

1-[5-(3-Chlorophenyl)-2-methyl-3-thienyl]-3,3,4,4,5,5-hexafluoro-2-(2-methoxyphenyl)cyclopent-1-ene

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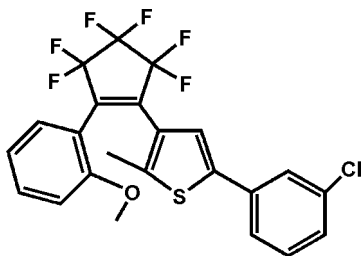
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.040; wR factor = 0.111; data-to-parameter ratio = 12.5.

The title compound, $\text{C}_{23}\text{H}_{15}\text{ClF}_6\text{OS}$, has thienyl and phenylene substituents on the double-bond C atoms of the envelope-shaped cyclopentenyl ring. The aromatic systems are aligned at 55.3 (4) (thienyl) and 60.8 (7)° (phenylene) with respect to the planar $\text{C}=\text{C}=\text{C}$ portion of the main central cyclopentenyl ring.

Related literature

For the synthesis of the precursors and related compounds, see: Fan *et al.* (2008, 2009); Pu *et al.* (2008).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{15}\text{ClF}_6\text{OS}$	$\gamma = 80.639$ (1)°
$M_r = 488.86$	$V = 1080.7$ (2) Å ³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.4057$ (10) Å	Mo $K\alpha$ radiation
$b = 10.2900$ (11) Å	$\mu = 0.34$ mm ⁻¹
$c = 11.9548$ (13) Å	$T = 291$ K
$\alpha = 83.529$ (1)°	$0.49 \times 0.45 \times 0.33$ mm
$\beta = 71.564$ (1)°	

Data collection

Bruker SMART CCD area-detector diffractometer	8254 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3997 independent reflections
$T_{\min} = 0.851$, $T_{\max} = 0.896$	3358 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	553 restraints
$wR(F^2) = 0.111$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.28$ e Å ⁻³
3997 reflections	$\Delta\rho_{\text{min}} = -0.36$ e Å ⁻³
321 parameters	

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2571).

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supplementary materials

Acta Cryst. (2009). E65, o1105 [doi:10.1107/S1600536809013993]

1-[5-(3-Chlorophenyl)-2-methyl-3-thienyl]-3,3,4,4,5,5-hexafluoro-2-(2-methoxyphenyl)cyclopent-1-ene

C. Fan, W. Liu, G. Liu and T. Yang

Comment

Crystal of the compound when dissolved in hexane can show photochromism. Upon irradiation with 365 nm light, the colorless crystals hexane solution turns blue rapidly. The blue compound displays an absorption maximum at 584 nm. Upon irradiation with visible light with a wavelength greater than 510 nm, the blue crystals hexane solution revert to their initial colorless state; a hexane solution has an absorption maximum at 291 nm. In a polymethylmethacrylate amorphous film, the title diarylethene also demonstrates photochromism as similar as that in hexane.

Experimental

The title compound was synthesized from the reaction of (2-methoxyphenyl)perfluorocyclopent-1-ene (1.43 g, 4.75 mmol) (Fan *et al.*, 2009) and 3-bromo-2-methyl-5-(3-chlorophenyl)thiophene (1.44 g, 5 mmol) (Fan *et al.*, 2008) with *n*-butyllithium (2.0 ml, 5 mmol) at 195 K under a nitrogen atmosphere. After an hour, the reaction was quenched by the addition of water. The solid product was purified by column chromatography on silica with petroleum ether as the eluent to give the title compound 1.58 g (3.23 mmol) in 68% yield. C&H elemental analysis. Calc. for C₂₃H₁₅ClF₆OS: C 56.51, H, 3.09%. Found C 55.92, H 3.15%.

Refinement

All H atoms attached to C were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl) or 0.93 Å (aromatic) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl})$. The cyclopent-1-ene ring in C9, C10, C11-environment conformation is disorder, the C8—C9, C8—C9', C12—C11 and C12—C11' distance was strained to 1.50 (1) Å, respectively. The distance of C9—F1, C9—F2, F9'—F1', C9'—F2', C10—F3, C10—F4, C10'—F3', C10'—F4', C11—F5, C11—F6, C11'—F5' and C11—F6' was restrained to 1.34 (1) Å, respectively. The occupancies of the disorder components refined to a 0.784 (3):0.216 (3) ratio for C9:C9', F1:F1' and F2:F2'. The occupancies of the disorder components refined to a 0.834 (5):0.166 (5) ratio for C11:C11', F5:F5', F6:F6' and 0.826 (2):0.174 (2) ratio for C10:C10', F3:F3', F4:F4'.

Figures

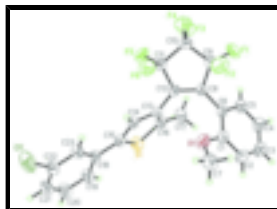


Fig. 1. Molecular view the atom-labelling scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

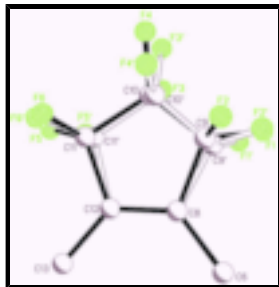


Fig. 2. The disorder in cyclopent-1-ene ring.

1-[5-(3-Chlorophenyl)-2-methyl-3-thienyl]-3,3,4,4,5,5- hexafluoro-2-(2-methoxyphenyl)cyclopent-1-ene

Crystal data

$C_{23}H_{15}ClF_6OS$

$M_r = 488.86$

Triclinic, $P\bar{1}$

$a = 9.4057 (10) \text{ \AA}$

$b = 10.2900 (11) \text{ \AA}$

$c = 11.9548 (13) \text{ \AA}$

$\alpha = 83.5290 (10)^\circ$

$\beta = 71.5640 (10)^\circ$

$\gamma = 80.6390 (10)^\circ$

$V = 1080.7 (2) \text{ \AA}^3$

$Z = 2$

$F_{000} = 496$

$D_x = 1.502 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4346 reflections

$\theta = 2.5\text{--}28.1^\circ$

$\mu = 0.34 \text{ mm}^{-1}$

$T = 291 \text{ K}$

Block, colourless

$0.49 \times 0.45 \times 0.33 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291 \text{ K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.851$, $T_{\max} = 0.896$

8254 measured reflections

3997 independent reflections

3358 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.011$

$\theta_{\max} = 25.5^\circ$

$\theta_{\min} = 2.5^\circ$

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.111$

$S = 1.03$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.4496P]$

where $P = (F_o^2 + 2F_c^2)/3$

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

3997 reflections $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$
 321 parameters $\Delta\rho_{\min} = -0.36 \text{ e \AA}^{-3}$
 553 restraints Extinction correction: none
 Primary atom site location: structure-invariant direct methods

Special details

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C9	0.7086 (3)	0.7630 (3)	0.5031 (3)	0.0560 (8)	0.784 (3)
F1	0.5898 (3)	0.8609 (2)	0.5303 (2)	0.0930 (9)	0.784 (3)
F2	0.7750 (3)	0.7584 (2)	0.58991 (15)	0.0854 (7)	0.784 (3)
C9'	0.6784 (10)	0.7854 (12)	0.4846 (8)	0.0560 (8)	0.216 (3)
F1'	0.5498 (9)	0.8402 (8)	0.4640 (8)	0.0930 (9)	0.216 (3)
F2'	0.6890 (12)	0.8415 (10)	0.5755 (6)	0.0854 (7)	0.216 (3)
C10	0.6576 (3)	0.6302 (3)	0.5013 (3)	0.0566 (7)	0.826 (2)
F3	0.52853 (19)	0.6483 (2)	0.4715 (2)	0.0893 (6)	0.826 (2)
F4	0.6283 (3)	0.56317 (19)	0.60649 (15)	0.0877 (6)	0.826 (2)
C10'	0.6811 (14)	0.6391 (15)	0.5180 (12)	0.0566 (7)	0.174 (2)
F3'	0.5428 (9)	0.6053 (11)	0.5606 (12)	0.0893 (6)	0.174 (2)
F4'	0.7538 (10)	0.5982 (9)	0.5974 (7)	0.0877 (6)	0.174 (2)
C11	0.7876 (3)	0.5608 (3)	0.4040 (3)	0.0504 (6)	0.834 (5)
F5	0.7345 (3)	0.4861 (3)	0.34450 (17)	0.0778 (7)	0.834 (5)
F6	0.8815 (3)	0.4760 (2)	0.4529 (2)	0.0771 (7)	0.834 (5)
C11'	0.7621 (15)	0.5759 (12)	0.4012 (12)	0.0504 (6)	0.166 (5)
F5'	0.6680 (14)	0.5518 (13)	0.3457 (9)	0.0778 (7)	0.166 (5)
F6'	0.8458 (15)	0.4630 (13)	0.4176 (13)	0.0771 (7)	0.166 (5)
C11	1.52883 (10)	0.09741 (8)	0.13406 (7)	0.1001 (3)	
S1	1.10823 (7)	0.64743 (6)	-0.01005 (5)	0.05725 (18)	
O1	1.11475 (18)	0.81945 (15)	0.35023 (17)	0.0710 (5)	
C1	1.0232 (2)	0.9304 (2)	0.32810 (19)	0.0521 (5)	
C2	1.0714 (3)	1.0534 (2)	0.2928 (2)	0.0672 (6)	
H2	1.1715	1.0634	0.2819	0.081*	
C3	0.9705 (3)	1.1608 (2)	0.2740 (2)	0.0705 (7)	
H3	1.0039	1.2427	0.2500	0.085*	
C4	0.8232 (3)	1.1490 (2)	0.2899 (2)	0.0650 (6)	

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H4	0.7562	1.2222	0.2777	0.078*
C5	0.7744 (3)	1.0270 (2)	0.32442 (19)	0.0539 (5)
H5	0.6738	1.0188	0.3356	0.065*
C6	0.8730 (2)	0.91614 (18)	0.34282 (16)	0.0444 (4)
C7	1.2362 (3)	0.8347 (3)	0.3900 (2)	0.0758 (7)
H7A	1.3143	0.8689	0.3256	0.114*
H7B	1.2755	0.7506	0.4198	0.114*
H7C	1.2017	0.8949	0.4518	0.114*
C8	0.8185 (2)	0.78631 (19)	0.38147 (17)	0.0442 (4)
C12	0.8636 (2)	0.67244 (18)	0.32766 (16)	0.0413 (4)
C13	0.9725 (2)	0.64498 (17)	0.21130 (16)	0.0419 (4)
C14	1.0915 (2)	0.53693 (18)	0.19382 (17)	0.0440 (4)
H14	1.1096	0.4798	0.2554	0.053*
C15	1.1763 (2)	0.52489 (19)	0.07887 (17)	0.0461 (4)
C16	0.9669 (2)	0.71461 (19)	0.10727 (18)	0.0488 (5)
C17	0.8561 (3)	0.8294 (2)	0.0852 (2)	0.0666 (6)
H17A	0.7603	0.8263	0.1451	0.100*
H17B	0.8439	0.8248	0.0090	0.100*
H17C	0.8932	0.9104	0.0873	0.100*
C18	1.3086 (2)	0.4285 (2)	0.02687 (17)	0.0480 (4)
C19	1.3943 (3)	0.4423 (2)	-0.0917 (2)	0.0658 (6)
H19	1.3666	0.5128	-0.1399	0.079*
C20	1.5198 (3)	0.3523 (3)	-0.1381 (2)	0.0772 (7)
H20	1.5763	0.3638	-0.2170	0.093*
C21	1.5622 (3)	0.2472 (3)	-0.0706 (2)	0.0681 (6)
H21	1.6467	0.1868	-0.1025	0.082*
C22	1.4773 (3)	0.2320 (2)	0.0462 (2)	0.0594 (5)
C23	1.3525 (2)	0.3205 (2)	0.09609 (19)	0.0531 (5)
H23	1.2977	0.3084	0.1754	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C9	0.0574 (15)	0.0530 (16)	0.0522 (14)	-0.0082 (12)	-0.0075 (11)	-0.0065 (12)
F1	0.0854 (14)	0.0621 (11)	0.0886 (17)	0.0065 (10)	0.0275 (13)	-0.0103 (11)
F2	0.1147 (17)	0.1028 (17)	0.0470 (9)	-0.0445 (13)	-0.0213 (10)	-0.0054 (10)
C9'	0.0574 (15)	0.0530 (16)	0.0522 (14)	-0.0082 (12)	-0.0075 (11)	-0.0065 (12)
F1'	0.0854 (14)	0.0621 (11)	0.0886 (17)	0.0065 (10)	0.0275 (13)	-0.0103 (11)
F2'	0.1147 (17)	0.1028 (17)	0.0470 (9)	-0.0445 (13)	-0.0213 (10)	-0.0054 (10)
C10	0.0539 (14)	0.0575 (13)	0.0548 (14)	-0.0176 (11)	-0.0095 (10)	0.0055 (11)
F3	0.0540 (9)	0.0936 (14)	0.1218 (17)	-0.0148 (9)	-0.0273 (11)	-0.0044 (12)
F4	0.1098 (16)	0.0752 (11)	0.0590 (10)	-0.0282 (11)	0.0021 (10)	0.0153 (8)
C10'	0.0539 (14)	0.0575 (13)	0.0548 (14)	-0.0176 (11)	-0.0095 (10)	0.0055 (11)
F3'	0.0540 (9)	0.0936 (14)	0.1218 (17)	-0.0148 (9)	-0.0273 (11)	-0.0044 (12)
F4'	0.1098 (16)	0.0752 (11)	0.0590 (10)	-0.0282 (11)	0.0021 (10)	0.0153 (8)
C11	0.0549 (14)	0.0419 (12)	0.0553 (11)	-0.0108 (11)	-0.0182 (10)	0.0030 (9)
F5	0.0961 (16)	0.0636 (15)	0.0790 (10)	-0.0429 (13)	-0.0152 (10)	-0.0131 (10)
F6	0.0748 (13)	0.0608 (10)	0.0836 (16)	0.0013 (9)	-0.0233 (9)	0.0277 (10)

C11'	0.0549 (14)	0.0419 (12)	0.0553 (11)	-0.0108 (11)	-0.0182 (10)	0.0030 (9)
F5'	0.0961 (16)	0.0636 (15)	0.0790 (10)	-0.0429 (13)	-0.0152 (10)	-0.0131 (10)
F6'	0.0748 (13)	0.0608 (10)	0.0836 (16)	0.0013 (9)	-0.0233 (9)	0.0277 (10)
C11	0.0996 (6)	0.0937 (5)	0.0895 (5)	0.0389 (4)	-0.0325 (4)	0.0023 (4)
S1	0.0699 (4)	0.0557 (3)	0.0415 (3)	0.0014 (3)	-0.0171 (2)	0.0026 (2)
O1	0.0581 (9)	0.0508 (9)	0.1132 (14)	-0.0043 (7)	-0.0380 (9)	-0.0118 (9)
C1	0.0571 (12)	0.0424 (10)	0.0589 (12)	-0.0093 (9)	-0.0176 (10)	-0.0084 (9)
C2	0.0677 (14)	0.0549 (13)	0.0837 (16)	-0.0232 (11)	-0.0220 (12)	-0.0052 (12)
C3	0.0964 (19)	0.0432 (12)	0.0748 (16)	-0.0224 (12)	-0.0259 (14)	0.0020 (11)
C4	0.0873 (17)	0.0440 (12)	0.0642 (14)	-0.0038 (11)	-0.0281 (12)	0.0024 (10)
C5	0.0603 (12)	0.0479 (11)	0.0533 (12)	-0.0045 (9)	-0.0188 (10)	-0.0024 (9)
C6	0.0521 (11)	0.0388 (10)	0.0421 (10)	-0.0067 (8)	-0.0127 (8)	-0.0059 (8)
C7	0.0712 (16)	0.0849 (18)	0.0764 (17)	-0.0021 (14)	-0.0305 (13)	-0.0147 (14)
C8	0.0458 (10)	0.0430 (10)	0.0450 (10)	-0.0086 (8)	-0.0144 (8)	-0.0029 (8)
C12	0.0436 (10)	0.0388 (9)	0.0451 (10)	-0.0074 (8)	-0.0181 (8)	-0.0002 (8)
C13	0.0484 (10)	0.0366 (9)	0.0436 (10)	-0.0088 (8)	-0.0171 (8)	-0.0016 (7)
C14	0.0510 (11)	0.0389 (9)	0.0429 (10)	-0.0050 (8)	-0.0171 (8)	0.0006 (8)
C15	0.0516 (11)	0.0424 (10)	0.0457 (10)	-0.0070 (8)	-0.0169 (9)	-0.0019 (8)
C16	0.0577 (12)	0.0431 (10)	0.0471 (11)	-0.0033 (9)	-0.0203 (9)	-0.0015 (8)
C17	0.0837 (17)	0.0573 (13)	0.0593 (13)	0.0119 (12)	-0.0337 (12)	-0.0014 (11)
C18	0.0510 (11)	0.0471 (11)	0.0461 (10)	-0.0060 (8)	-0.0142 (9)	-0.0067 (8)
C19	0.0787 (16)	0.0590 (13)	0.0490 (12)	-0.0009 (11)	-0.0087 (11)	-0.0029 (10)
C20	0.0825 (17)	0.0754 (17)	0.0548 (14)	-0.0005 (14)	0.0032 (12)	-0.0114 (12)
C21	0.0601 (14)	0.0703 (15)	0.0658 (15)	0.0051 (11)	-0.0086 (11)	-0.0218 (12)
C22	0.0572 (13)	0.0586 (13)	0.0627 (13)	0.0036 (10)	-0.0226 (11)	-0.0089 (10)
C23	0.0513 (11)	0.0568 (12)	0.0477 (11)	-0.0030 (9)	-0.0115 (9)	-0.0058 (9)

Geometric parameters (Å, °)

C9—F1	1.359 (3)	C3—H3	0.9300
C9—F2	1.363 (4)	C4—C5	1.381 (3)
C9—C8	1.514 (3)	C4—H4	0.9300
C9—C10	1.525 (4)	C5—C6	1.392 (3)
C9'—F2'	1.324 (9)	C5—H5	0.9300
C9'—F1'	1.332 (9)	C6—C8	1.481 (3)
C9'—C8	1.493 (8)	C7—H7A	0.9600
C9'—C10'	1.512 (9)	C7—H7B	0.9600
C10—F4	1.334 (3)	C7—H7C	0.9600
C10—F3	1.349 (3)	C8—C12	1.342 (3)
C10—C11	1.536 (4)	C12—C13	1.469 (3)
C10'—F3'	1.327 (10)	C13—C16	1.375 (3)
C10'—F4'	1.332 (10)	C13—C14	1.426 (3)
C10'—C11'	1.524 (9)	C14—C15	1.361 (3)
C11—F5	1.348 (3)	C14—H14	0.9300
C11—F6	1.358 (3)	C15—C18	1.472 (3)
C11—C12	1.510 (3)	C16—C17	1.502 (3)
C11'—F6'	1.326 (10)	C17—H17A	0.9600
C11'—F5'	1.328 (10)	C17—H17B	0.9600
C11'—C12	1.500 (9)	C17—H17C	0.9600

supplementary materials

Cl1—C22	1.741 (2)	C18—C19	1.396 (3)
S1—C16	1.718 (2)	C18—C23	1.396 (3)
S1—C15	1.731 (2)	C19—C20	1.380 (3)
O1—C1	1.368 (3)	C19—H19	0.9300
O1—C7	1.405 (3)	C20—C21	1.359 (4)
C1—C2	1.389 (3)	C20—H20	0.9300
C1—C6	1.398 (3)	C21—C22	1.377 (3)
C2—C3	1.383 (4)	C21—H21	0.9300
C2—H2	0.9300	C22—C23	1.376 (3)
C3—C4	1.361 (4)	C23—H23	0.9300
F1—C9—F2	105.1 (3)	C5—C6—C8	120.66 (18)
F1—C9—C8	112.8 (2)	C1—C6—C8	120.53 (17)
F2—C9—C8	111.9 (2)	O1—C7—H7A	109.5
F1—C9—C10	111.9 (3)	O1—C7—H7B	109.5
F2—C9—C10	110.1 (2)	H7A—C7—H7B	109.5
C8—C9—C10	105.2 (2)	O1—C7—H7C	109.5
F2'—C9'—F1'	107.7 (8)	H7A—C7—H7C	109.5
F2'—C9'—C8	112.1 (8)	H7B—C7—H7C	109.5
F1'—C9'—C8	116.1 (8)	C12—C8—C6	129.26 (18)
F2'—C9'—C10'	107.2 (10)	C12—C8—C9'	114.1 (5)
F1'—C9'—C10'	112.4 (9)	C6—C8—C9'	115.9 (5)
C8—C9'—C10'	100.9 (7)	C12—C8—C9	109.6 (2)
F4—C10—F3	106.5 (2)	C6—C8—C9	120.8 (2)
F4—C10—C9	112.6 (3)	C8—C12—C13	129.51 (17)
F3—C10—C9	110.1 (3)	C8—C12—C11'	106.6 (6)
F4—C10—C11	114.1 (3)	C13—C12—C11'	123.3 (6)
F3—C10—C11	110.1 (2)	C8—C12—C11	111.5 (2)
C9—C10—C11	103.5 (2)	C13—C12—C11	118.97 (19)
F3'—C10'—F4'	108.1 (9)	C16—C13—C14	112.60 (17)
F3'—C10'—C9'	111.9 (11)	C16—C13—C12	124.45 (17)
F4'—C10'—C9'	112.7 (11)	C14—C13—C12	122.85 (16)
F3'—C10'—C11'	109.3 (11)	C15—C14—C13	113.91 (17)
F4'—C10'—C11'	111.1 (11)	C15—C14—H14	123.0
C9'—C10'—C11'	103.7 (9)	C13—C14—H14	123.0
F5—C11—F6	105.7 (2)	C14—C15—C18	129.71 (18)
F5—C11—C12	113.2 (2)	C14—C15—S1	109.86 (15)
F6—C11—C12	112.9 (2)	C18—C15—S1	120.42 (15)
F5—C11—C10	110.8 (2)	C13—C16—C17	129.94 (19)
F6—C11—C10	110.0 (3)	C13—C16—S1	110.41 (15)
C12—C11—C10	104.2 (2)	C17—C16—S1	119.58 (16)
F6'—C11'—F5'	107.9 (9)	C16—C17—H17A	109.5
F6'—C11'—C12	109.1 (11)	C16—C17—H17B	109.5
F5'—C11'—C12	110.4 (9)	H17A—C17—H17B	109.5
F6'—C11'—C10'	110.9 (12)	C16—C17—H17C	109.5
F5'—C11'—C10'	113.1 (11)	H17A—C17—H17C	109.5
C12—C11'—C10'	105.4 (8)	H17B—C17—H17C	109.5
C16—S1—C15	93.21 (9)	C19—C18—C23	118.1 (2)
C1—O1—C7	118.22 (18)	C19—C18—C15	121.50 (19)
O1—C1—C2	123.8 (2)	C23—C18—C15	120.39 (18)

O1—C1—C6	116.66 (17)	C20—C19—C18	120.6 (2)
C2—C1—C6	119.5 (2)	C20—C19—H19	119.7
C3—C2—C1	120.0 (2)	C18—C19—H19	119.7
C3—C2—H2	120.0	C21—C20—C19	121.2 (2)
C1—C2—H2	120.0	C21—C20—H20	119.4
C4—C3—C2	121.2 (2)	C19—C20—H20	119.4
C4—C3—H3	119.4	C20—C21—C22	118.6 (2)
C2—C3—H3	119.4	C20—C21—H21	120.7
C3—C4—C5	119.2 (2)	C22—C21—H21	120.7
C3—C4—H4	120.4	C23—C22—C21	122.0 (2)
C5—C4—H4	120.4	C23—C22—C11	118.70 (18)
C4—C5—C6	121.3 (2)	C21—C22—C11	119.34 (18)
C4—C5—H5	119.4	C22—C23—C18	119.6 (2)
C6—C5—H5	119.4	C22—C23—H23	120.2
C5—C6—C1	118.77 (18)	C18—C23—H23	120.2
F1—C9—C10—F4	-89.8 (3)	C10—C9—C8—C12	-16.4 (3)
F2—C9—C10—F4	26.7 (4)	F1—C9—C8—C6	47.6 (4)
C8—C9—C10—F4	147.5 (3)	F2—C9—C8—C6	-70.7 (3)
F1—C9—C10—F3	28.9 (4)	C10—C9—C8—C6	169.7 (2)
F2—C9—C10—F3	145.4 (3)	F1—C9—C8—C9'	-29.7 (18)
C8—C9—C10—F3	-93.8 (3)	F2—C9—C8—C9'	-148 (2)
F1—C9—C10—C11	146.6 (3)	C10—C9—C8—C9'	92 (2)
F2—C9—C10—C11	-96.9 (3)	C6—C8—C12—C13	-4.6 (3)
C8—C9—C10—C11	23.8 (3)	C9'—C8—C12—C13	165.0 (5)
F2'—C9'—C10'—F3'	95.5 (11)	C9—C8—C12—C13	-177.7 (2)
F1'—C9'—C10'—F3'	-22.6 (13)	C6—C8—C12—C11'	-175.7 (6)
C8—C9'—C10'—F3'	-147.0 (10)	C9'—C8—C12—C11'	-6.1 (7)
F2'—C9'—C10'—F4'	-26.6 (13)	C9—C8—C12—C11'	11.2 (6)
F1'—C9'—C10'—F4'	-144.7 (9)	C6—C8—C12—C11	174.6 (2)
C8—C9'—C10'—F4'	90.9 (11)	C9'—C8—C12—C11	-15.8 (5)
F2'—C9'—C10'—C11'	-146.8 (9)	C9—C8—C12—C11	1.5 (3)
F1'—C9'—C10'—C11'	95.0 (10)	F6'—C11'—C12—C8	-132.9 (9)
C8—C9'—C10'—C11'	-29.3 (10)	F5'—C11'—C12—C8	108.7 (10)
F4—C10—C11—F5	92.5 (3)	C10'—C11'—C12—C8	-13.7 (10)
F3—C10—C11—F5	-27.2 (3)	F6'—C11'—C12—C13	55.3 (12)
C9—C10—C11—F5	-144.9 (2)	F5'—C11'—C12—C13	-63.1 (12)
F4—C10—C11—F6	-24.1 (3)	C10'—C11'—C12—C13	174.5 (6)
F3—C10—C11—F6	-143.8 (3)	F6'—C11'—C12—C11	-13 (3)
C9—C10—C11—F6	98.5 (3)	F5'—C11'—C12—C11	-131 (4)
F4—C10—C11—C12	-145.4 (2)	C10'—C11'—C12—C11	106 (4)
F3—C10—C11—C12	94.9 (3)	F5—C11—C12—C8	134.4 (2)
C9—C10—C11—C12	-22.8 (3)	F6—C11—C12—C8	-105.5 (3)
F3'—C10'—C11'—F6'	-95.1 (13)	C10—C11—C12—C8	13.9 (3)
F4'—C10'—C11'—F6'	24.1 (13)	F5—C11—C12—C13	-46.3 (3)
C9'—C10'—C11'—F6'	145.4 (10)	F6—C11—C12—C13	73.8 (3)
F3'—C10'—C11'—F5'	26.3 (13)	C10—C11—C12—C13	-166.80 (19)
F4'—C10'—C11'—F5'	145.5 (10)	F5—C11—C12—C11'	71 (4)
C9'—C10'—C11'—F5'	-93.2 (10)	F6—C11—C12—C11'	-169 (4)
F3'—C10'—C11'—C12	146.9 (10)	C10—C11—C12—C11'	-49 (4)

supplementary materials

F4'—C10'—C11'—C12	-93.8 (11)	C8—C12—C13—C16	-52.8 (3)
C9'—C10'—C11'—C12	27.4 (12)	C11'—C12—C13—C16	117.0 (7)
C7—O1—C1—C2	28.8 (3)	C11—C12—C13—C16	128.1 (2)
C7—O1—C1—C6	-151.1 (2)	C8—C12—C13—C14	131.0 (2)
O1—C1—C2—C3	-179.1 (2)	C11'—C12—C13—C14	-59.2 (7)
C6—C1—C2—C3	0.8 (4)	C11—C12—C13—C14	-48.2 (3)
C1—C2—C3—C4	0.3 (4)	C16—C13—C14—C15	0.6 (2)
C2—C3—C4—C5	-0.6 (4)	C12—C13—C14—C15	177.27 (17)
C3—C4—C5—C6	-0.2 (3)	C13—C14—C15—C18	178.83 (18)
C4—C5—C6—C1	1.3 (3)	C13—C14—C15—S1	-0.5 (2)
C4—C5—C6—C8	179.1 (2)	C16—S1—C15—C14	0.21 (16)
O1—C1—C6—C5	178.30 (19)	C16—S1—C15—C18	-179.19 (16)
C2—C1—C6—C5	-1.6 (3)	C14—C13—C16—C17	176.5 (2)
O1—C1—C6—C8	0.5 (3)	C12—C13—C16—C17	-0.1 (3)
C2—C1—C6—C8	-179.4 (2)	C14—C13—C16—S1	-0.4 (2)
C5—C6—C8—C12	120.1 (2)	C12—C13—C16—S1	-177.03 (14)
C1—C6—C8—C12	-62.1 (3)	C15—S1—C16—C13	0.14 (16)
C5—C6—C8—C9'	-49.3 (5)	C15—S1—C16—C17	-177.15 (18)
C1—C6—C8—C9'	128.5 (5)	C14—C15—C18—C19	-171.0 (2)
C5—C6—C8—C9	-67.4 (3)	S1—C15—C18—C19	8.3 (3)
C1—C6—C8—C9	110.4 (2)	C14—C15—C18—C23	8.5 (3)
F2'—C9'—C8—C12	137.0 (8)	S1—C15—C18—C23	-172.26 (16)
F1'—C9'—C8—C12	-98.6 (9)	C23—C18—C19—C20	-0.8 (4)
C10'—C9'—C8—C12	23.2 (8)	C15—C18—C19—C20	178.7 (2)
F2'—C9'—C8—C6	-52.0 (10)	C18—C19—C20—C21	0.9 (4)
F1'—C9'—C8—C6	72.5 (10)	C19—C20—C21—C22	-0.2 (4)
C10'—C9'—C8—C6	-165.8 (6)	C20—C21—C22—C23	-0.6 (4)
F2'—C9'—C8—C9	59.4 (16)	C20—C21—C22—C11	179.5 (2)
F1'—C9'—C8—C9	-176 (3)	C21—C22—C23—C18	0.6 (3)
C10'—C9'—C8—C9	-54.4 (18)	C11—C22—C23—C18	-179.50 (17)
F1—C9—C8—C12	-138.6 (3)	C19—C18—C23—C22	0.1 (3)
F2—C9—C8—C12	103.1 (3)	C15—C18—C23—C22	-179.40 (19)

Fig. 1

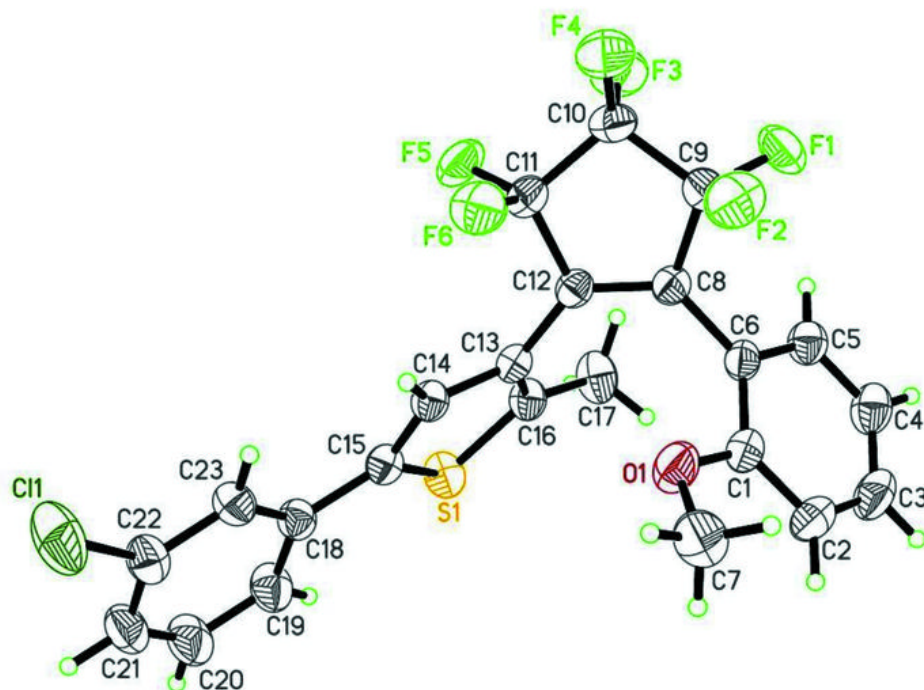


Fig. 2

