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3,5-Dibromo-2-[2,5-dibutoxy-4-(3,5-dibromothiophen-2-yl)phenyl]thiophene

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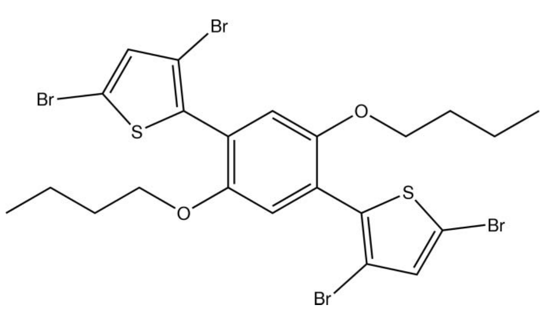
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.084; data-to-parameter ratio = 17.0.

The title molecule, $C_{22}H_{22}Br_4O_2S_2$, is centrosymmetric with an inversion centre located at the centre of the benzene ring. The 3,5-dibromothiophene groups are twisted relative to the benzene ring, making a dihedral angle of 41.43 (9)°.

Related literature

The title compound belongs to the family of arylthiophenes, compounds frequently used as electroluminescent oligomers to produce polymers for LED applications. For a related structure and background references, see: Promarak & Ruchirawat (2007); Huang *et al.* (2007). For related structures, see: Li *et al.* (2008); Kuriger *et al.* (2008); Ali *et al.* (2008).



Experimental

Crystal data

$C_{22}H_{22}Br_4O_2S_2$	V = 1220.78 (5) Å^3
M_r = 702.16	Z = 2
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
a = 13.0156 (3) Å	μ = 9.79 mm^{-1}
b = 7.8157 (2) Å	T = 150 K
c = 12.2264 (2) Å	0.24 × 0.10 × 0.07 mm
β = 101.027 (2)°	

Data collection

Oxford Diffraction Gemini diffractometer	12067 measured reflections
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)	2349 independent reflections
T_{min} = 0.202, T_{max} = 0.547	2272 reflections with $I > 2\sigma(I)$
	R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	138 parameters
$wR(F^2)$ = 0.084	H-atom parameters constrained
S = 1.10	$\Delta\rho_{max}$ = 0.98 e Å^{-3}
2349 reflections	$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2422).

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3,5-Dibromo-2-[2,5-dibutoxy-4-(3,5-dibromothiophen-2-yl)phenyl]thiophene

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S1. Comment

Brominated thiophene-phenylene oligomer with enhanced solubility characteristics due to the presence of alkyloxy substituents such as in the title compound, (I) is an important intermediate to engineer soluble electroluminescent oligomers and polymers for LED applications (Huang *et al.*, 2007).

The structure of I is centrosymmetric with an inversion centre located at the centre of the benzene ring. The mean plane of the central unit [O1/C1/C5/C6/C7/C8/C9/O1A/C1A/C5A/C6A/C7A/C8A/C9A] (A) is approximately planar with the highest deviation of ±0.023 (2)° for atoms O1/O1A and the 3,5-dibromothiophene rings are twisted relative to the plane forming a dihedral angle of 41.43 (9)°. Half of the butyloxy groups lie above/below the mean plane A and the mean planes of [C8C9C10C11A] and [C8AC9AC10AC11A] make a dihedral angle of 59.5 (3)° with A. The torsion angle C8-C9-C10-C11 is 179.7 (3)° and this conformation does not allow for stacking interactions of the aromatic units. Thus quenching of the luminescent effect for polymer generated from this oligomer can be avoided (Fig. 2).

S2. Experimental

The title compound was prepared according to previously published procedure (Promarak & Ruchirawat, 2007) with a slight modification. *N*-Bromosuccinimide (0.58 g, 3.26 mmol) was added into a solution of 1,4-bis(thiophen-2-yl)-2,5-bis(butyloxy)benzene (0.60 g, 1.55 mmol) in THF:DMF (*v*/*v*=1:1). The mixture was heated under reflux overnight and allowed to cool to ambient temperature prior to addition of water. The compound was extracted into dichloromethane, washed with water and brine solution, dried over anhydrous $MgSO_4$ and the solvent was removed by evaporation. Recrystallization of the product from hot dichloromethane solution afforded crystals suitable for single-crystal X-ray diffraction (yield: 63%; m.p. 417-419 K).

S3. Refinement

The hydrogen positions were calculated geometrically and refined in a riding model approximation with C–H bond lengths in the range 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and CH_2 group, and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl group.

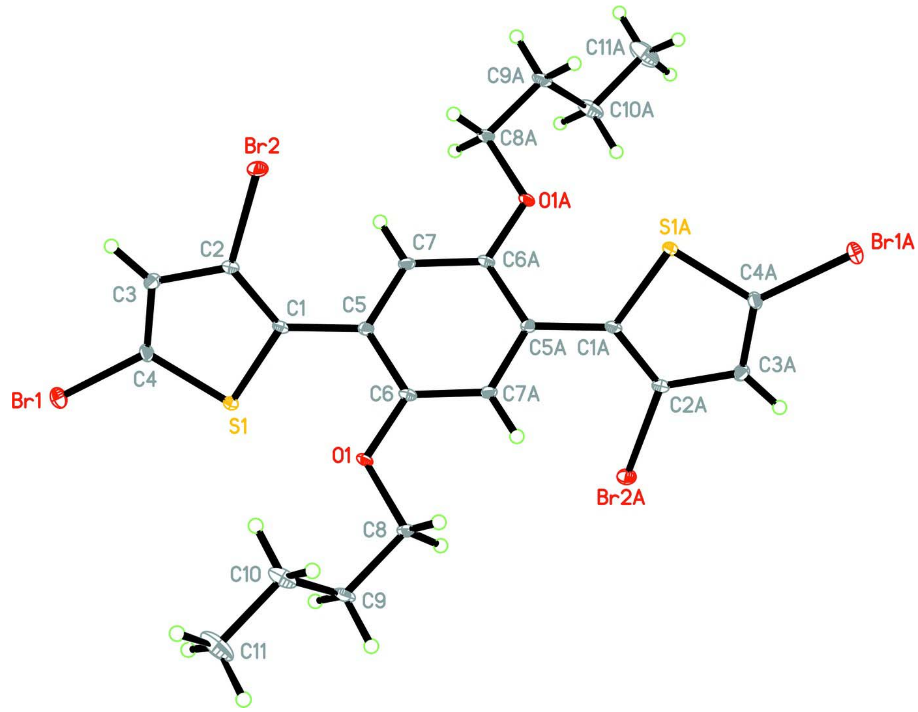

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Symmetry code for atoms with the A label: -*x*, 1 - *y*, 1 - *z*.

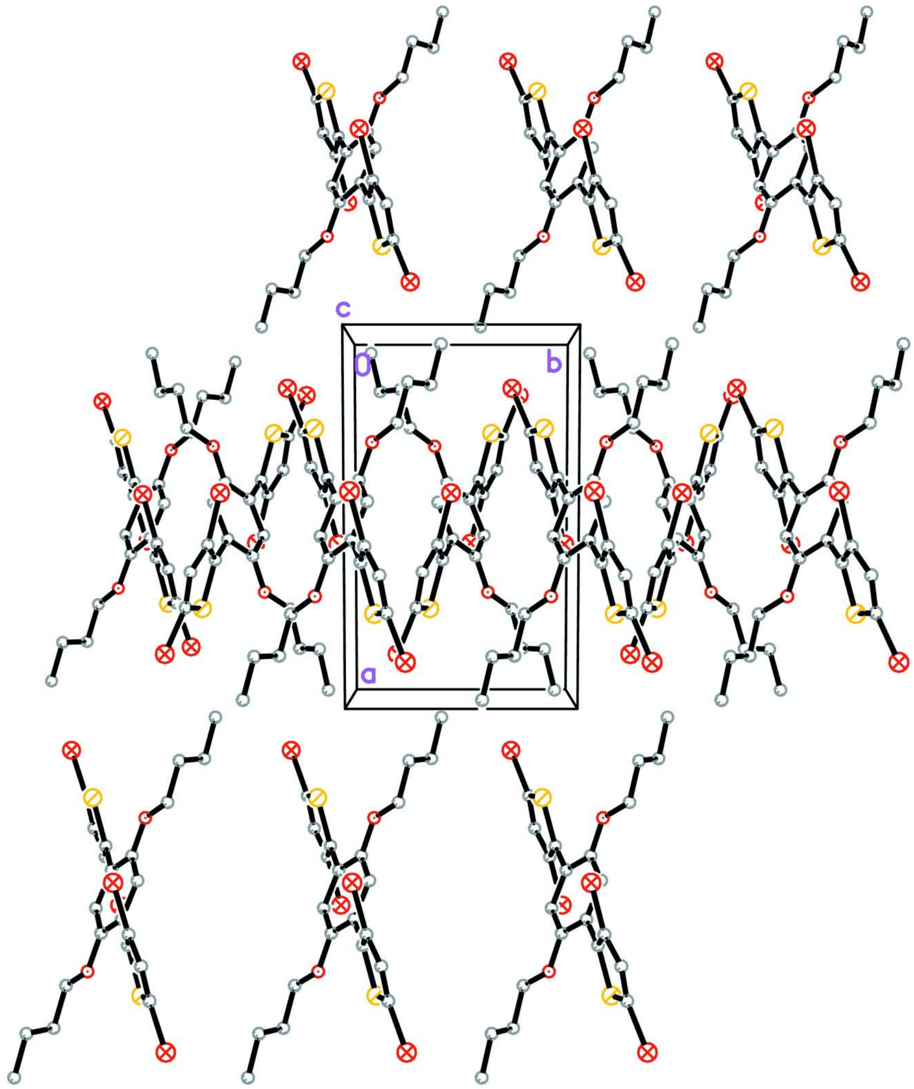

Figure 2

Crystal packing of the title compound viewed down the *c*-axis.

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Crystal data

$C_{22}H_{22}Br_4O_2S_2$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
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b = 7.8157 (2) Å
c = 12.2264 (2) Å
β = 101.027 (2)°
V = 1220.78 (5) Å^3
Z = 2

F(000) = 684
D_x = 1.910 Mg m^{-3}
Melting point = 417–419 K
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7985 reflections
θ = 3–71°
μ = 9.79 mm^{-1}
T = 150 K
Prismatic, yellow
0.24 × 0.10 × 0.07 mm

Data collection

Oxford Diffraction Gemini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.202$, $T_{max} = 0.547$
12067 measured reflections
2349 independent reflections
2272 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 71.0°$, $\theta_{min} = 3.5°$
$h = -15 \rightarrow 15$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 1.10$
2349 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.8587P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.98$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0057 (3)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat (Cosier & Glazer, 1986) with a nominal stability of 0.1 K.
(Cosier, J. & Glazer, A.M., 1986. J. Appl. Cryst. 105 107.)

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	0.85564 (2)	0.22645 (4)	0.97981 (2)	0.02292 (14)
Br2	0.43160 (2)	0.45620 (4)	0.82956 (2)	0.01904 (14)
S1	0.74092 (5)	0.36060 (9)	0.74910 (5)	0.01532 (18)
O1	0.70059 (14)	0.6197 (2)	0.57663 (15)	0.0142 (4)
C1	0.60951 (19)	0.4183 (3)	0.7216 (2)	0.0119 (5)
C2	0.56937 (19)	0.3982 (3)	0.8164 (2)	0.0122 (5)
C3	0.6403 (2)	0.3333 (3)	0.9103 (2)	0.0143 (5)
H3	0.6239	0.3128	0.9799	0.017*
C4	0.7353 (2)	0.3055 (3)	0.8837 (2)	0.0143 (5)
C5	0.5550 (2)	0.4615 (3)	0.6079 (2)	0.0115 (5)
C6	0.6011 (2)	0.5643 (3)	0.5364 (2)	0.0113 (5)
C7	0.4545 (2)	0.3963 (3)	0.5690 (2)	0.0125 (5)

H7	0.4243	0.3250	0.6150	0.015*
C8	0.7491 (2)	0.7284 (3)	0.5067 (2)	0.0158 (5)
H8A	0.7077	0.8311	0.4876	0.019*
H8B	0.7549	0.6692	0.4384	0.019*
C9	0.8561 (2)	0.7736 (4)	0.5713 (3)	0.0193 (6)
H9A	0.8917	0.8437	0.5247	0.023*
H9B	0.8963	0.6692	0.5884	0.023*
C10	0.8543 (2)	0.8692 (4)	0.6796 (3)	0.0278 (7)
H10A	0.8147	0.9742	0.6627	0.033*
H10B	0.8186	0.7995	0.7264	0.033*
C11	0.9630 (3)	0.9124 (5)	0.7433 (4)	0.0411 (9)
H11A	1.0016	0.8086	0.7632	0.062*
H11B	0.9574	0.9744	0.8097	0.062*
H11C	0.9987	0.9813	0.6974	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0157 (2)	0.0312 (2)	0.0196 (2)	0.00459 (11)	−0.00230 (13)	0.00655 (11)
Br2	0.01321 (19)	0.0258 (2)	0.0200 (2)	0.00462 (10)	0.00798 (13)	0.00194 (10)
S1	0.0078 (3)	0.0252 (4)	0.0130 (3)	0.0008 (2)	0.0021 (2)	0.0026 (2)
O1	0.0071 (8)	0.0178 (9)	0.0168 (9)	−0.0044 (7)	−0.0001 (7)	0.0041 (7)
C1	0.0075 (11)	0.0121 (11)	0.0160 (13)	−0.0016 (9)	0.0024 (10)	0.0002 (10)
C2	0.0096 (12)	0.0118 (11)	0.0157 (12)	−0.0010 (9)	0.0037 (9)	−0.0015 (10)
C3	0.0155 (13)	0.0152 (13)	0.0130 (12)	0.0008 (10)	0.0045 (10)	0.0012 (10)
C4	0.0131 (13)	0.0151 (12)	0.0133 (12)	0.0009 (10)	−0.0010 (10)	0.0026 (10)
C5	0.0097 (12)	0.0128 (12)	0.0124 (12)	0.0005 (9)	0.0034 (10)	0.0003 (9)
C6	0.0078 (12)	0.0104 (12)	0.0160 (12)	−0.0019 (9)	0.0032 (10)	−0.0024 (9)
C7	0.0099 (12)	0.0130 (12)	0.0157 (12)	−0.0018 (10)	0.0048 (9)	0.0023 (9)
C8	0.0102 (13)	0.0181 (13)	0.0193 (13)	−0.0037 (10)	0.0036 (11)	0.0050 (10)
C9	0.0080 (13)	0.0205 (14)	0.0289 (15)	−0.0028 (10)	0.0025 (11)	0.0040 (11)
C10	0.0136 (15)	0.0281 (16)	0.0406 (19)	−0.0038 (11)	0.0021 (13)	−0.0073 (13)
C11	0.0219 (17)	0.039 (2)	0.057 (2)	−0.0070 (15)	−0.0077 (16)	−0.0178 (18)

Geometric parameters (Å, º)

Br1—C4	1.874 (3)	C7—C6^{i}	1.387 (4)
Br2—C2	1.886 (3)	C7—H7	0.9300
S1—C4	1.716 (3)	C8—C9	1.506 (4)
S1—C1	1.738 (3)	C8—H8A	0.9700
O1—C6	1.365 (3)	C8—H8B	0.9700
O1—C8	1.434 (3)	C9—C10	1.524 (4)
C1—C2	1.368 (4)	C9—H9A	0.9700
C1—C5	1.474 (4)	C9—H9B	0.9700
C2—C3	1.422 (4)	C10—C11	1.517 (4)
C3—C4	1.355 (4)	C10—H10A	0.9700
C3—H3	0.9300	C10—H10B	0.9700
C5—C7	1.400 (4)	C11—H11A	0.9600

C5—C6	1.404 (4)	C11—H11B	0.9600
C6—C7^{i}	1.387 (4)	C11—H11C	0.9600
C4—S1—C1	91.70 (13)	O1—C8—H8A	110.3
C6—O1—C8	117.94 (19)	C9—C8—H8A	110.3
C2—C1—C5	129.2 (2)	O1—C8—H8B	110.3
C2—C1—S1	109.12 (19)	C9—C8—H8B	110.3
C5—C1—S1	121.31 (19)	H8A—C8—H8B	108.5
C1—C2—C3	115.5 (2)	C8—C9—C10	113.8 (2)
C1—C2—Br2	124.6 (2)	C8—C9—H9A	108.8
C3—C2—Br2	119.89 (19)	C10—C9—H9A	108.8
C4—C3—C2	110.2 (2)	C8—C9—H9B	108.8
C4—C3—H3	124.9	C10—C9—H9B	108.8
C2—C3—H3	124.9	H9A—C9—H9B	107.7
C3—C4—S1	113.4 (2)	C11—C10—C9	112.8 (3)
C3—C4—Br1	126.5 (2)	C11—C10—H10A	109.0
S1—C4—Br1	120.03 (15)	C9—C10—H10A	109.0
C7—C5—C6	118.7 (2)	C11—C10—H10B	109.0
C7—C5—C1	119.1 (2)	C9—C10—H10B	109.0
C6—C5—C1	122.2 (2)	H10A—C10—H10B	107.8
O1—C6—C7^{i}	123.7 (2)	C10—C11—H11A	109.5
O1—C6—C5	116.5 (2)	C10—C11—H11B	109.5
C7^{i}—C6—C5	119.8 (2)	H11A—C11—H11B	109.5
C6^{i}—C7—C5	121.5 (2)	C10—C11—H11C	109.5
C6^{i}—C7—H7	119.2	H11A—C11—H11C	109.5
C5—C7—H7	119.2	H11B—C11—H11C	109.5
O1—C8—C9	107.2 (2)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···Br2	0.93	2.80	3.289 (2)	114