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1,2-Bis[2-*n*-butyl-5-(2,2-dicyanovinyl)-3-thienyl]-3,3,4,4,5,5-hexafluorocyclopent-1-ene: a new photochromic diarylethene compound

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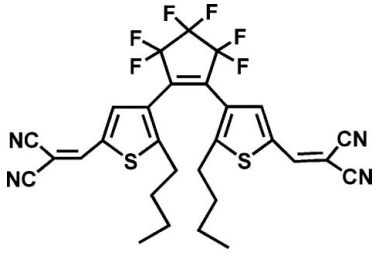
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.109; data-to-parameter ratio = 14.7.

In the title compound, $C_{29}H_{22}F_6N_4S_2$, a new symmetric photochromic dithienylethene, the distance between the two reactive C atoms in the molecule is 3.475 (3) Å. The dihedral angles between the central cyclopentene ring and the two thiophene rings are 57.9 (1) and 51.2 (1)°. The molecule has no imposed crystallographic symmetry.

Related literature

For related literature, see: Irie (2000); Pu *et al.* (2003); Pu, Liu, Chen & Xu (2005); Pu, Yang, Wang & Xu (2005); Ramamurthy & Venkatesan (1987); Tian & Yang (2004); Shibata *et al.* (2002); Yamaguchi & Irie (2006).



Experimental

Crystal data

$C_{29}H_{22}F_6N_4S_2$
M_r = 604.63
Monoclinic, $C2/c$
a = 20.8617 (17) Å
b = 8.9036 (7) Å
c = 31.734 (3) Å
β = 91.801 (10)°
V = 5891.4 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 291 (2) K
0.41 × 0.25 × 0.17 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.907, T_{max} = 0.960
21870 measured reflections
5473 independent reflections
3854 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.109
S = 1.01
5473 reflections
372 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C13—C17	1.342 (3)	C15—C16	1.527 (3)
C13—C14	1.501 (3)	C16—C17	1.501 (3)
C14—C15	1.530 (3)		
C1—C5—C6—C7	−176.5 (3)	C3—C13—C17—C19	4.9 (4)
C1—C5—C6—C8	2.9 (4)	C14—C13—C17—C16	2.9 (3)
C3—C4—C9—C10	−122.6 (3)	C15—C16—C17—C13	−12.3 (3)
C9—C10—C11—C12	−179.2 (3)	C15—C16—C17—C19	169.3 (2)
C13—C14—C15—C16	−14.5 (3)	C19—C18—C26—C27	−129.4 (2)
C14—C15—C16—C17	16.1 (2)	C26—C27—C28—C29	−177.5 (3)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2066).

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supplementary materials

Acta Cryst. (2007). E**63**, o2578 [**doi:10.1107/S1600536807018909**]

1,2-Bis[2-*n*-butyl-5-(2,2-dicyanovinyl)-3-thienyl]-3,3,4,4,5,5-hexafluorocyclopent-1-ene: a new photochromic diarylethene compound

C.-H. Zheng, S.-Z. Pu, Z.-G. Le, M.-B. Luo and D.-C. Huang

Comment

Photochromic diarylethenes have attracted considerable attention for their possible application in optical recording and photoswitches (Irie, 2000; Tian & Yang, 2004). For further background information, see Pu, Liu *et al.* (2005). In the present work, the title photochromic diarylethene, (Ia), was synthesized, and its structure is presented here. Previously, we have reported the structure of the 2-methyl, (II), and 2-ethyl analog, (III), of this compound (Pu *et al.*, 2003; Pu, Yang *et al.*, 2005). In order to investigate the substituent effect at the 2-position of the thiophene on the photochemical properties, we have now determined the structure of (Ia).

The molecular structure of (Ia) is shown in Fig. 2. In the cyclopent-1-ene ring, the C13—C17 bond is clearly a double bond, while the other bonds in the ring are clearly single bonds (Table 1). The two thiophene rings are linked by the C13=C17 double bond. The two *n*-butyl groups are located on opposite sides of the double bond and are directed *trans* relative to the thiophene planes, as reflected in the torsion angles C3—C4—C9—C10 and C19—C18—C26—C27 (Table 1). The dihedral angle between the central cyclopent-1-ene ring and each adjacent thiophene ring are 57.9 (1)° for S1/C1—C4 and 51.2 (1)° for S2/C18—C21. The corresponding values in the methyl analog, (II), and that in the ethyl analog, (III), are 44.9° and 48.0 (2)°, respectively. This conformation leads to a C4—C18 separation of 3.475 (3) Å in (Ia) [compared with 3.589 Å in (II) and 3.642 (7) in (III)]. This distance is short enough, theoretically, for a ring-closure reaction to take place in the crystalline phase to generate compound (Ib) (see scheme), (Ramamurthy &Venkatesan, 1987; Shibata *et al.*, 2002; Yamaguchi & Irie, 2006).

Crystals of (Ia) show photochromism in accordance with the expected ring closure, to form (Ib). Upon irradiation with 365 nm light, the colorless single crystals of (Ia) turned green quickly. When the green crystal was dissolved in dichloromethane, the solution also showed a green color, with an absorption maximum at 772 nm, consistent with the presence of the closed-ring isomer, (Ib). Upon irradiation with visible light with wavelength greater than 510 nm, the green crystal can return to its initial colorless state, and the absorption spectrum of the dichloromethane solution containing the colorless crystal is the same as that of solution of the open-ring form, (Ia), with the absorption maximum at 366 nm.

Experimental

The title compound, (Ia), was synthesized in 29% total yield by the literature method (Pu, Yang *et al.*, 2005) using 5-*n*-butylthiophene- 2-carbaldehyde as the start material. Crystal suitable for X-ray analysis were grown from a solution (diethyl ether/hexane 1/3) by slow evaporation at room temperature (m.p. 403 K).

Refinement

All H atoms were placed in calculated positions, with C—H distances of 0.93 Å (aromatic), 0.97 Å (CH_2) and 0.96 Å (CH_3). They were included in the refinement in the riding model approximation with isotropic displacement parameters set equal to $1.2U_{eq}$ of the carrier atom for the aromatic_H, and $1.5U_{eq}$ of the carrier for CH_3.

Figures

Fig. 1. The photochromism scheme of the title compound.

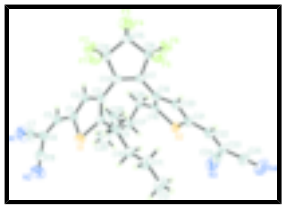


Fig. 2. The structure of compound (Ia) with 35% probability ellipsoids, showing the atomic numbering scheme.

1,2-Bis[5-(2,2-dicyanovinyl)-2-*n*-butyl-3-thienyl]-3,3,4,4,5,5- hexafluorocyclopent-1-ene

Crystal data

$C_{29}H_{22}F_6N_4S_2$	$D_x = 1.363$ Mg m^{-3}
$M_r = 604.63$	Melting point: 403 K
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 20.8617$ (17) Å	Cell parameters from 4441 reflections
$b = 8.9036$ (7) Å	$\theta = 2.3$–21.4º
$c = 31.734$ (3) Å	$\mu = 0.24$ mm^{-1}
$\beta = 91.801$ (10)º	$T = 291$ (2) K
$V = 5891.4$ (8) Å^3	Block, yellow
$Z = 8$	0.41 × 0.25 × 0.17 mm
$F_{000} = 2480$	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	5473 independent reflections
Radiation source: fine-focus sealed tube	3854 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.036$
$T = 291(2)$ K	$\theta_{max} = 25.5$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -25\rightarrow25$
$T_{min} = 0.907$, $T_{max} = 0.960$	$k = -10\rightarrow10$

21870 measured reflections

$l = -38 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.109$

$S = 1.01$

5473 reflections

372 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 3.9867P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.39$ e Å^{-3}

$\Delta\rho_{min} = -0.19$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
S1	0.34614 (3)	0.94519 (7)	0.065367 (18)	0.05087 (18)
S2	0.14181 (3)	0.60393 (7)	0.140966 (18)	0.04928 (17)
F1	0.42234 (9)	0.9638 (2)	0.21678 (5)	0.1019 (7)
F2	0.45948 (7)	0.7450 (3)	0.20272 (5)	0.0980 (6)
F3	0.40242 (8)	0.8486 (2)	0.28890 (5)	0.0826 (5)
F4	0.40837 (8)	0.62479 (19)	0.26488 (5)	0.0826 (5)
F5	0.28255 (7)	0.86621 (19)	0.27031 (4)	0.0741 (5)
F6	0.28808 (8)	0.62501 (19)	0.26922 (4)	0.0763 (5)
N1	0.51996 (15)	0.6334 (3)	−0.04852 (9)	0.0993 (9)
N2	0.39603 (15)	1.0267 (4)	−0.03025 (8)	0.0936 (9)
N3	−0.00732 (13)	0.5521 (4)	0.11051 (9)	0.0982 (9)
N4	−0.10277 (13)	0.8596 (4)	0.19571 (10)	0.1049 (10)
C1	0.40108 (11)	0.8007 (3)	0.07207 (7)	0.0466 (6)
C2	0.40147 (11)	0.7500 (3)	0.11275 (7)	0.0478 (6)
H2	0.4282	0.6732	0.1226	0.057*

C3	0.35760 (10)	0.8250 (3)	0.13843 (7)	0.0430 (5)
C4	0.32340 (10)	0.9344 (3)	0.11691 (7)	0.0441 (5)
C5	0.44063 (11)	0.7407 (3)	0.04009 (7)	0.0540 (6)
H5	0.4642	0.6560	0.0480	0.065*
C6	0.44910 (12)	0.7877 (3)	0.00030 (8)	0.0553 (6)
C7	0.48911 (15)	0.7033 (3)	−0.02698 (9)	0.0710 (8)
C8	0.41933 (14)	0.9200 (4)	−0.01715 (8)	0.0641 (7)
C9	0.27335 (11)	1.0394 (3)	0.13239 (7)	0.0502 (6)
H9A	0.2893	1.1414	0.1304	0.060*
H9B	0.2666	1.0185	0.1619	0.060*
C10	0.20903 (11)	1.0292 (3)	0.10836 (7)	0.0565 (6)
H10A	0.2155	1.0516	0.0789	0.068*
H10B	0.1933	0.9270	0.1100	0.068*
C11	0.15891 (13)	1.1340 (4)	0.12458 (10)	0.0752 (8)
H11A	0.1532	1.1124	0.1542	0.090*
H11B	0.1748	1.2361	0.1226	0.090*
C12	0.09494 (14)	1.1258 (4)	0.10217 (11)	0.1019 (12)
H12A	0.0998	1.1470	0.0728	0.153*
H12B	0.0666	1.1982	0.1140	0.153*
H12C	0.0774	1.0269	0.1053	0.153*
C13	0.35057 (10)	0.7935 (2)	0.18383 (7)	0.0421 (5)
C14	0.40648 (12)	0.8170 (3)	0.21406 (7)	0.0543 (6)
C15	0.38374 (12)	0.7624 (3)	0.25679 (7)	0.0521 (6)
C16	0.31102 (11)	0.7497 (3)	0.25091 (7)	0.0457 (5)
C17	0.29797 (10)	0.7518 (2)	0.20414 (6)	0.0399 (5)
C18	0.22207 (10)	0.6157 (2)	0.15356 (6)	0.0418 (5)
C19	0.23383 (10)	0.7151 (2)	0.18654 (6)	0.0404 (5)
C20	0.17678 (11)	0.7758 (3)	0.20215 (7)	0.0462 (6)
H20	0.1761	0.8417	0.2249	0.055*
C21	0.12248 (11)	0.7285 (3)	0.18066 (7)	0.0471 (6)
C22	0.05937 (11)	0.7794 (3)	0.18969 (8)	0.0541 (6)
H22	0.0572	0.8483	0.2116	0.065*
C23	0.00241 (11)	0.7422 (3)	0.17107 (8)	0.0552 (6)
C24	−0.00339 (12)	0.6364 (4)	0.13732 (9)	0.0655 (7)
C25	−0.05601 (14)	0.8085 (4)	0.18485 (9)	0.0718 (8)
C26	0.26755 (11)	0.5151 (3)	0.13119 (7)	0.0479 (6)
H26A	0.2590	0.4119	0.1390	0.058*
H26B	0.3110	0.5387	0.1408	0.058*
C27	0.26367 (12)	0.5275 (3)	0.08337 (7)	0.0570 (6)
H27A	0.2200	0.5077	0.0736	0.068*
H27B	0.2744	0.6293	0.0753	0.068*
C28	0.30844 (15)	0.4190 (3)	0.06202 (8)	0.0733 (8)
H28A	0.3518	0.4358	0.0728	0.088*
H28B	0.2965	0.3171	0.0693	0.088*
C29	0.30730 (18)	0.4343 (5)	0.01458 (9)	0.1064 (13)
H29A	0.2647	0.4148	0.0035	0.160*
H29B	0.3366	0.3634	0.0030	0.160*
H29C	0.3198	0.5343	0.0071	0.160*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0526 (4)	0.0588 (4)	0.0414 (3)	0.0081 (3)	0.0049 (3)	0.0056 (3)
S2	0.0442 (3)	0.0554 (4)	0.0481 (3)	−0.0065 (3)	−0.0014 (3)	−0.0082 (3)
F1	0.1225 (16)	0.0941 (14)	0.0867 (12)	−0.0608 (12)	−0.0356 (11)	0.0229 (10)
F2	0.0430 (9)	0.185 (2)	0.0652 (10)	0.0228 (11)	−0.0041 (7)	0.0040 (11)
F3	0.0709 (11)	0.1161 (14)	0.0602 (9)	−0.0181 (10)	−0.0103 (8)	−0.0263 (9)
F4	0.0750 (11)	0.0842 (12)	0.0885 (11)	0.0232 (9)	0.0001 (9)	0.0244 (9)
F5	0.0682 (10)	0.0998 (12)	0.0539 (9)	0.0237 (9)	−0.0059 (7)	−0.0243 (8)
F6	0.0842 (11)	0.0892 (12)	0.0547 (9)	−0.0293 (9)	−0.0103 (8)	0.0258 (8)
N1	0.109 (2)	0.101 (2)	0.090 (2)	0.0179 (18)	0.0436 (18)	−0.0081 (17)
N2	0.110 (2)	0.101 (2)	0.0704 (17)	0.0258 (19)	0.0174 (15)	0.0171 (16)
N3	0.0700 (18)	0.124 (3)	0.101 (2)	−0.0288 (17)	−0.0049 (15)	−0.0350 (19)
N4	0.0583 (17)	0.139 (3)	0.118 (2)	0.0177 (18)	0.0008 (16)	−0.018 (2)
C1	0.0423 (13)	0.0531 (14)	0.0447 (13)	−0.0005 (11)	0.0036 (10)	−0.0010 (11)
C2	0.0414 (13)	0.0498 (14)	0.0522 (14)	0.0042 (11)	0.0024 (11)	0.0033 (11)
C3	0.0385 (12)	0.0487 (13)	0.0417 (12)	−0.0049 (10)	0.0002 (10)	0.0014 (10)
C4	0.0413 (12)	0.0479 (13)	0.0432 (12)	0.0002 (10)	0.0015 (10)	0.0014 (10)
C5	0.0477 (14)	0.0600 (16)	0.0548 (15)	0.0027 (12)	0.0059 (11)	−0.0040 (12)
C6	0.0505 (15)	0.0651 (17)	0.0509 (15)	−0.0013 (13)	0.0107 (12)	−0.0076 (13)
C7	0.0716 (19)	0.078 (2)	0.0648 (17)	0.0029 (16)	0.0234 (15)	−0.0023 (15)
C8	0.0659 (18)	0.080 (2)	0.0469 (15)	0.0015 (16)	0.0151 (13)	−0.0006 (14)
C9	0.0534 (15)	0.0513 (14)	0.0459 (13)	0.0057 (12)	0.0026 (11)	0.0009 (11)
C10	0.0506 (15)	0.0700 (17)	0.0489 (14)	0.0081 (13)	−0.0001 (11)	−0.0022 (12)
C11	0.0549 (17)	0.084 (2)	0.086 (2)	0.0157 (15)	−0.0056 (15)	−0.0149 (17)
C12	0.059 (2)	0.138 (3)	0.108 (3)	0.025 (2)	−0.0073 (18)	−0.004 (2)
C13	0.0406 (13)	0.0443 (13)	0.0410 (12)	0.0017 (10)	−0.0036 (10)	0.0009 (10)
C14	0.0430 (14)	0.0665 (17)	0.0531 (15)	−0.0053 (13)	−0.0042 (11)	0.0030 (12)
C15	0.0550 (15)	0.0575 (16)	0.0431 (13)	0.0005 (12)	−0.0099 (11)	−0.0020 (11)
C16	0.0479 (14)	0.0505 (14)	0.0387 (12)	−0.0021 (11)	−0.0012 (10)	0.0006 (11)
C17	0.0393 (12)	0.0414 (12)	0.0388 (11)	−0.0003 (10)	−0.0019 (9)	−0.0009 (9)
C18	0.0437 (13)	0.0435 (13)	0.0382 (11)	−0.0035 (10)	0.0003 (10)	0.0013 (10)
C19	0.0410 (12)	0.0442 (13)	0.0361 (11)	−0.0020 (10)	0.0000 (9)	0.0012 (9)
C20	0.0449 (13)	0.0514 (14)	0.0424 (12)	−0.0058 (11)	0.0030 (10)	−0.0074 (10)
C21	0.0419 (13)	0.0516 (14)	0.0480 (13)	−0.0038 (11)	0.0039 (10)	−0.0050 (11)
C22	0.0488 (15)	0.0588 (16)	0.0548 (14)	−0.0026 (12)	0.0031 (11)	−0.0063 (12)
C23	0.0419 (14)	0.0617 (16)	0.0621 (16)	−0.0050 (12)	0.0022 (12)	0.0007 (13)
C24	0.0413 (15)	0.084 (2)	0.0705 (18)	−0.0159 (14)	−0.0027 (13)	−0.0030 (16)
C25	0.0457 (16)	0.091 (2)	0.079 (2)	0.0015 (16)	−0.0016 (14)	−0.0063 (17)
C26	0.0490 (14)	0.0460 (14)	0.0488 (13)	−0.0002 (11)	0.0010 (11)	−0.0024 (11)
C27	0.0535 (15)	0.0689 (17)	0.0487 (14)	−0.0014 (13)	0.0015 (11)	−0.0082 (12)
C28	0.078 (2)	0.085 (2)	0.0579 (17)	0.0066 (16)	0.0129 (14)	−0.0123 (15)
C29	0.113 (3)	0.146 (4)	0.062 (2)	0.007 (3)	0.0155 (19)	−0.024 (2)

Geometric parameters (Å, °)

S1—C4	1.720 (2)	C11—H11B	0.9700

S1—C1	1.732 (2)	C12—H12A	0.9600
S2—C18	1.712 (2)	C12—H12B	0.9600
S2—C21	1.736 (2)	C12—H12C	0.9600
F1—C14	1.350 (3)	C13—C17	1.342 (3)
F2—C14	1.337 (3)	C13—C14	1.501 (3)
F3—C15	1.324 (3)	C14—C15	1.530 (3)
F4—C15	1.350 (3)	C15—C16	1.527 (3)
F5—C16	1.353 (3)	C16—C17	1.501 (3)
F6—C16	1.348 (3)	C17—C19	1.470 (3)
N1—C7	1.139 (3)	C18—C19	1.387 (3)
N2—C8	1.140 (4)	C18—C26	1.499 (3)
N3—C24	1.136 (3)	C19—C20	1.411 (3)
N4—C25	1.139 (3)	C20—C21	1.370 (3)
C1—C2	1.367 (3)	C20—H20	0.9300
C1—C5	1.431 (3)	C21—C22	1.430 (3)
C2—C3	1.412 (3)	C22—C23	1.352 (3)
C2—H2	0.9300	C22—H22	0.9300
C3—C4	1.376 (3)	C23—C24	1.429 (4)
C3—C13	1.480 (3)	C23—C25	1.435 (4)
C4—C9	1.496 (3)	C26—C27	1.521 (3)
C5—C6	1.347 (3)	C26—H26A	0.9700
C5—H5	0.9300	C26—H26B	0.9700
C6—C7	1.434 (4)	C27—C28	1.518 (3)
C6—C8	1.434 (4)	C27—H27A	0.9700
C9—C10	1.525 (3)	C27—H27B	0.9700
C9—H9A	0.9700	C28—C29	1.511 (4)
C9—H9B	0.9700	C28—H28A	0.9700
C10—C11	1.504 (3)	C28—H28B	0.9700
C10—H10A	0.9700	C29—H29A	0.9600
C10—H10B	0.9700	C29—H29B	0.9600
C11—C12	1.494 (4)	C29—H29C	0.9600
C11—H11A	0.9700		
C4—S1—C1	92.46 (11)	F4—C15—C16	109.1 (2)
C18—S2—C21	92.25 (11)	F3—C15—C14	113.9 (2)
C2—C1—C5	124.0 (2)	F4—C15—C14	109.3 (2)
C2—C1—S1	110.21 (17)	C16—C15—C14	104.47 (18)
C5—C1—S1	125.75 (18)	F6—C16—F5	105.58 (18)
C1—C2—C3	113.8 (2)	F6—C16—C17	112.39 (18)
C1—C2—H2	123.1	F5—C16—C17	111.82 (18)
C3—C2—H2	123.1	F6—C16—C15	111.92 (19)
C4—C3—C2	112.6 (2)	F5—C16—C15	109.65 (19)
C4—C3—C13	123.5 (2)	C17—C16—C15	105.58 (18)
C2—C3—C13	123.9 (2)	C13—C17—C19	128.82 (19)
C3—C4—C9	129.5 (2)	C13—C17—C16	110.65 (19)
C3—C4—S1	110.93 (16)	C19—C17—C16	120.51 (19)
C9—C4—S1	119.58 (16)	C19—C18—C26	129.7 (2)
C6—C5—C1	130.1 (2)	C19—C18—S2	111.33 (16)
C6—C5—H5	115.0	C26—C18—S2	118.78 (16)
C1—C5—H5	115.0	C18—C19—C20	112.23 (19)

C5—C6—C7	119.9 (3)	C18—C19—C17	124.45 (19)
C5—C6—C8	123.3 (2)	C20—C19—C17	123.30 (19)
C7—C6—C8	116.8 (2)	C21—C20—C19	113.6 (2)
N1—C7—C6	178.4 (3)	C21—C20—H20	123.2
N2—C8—C6	178.5 (3)	C19—C20—H20	123.2
C4—C9—C10	114.3 (2)	C20—C21—C22	123.7 (2)
C4—C9—H9A	108.7	C20—C21—S2	110.51 (17)
C10—C9—H9A	108.7	C22—C21—S2	125.76 (18)
C4—C9—H9B	108.7	C23—C22—C21	129.6 (2)
C10—C9—H9B	108.7	C23—C22—H22	115.2
H9A—C9—H9B	107.6	C21—C22—H22	115.2
C11—C10—C9	113.7 (2)	C22—C23—C24	122.8 (2)
C11—C10—H10A	108.8	C22—C23—C25	120.6 (2)
C9—C10—H10A	108.8	C24—C23—C25	116.6 (2)
C11—C10—H10B	108.8	N3—C24—C23	179.3 (3)
C9—C10—H10B	108.8	N4—C25—C23	179.2 (4)
H10A—C10—H10B	107.7	C18—C26—C27	114.51 (19)
C12—C11—C10	115.3 (3)	C18—C26—H26A	108.6
C12—C11—H11A	108.5	C27—C26—H26A	108.6
C10—C11—H11A	108.5	C18—C26—H26B	108.6
C12—C11—H11B	108.5	C27—C26—H26B	108.6
C10—C11—H11B	108.5	H26A—C26—H26B	107.6
H11A—C11—H11B	107.5	C28—C27—C26	112.7 (2)
C11—C12—H12A	109.5	C28—C27—H27A	109.1
C11—C12—H12B	109.5	C26—C27—H27A	109.1
H12A—C12—H12B	109.5	C28—C27—H27B	109.1
C11—C12—H12C	109.5	C26—C27—H27B	109.1
H12A—C12—H12C	109.5	H27A—C27—H27B	107.8
H12B—C12—H12C	109.5	C29—C28—C27	113.4 (3)
C17—C13—C3	128.9 (2)	C29—C28—H28A	108.9
C17—C13—C14	111.24 (19)	C27—C28—H28A	108.9
C3—C13—C14	119.80 (19)	C29—C28—H28B	108.9
F2—C14—F1	106.2 (2)	C27—C28—H28B	108.9
F2—C14—C13	113.2 (2)	H28A—C28—H28B	107.7
F1—C14—C13	111.1 (2)	C28—C29—H29A	109.5
F2—C14—C15	111.6 (2)	C28—C29—H29B	109.5
F1—C14—C15	109.5 (2)	H29A—C29—H29B	109.5
C13—C14—C15	105.40 (19)	C28—C29—H29C	109.5
F3—C15—F4	106.16 (19)	H29A—C29—H29C	109.5
F3—C15—C16	113.8 (2)	H29B—C29—H29C	109.5
C4—S1—C1—C2	−0.82 (19)	F3—C15—C16—F5	20.4 (3)
C4—S1—C1—C5	178.9 (2)	F4—C15—C16—F5	138.75 (19)
C5—C1—C2—C3	−179.1 (2)	C14—C15—C16—F5	−104.5 (2)
S1—C1—C2—C3	0.6 (3)	F3—C15—C16—C17	141.0 (2)
C1—C2—C3—C13	−178.1 (2)	F4—C15—C16—C17	−100.6 (2)
C2—C3—C4—C9	−179.3 (2)	C14—C15—C16—C17	16.1 (2)
C13—C3—C4—C9	−1.2 (4)	C3—C13—C17—C19	4.9 (4)
C2—C3—C4—S1	−0.6 (2)	C14—C13—C17—C19	−178.8 (2)
C13—C3—C4—S1	177.43 (17)	C3—C13—C17—C16	−173.4 (2)

C1—S1—C4—C3	0.81 (18)	C14—C13—C17—C16	2.9 (3)
C1—S1—C4—C9	179.64 (19)	F6—C16—C17—C13	−134.6 (2)
C2—C1—C5—C6	−173.6 (3)	F5—C16—C17—C13	106.9 (2)
S1—C1—C5—C6	6.8 (4)	C15—C16—C17—C13	−12.3 (3)
C1—C5—C6—C7	−176.5 (3)	F6—C16—C17—C19	47.0 (3)
C1—C5—C6—C8	2.9 (4)	F5—C16—C17—C19	−71.6 (3)
C3—C4—C9—C10	−122.6 (3)	C15—C16—C17—C19	169.3 (2)
S1—C4—C9—C10	58.8 (3)	C21—S2—C18—C19	−1.47 (17)
C4—C9—C10—C11	179.3 (2)	C21—S2—C18—C26	174.30 (18)
C9—C10—C11—C12	−179.2 (3)	C26—C18—C19—C20	−172.8 (2)
C4—C3—C13—C17	59.4 (3)	S2—C18—C19—C20	2.4 (2)
C2—C3—C13—C17	−122.8 (3)	C26—C18—C19—C17	5.9 (4)
C4—C3—C13—C14	−116.6 (3)	S2—C18—C19—C17	−178.88 (17)
C2—C3—C13—C14	61.2 (3)	C13—C17—C19—C18	50.1 (3)
C17—C13—C14—F2	129.9 (2)	C16—C17—C19—C18	−131.8 (2)
C3—C13—C14—F2	−53.5 (3)	C13—C17—C19—C20	−131.3 (3)
C17—C13—C14—F1	−110.8 (2)	C16—C17—C19—C20	46.8 (3)
C3—C13—C14—F1	65.8 (3)	C18—C19—C20—C21	−2.3 (3)
C17—C13—C14—C15	7.6 (3)	C17—C19—C20—C21	179.0 (2)
C3—C13—C14—C15	−175.7 (2)	C19—C20—C21—C22	−177.5 (2)
F2—C14—C15—F3	97.4 (3)	C19—C20—C21—S2	1.1 (3)
F1—C14—C15—F3	−19.8 (3)	C18—S2—C21—C20	0.19 (19)
C13—C14—C15—F3	−139.3 (2)	C18—S2—C21—C22	178.8 (2)
F2—C14—C15—F4	−21.1 (3)	C20—C21—C22—C23	179.9 (3)
F1—C14—C15—F4	−138.4 (2)	S2—C21—C22—C23	1.5 (4)
C13—C14—C15—F4	102.1 (2)	C21—C22—C23—C24	1.0 (4)
F2—C14—C15—C16	−137.8 (2)	C21—C22—C23—C25	−179.2 (3)
F1—C14—C15—C16	105.0 (2)	C19—C18—C26—C27	−129.4 (2)
C13—C14—C15—C16	−14.5 (3)	S2—C18—C26—C27	55.7 (3)
F3—C15—C16—F6	−96.4 (2)	C18—C26—C27—C28	−177.5 (2)
F4—C15—C16—F6	21.9 (3)	C26—C27—C28—C29	−177.5 (3)
C14—C15—C16—F6	138.7 (2)		

Fig. 1

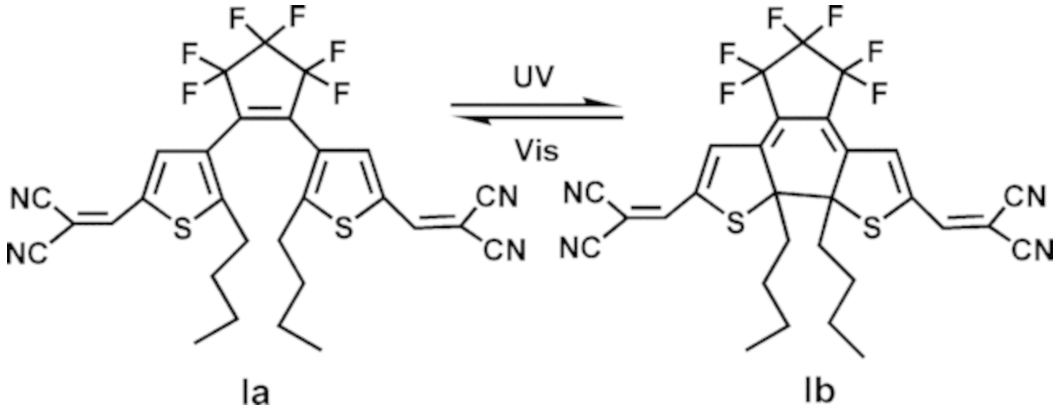
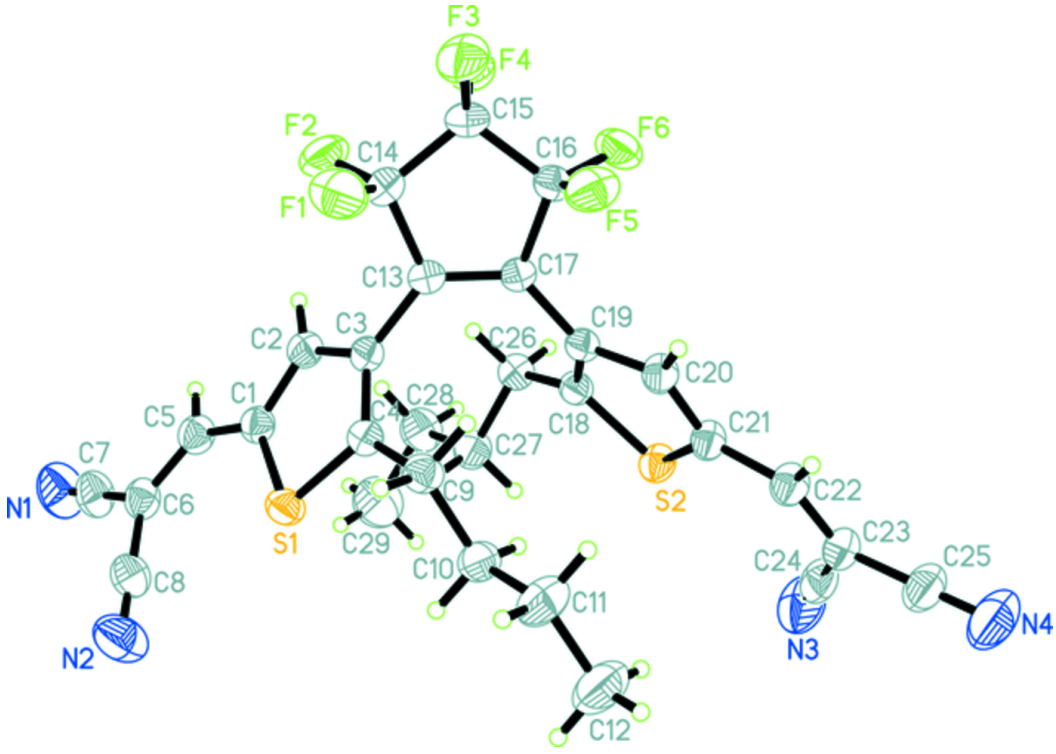


Fig. 2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