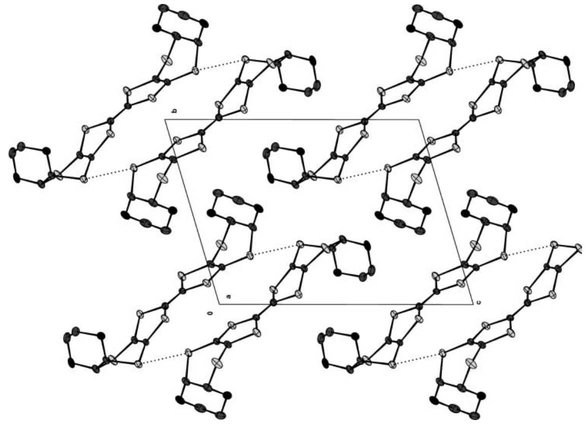


Figure 3
Packing of (I), projected down the *a* axis. Short intradimer S···S interactions (<3.7 Å) are shown as dotted lines. H atoms have been omitted.

Crystals were grown by the slow evaporation, at room temperature, of a toluene and dichloromethane (1:1) solution of (I).

Crystal data

$C_{14}H_{12}O_4S_8$
$M_r = 500.72$
Triclinic, $P\overline{1}$
$a = 6.3867$ (7) Å
$b = 11.0716$ (13) Å
$c = 14.1462$ (16) Å
$\alpha = 106.009$ (11)°
$\beta = 90.107$ (12)°
$\gamma = 103.453$ (12)°
$V = 932.78$ (18) Å^3

$Z = 2$
$D_x = 1.783$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8000 reflections
$\theta = 1.7$–26.1°
$\mu = 0.98$ mm^{-1}
$T = 180$ K
Plate, orange–yellow
0.5 × 0.3 × 0.02 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.712$, $T_{\max} = 0.979$
7398 measured reflections
2733 independent reflections

2225 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 24.1°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

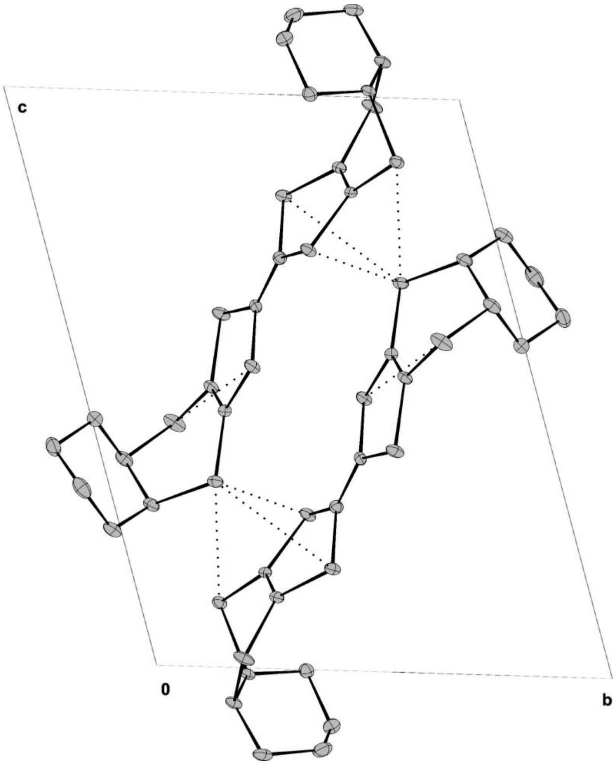


Figure 4
Mode of overlap of the pairs of (I). Dotted lines indicate the short S···S contacts (<3.7 Å). H atoms have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.060$
$S = 0.97$
2733 reflections
235 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

The H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined as riding on their parent atoms, with $U_{\rm iso}(H) = 1.2U_{\rm eq}$(carrier). The fixed φ spindle of the Stoe *IPDS* does not allow easy access to the cusp of data along the mount axis and hence $\theta_{\max}$ was restricted to 24.1°.

Data collection: *IPDS Software* (Stoe, 1996); cell refinement: *IPDS Software*; data reduction: *X-RED* (Stoe, 1996); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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