Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[2-(*N*,*N*-Dimethylamino)phenyl]-3,3,4,4,5,5-hexafluoro-1-(2-methyl-5-phenyl-3-thienyl)cyclopent-1-ene

Fan Congbin,* Yang Tianshe, Tu Qidong and Liu Gang

Jiangxi Key Laboratory of Organic Chemistry, Jiangxi Science and Technology Normal University, Nanchang 330013, People's Republic of China
Correspondence e-mail: congbinfan@yahoo.com.cn

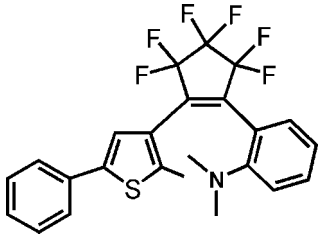
Received 27 August 2007; accepted 7 November 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.037; wR factor = 0.101; data-to-parameter ratio = 11.6.

The title compound, $C_{24}H_{19}F_6NS$, is a new hybrid diarylethene derivative with a 3-thienyl and a six-membered aryl unit as the substituents. In the crystal structure, the F atoms of the central hexafluorocyclopentene ring are disordered, with site occupancies of 0.641 (3) and 0.359 (3). The molecule adopts a photoactive antiparallel conformation. The distance between the two reactive C atoms, between which a new C—C bond can be formed upon UV irradiation, is 3.504 (5) Å. The dihedral angle between the thienyl ring and the adjacent phenyl ring is 12.4 (2)°. The dihedral angle between the central cyclopentene ring and the thienyl ring is 46.6 (7)°, and that between the central cyclopentene ring and the dimethylaminophenyl ring is 49.7 (4)°.

Related literature

Pu *et al.* (2005) and Peters *et al.* (2003) describe the synthesis of diarylethene compounds. Dürr & Bouas-Laurent (1990), Morimoto & Irie (2005), Tian & Yang (2004) and Woodward & Hoffmann (1970) give background information on the photochemistry of diarylethene compounds. Ramamurthy & Venkatesan (1987), Shibata *et al.* (2002) and Kobatake & Irie (2004) describe the theoretical basis of the photochemistry of diarylethenes.



Experimental

Crystal data

$C_{24}H_{19}F_6NS$
M_r = 467.46
Triclinic, $P\overline{1}$
a = 8.6995 (10) Å
b = 10.0545 (12) Å
c = 12.8146 (15) Å
α = 81.0190 (10)°
β = 82.6970 (10)°
γ = 80.3470 (10)°
V = 1085.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 293 (2) K
0.44 × 0.33 × 0.30 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.813, T_{max} = 0.939
7078 measured reflections
3712 independent reflections
2969 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.101
S = 1.03
3712 reflections
320 parameters
343 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); 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*.

This work was supported by the Natural Science Foundation of Jiangxi, China (grant Nos. 0620012, 050017) and the Science Fund of the Education Office of Jiangxi, China [grant No. (2007)279].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2061).

References

Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). *APEX2* (Version 1.027), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.
Dürr, H. & Bouas-Laurent, H. (1990). *Photochromism, Molecules and Systems in Organic Chemistry*. Amsterdam: Elsevier.
Kobatake, S. & Irie, M. (2004). *Bull. Chem. Soc. Jpn*, **77**, 195–210.
Morimoto, M. & Irie, M. (2005). *Chem. Commun.* pp. 3895–3905.
Peters, A., Vitols, C., McDonald, R. & Branda, N.-R. (2003). *Org. Lett.* **5**, 1183–1186.
Pu, S.-Z., Yang, T.-S., Xu, J.-K., Shen, L., Li, G.-Z., Xiao, Q. & Chen, B. (2005). *Tetrahedron*, **61**, 6623–6629.
Ramamurthy, V. & Venkatesan, K. (1987). *Chem. Rev.* **87**, 433–481.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Shibata, K., Muto, K., Kobatake, S. & Irie, M. (2002). *J. Phys. Chem. A*, **106**, 209–214.
Tian, H. & Yang, S.-J. (2004). *Chem. Soc. Rev.* **33**, 85–97.
Woodward, R. B. & Hoffmann, R. (1970). *The Conservation of Orbital Symmetry*. Weinheim: Verlag Chemie GmbH.

 doi:10.1107/S1600536807056899

supplementary materials

Acta Cryst. (2007). E**63**, o4721 [**doi:10.1107/S1600536807056899**]

2-[2-(*N*,*N*-Dimethylamino)phenyl]-3,3,4,4,5,5-hexafluoro-1-(2-methyl-5-phenyl-3-thienyl)cyclopent-1-ene

F. Congbin, Y. Tianshe, T. Qidong and L. Gang

Comment

Photochromic diarylethenes have been extensively studied for optoelectronic applications, optical storage, photo-switches and waveguides, *etc* (Dürr & Bouas-Laurent, 1990). The design and synthesis of new photochromic compounds is a hotspot in this research field, and many publications concerning synthesis and investigation of the properties of diarylethenes with heterocyclic aryl rings have been reported (Pu *et al.*, 2005). Almost all photochromic perfluorocyclopentene systems have five-membered heterocyclic rings as the aryl substitituents (Tian & Yang, 2004). We supposed that a diarylethene would have some novel characteristics when replacing one of the five-membered heterocyclic rings with a six-membered aryl ring. This paper presents the synthesis and crystal structure of a new asymmetric diarylethene with a six-membered aryl ring group.

The C11—C15 bond (1.344 (3) Å) in the cyclopent-1-ene ring of the title compound is clearly a double bond, being significantly shorter than all other C—C single bonds in the molecule. The hexafluoropropylene unit tethered to the double bond forces the methylthiophene and the six-membered aryl groups into a *cis* position. The thiophene ring and six-membered aryl systems bonded to the cyclopentene have similar dihedral angles to the least-square plane of the cyclopentene ring, which are 46.6 (7)° and 49.7 (4)°, respectively. This geometry brings the potential photoreactive centers in close proximity (Fig.1). Such a conformation is crucial for the compound to exhibit photochromic and photoinduced properties (Woodward & Hoffmann, 1970). The distance between the two reactive C atoms C4 in the thienyl and C21 in the aryl unit of the molecule is 3.504 (5) Å. This distance indicates that the crystal can be expected to undergo photochromism to generate the photoisomer of the title compound upon irradiation (Fig. 4) (Morimoto & Irie, 2005), as photochromic reactivity usually appears when the distance between the reactive C atoms is less than 4.2 Å (Ramamurthy & Venkatesan, 1987; Shibata *et al.*, 2002; Kobatake & Irie, 2004). When the crystal was dissolved in hexane the absorption peak of 283 nm decreased significantly upon irradiation with 365 nm UV light, but the absorption at 228 nm increased dramatically. Upon irradiation with visible light with wavelengths greater than 510 nm, the change of the absorption spectrum of the hexane solution reversed.

Experimental

The title compound was prepared from (2-methyl-5-phenyl-3-thienyl)-3,3,4,4,5,5-hexafluorocyclopent-1-ene (1.83 g, 5.00 mmol) (Peters *et al.*, 2003) and 2-bromo-*N*,*N*-dimethylbenzeneamine (1.00 g, 5.00 mmol) (Fig 3). To a stirred solution of 2-bromo-*N*,*N*-dimethylbenzeneamine (1.00 g, 5.00 mmol) in THF (50 ml) was added dropwise an n-BuLi solution (2.0 ml, 2.5 mol/l) at 195 K under a nitrogen atmosphere. Stirring was continued for 30 min, (2-methyl-5-phenyl-3-thienyl)-3,3,4,4,5,5-hexafluorocyclopent-1-ene (1.83 g, 5.00 mmol) was slowly added to the reaction mixture, and the mixture was stirred for 2.0 h at 195 K. The reaction was stopped by the addition of water. 1-(2-Methyl-5-phenyl-3-thienyl)-2-(2-*N*,*N*-dimethylphenyl) −3,3,4,4,5,5-hexafluorocyclopent-1-ene (1.56 g, 3.34 mmol) was obtained in 66.7% yield by column chromatography on SiO_2 using petroleum ether [boiling range (333–363 K)] as the eluent. Finally yellow crystals were obtained by slow vapour diffusion of hexane into a solution of chloroform. The title compound was characterized by melting point, elemental analysis

and NMR spectroscopy: m.p.398–399 K; ^{1}HNMR (400 MHz, $CDCl_3$, TMS): δ 1.69 (s, 3H, $–CH_3$), 2.36 (s, 6H, $–CH_3$), 6.84, 6.86 (d, 1H, J = 8.0 Hz, benzene-H), 7.02 (t, 1H, J = 8.0 Hz, benzene-H), 7.14 (s, 1H, thiophene-H), 7.29 (m, 2H, benzene-H), 7.37 (t, 2H, J = 8.0 Hz, benzene-H), 7.51 (t, 2H, J = 8.0 Hz, benzene-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.92, 43.07, 118.90, 120.67, 122.46, 125.56, 126.99, 127.60, 128.97, 129.74, 130.94, 133.91, 139.57, 140.96, 152.98; IR (KBr, cm^{-1}): 751, 849, 951, 990, 1062, 1130, 1257, 1344, 1431, 1504, 1597, 1626, 2792, 1865, 2941; Anal. Calcd. for $C_{24}H_{19}F_6NS$(%): C, 61.66, H, 4.10, Found: C, 62.05, H, 4.09.

Refinement

Six F atoms of the hexafluorocyclopentene ring are disordered over two positions. The C—F bonds of the hexafluorocyclopentene ring were restrained to be 1.34 (2) Å. The bonds C12—C13 and C13—C14 were restrained to have the same length within a standard deviation of 0.02 Å. From anisotropic refinement, the site occupancies for the two moieties were refined to be 0.641 (3) (primed) and 0.359 (3) (unprimed), respectively. The H atoms were positioned theoretically and allowed to ride on their parent atoms in the final refinement [C—H = 0.93–0.96Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C)]. The methyl groups were treated as rigid groups and allowed to rotate about the C—C bond.

Figures

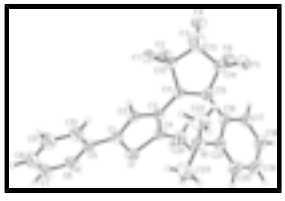


Fig. 1. The molecular structure of the title compound, showing 35% probability ellipsoids and the atom-numbering scheme. For clarity only the major component is shown for the disordered moiety.

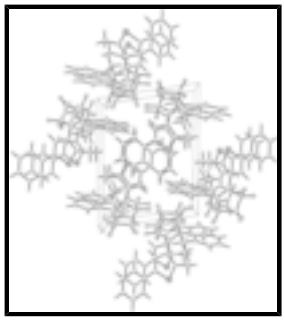


Fig. 2. Packing diagram viewed down the *b* axis.

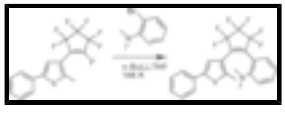


Fig. 3. Synthesis of the title compound.

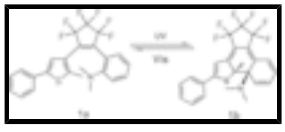


Fig. 4. Photochromic interconversion of the title compound (compound Ia) and its photoisomer Ib.

2-[2-(*N*,*N*-Dimethylamino)phenyl]-3,3,4,4,5,5-hexafluoro- 1-(2-methyl-5-phenyl-3-thienyl)cyclopent-1-ene

Crystal data

$C_{24}H_{19}F_6NS$	$F_{000} = 480$
$M_r = 467.46$	$D_x = 1.430$ Mg m^{-3}
Triclinic, $P\overline{1}$	Melting point: 399 K
$a = 8.6995$ (10) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.0545$ (12) Å	Cell parameters from 2875 reflections
$c = 12.8146$ (15) Å	$\theta = 2.4$–26.9º
$\alpha = 81.0190$ (10)º	$\mu = 0.21$ mm^{-1}
$\beta = 82.6970$ (10)º	$T = 293$ (2) K
$\gamma = 80.3470$ (10)º	Block, yellow
$V = 1085.6$ (2) Å^3	0.44 × 0.33 × 0.30 mm
$Z = 2$	

Data collection

Bruker APEXII CCD area-detector diffractometer	3712 independent reflections
Radiation source: fine-focus sealed tube	2969 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.015$
$T = 293$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (SADABS; Bruker, 2004?)	$h = -10 \rightarrow 10$
$T_{min} = 0.813$, $T_{max} = 0.939$	$k = -11 \rightarrow 10$
7078 measured reflections	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.101$	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.285P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
3712 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
320 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
343 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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
F1	0.8341 (2)	0.48840 (15)	0.89767 (14)	0.0790 (5)	0.9045 (18)
F2	0.6224 (2)	0.5981 (2)	0.83418 (13)	0.0860 (6)	0.9045 (18)
F3	0.7941 (2)	0.61311 (17)	1.06001 (11)	0.0894 (6)	0.9045 (18)
F4	0.5507 (2)	0.62687 (18)	1.03391 (17)	0.0967 (6)	0.9045 (18)
F5	0.71145 (19)	0.87168 (17)	1.04886 (11)	0.0715 (4)	0.9045 (18)
F6	0.53483 (17)	0.87590 (16)	0.94364 (14)	0.0703 (4)	0.9045 (18)
C12	0.7469 (3)	0.6129 (3)	0.8840 (2)	0.0557 (6)	0.9045 (18)
C13	0.6879 (3)	0.6637 (3)	0.9903 (2)	0.0606 (6)	0.9045 (18)
C14	0.6830 (3)	0.8176 (2)	0.96481 (19)	0.0522 (5)	0.9045 (18)
F1'	0.885 (2)	0.5155 (17)	0.9319 (14)	0.0790 (5)	0.0955 (18)
F2'	0.705 (2)	0.536 (2)	0.8269 (14)	0.0860 (6)	0.0955 (18)
F3'	0.671 (3)	0.5792 (17)	1.0677 (11)	0.0894 (6)	0.0955 (18)
F4'	0.5137 (17)	0.6690 (16)	0.9503 (13)	0.0967 (6)	0.0955 (18)
F5'	0.7913 (16)	0.7914 (16)	1.0580 (10)	0.0715 (4)	0.0955 (18)
F6'	0.5787 (17)	0.8815 (17)	0.9882 (15)	0.0703 (4)	0.0955 (18)
C12'	0.770 (2)	0.6037 (17)	0.8870 (14)	0.0557 (6)	0.0955 (18)
C13'	0.661 (2)	0.660 (2)	0.9745 (14)	0.0606 (6)	0.0955 (18)
C14'	0.7125 (18)	0.7949 (19)	0.9737 (10)	0.0522 (5)	0.0955 (18)
S1	1.05466 (7)	0.71277 (6)	0.53074 (4)	0.06182 (18)	
N1	1.1320 (2)	0.86263 (19)	0.81512 (14)	0.0601 (5)	

C1	1.1269 (2)	0.5736 (2)	0.61735 (15)	0.0504 (5)
C2	1.0496 (2)	0.58068 (19)	0.71592 (14)	0.0482 (5)
H2	1.0710	0.5147	0.7738	0.058*
C3	0.9333 (2)	0.69751 (18)	0.72311 (14)	0.0461 (4)
C4	0.9214 (2)	0.7794 (2)	0.62758 (15)	0.0524 (5)
C5	1.2517 (2)	0.4665 (2)	0.58193 (15)	0.0526 (5)
C6	1.2966 (3)	0.4577 (3)	0.47489 (18)	0.0685 (6)
H6	1.2484	0.5214	0.4237	0.082*
C7	1.4121 (3)	0.3554 (3)	0.4439 (2)	0.0805 (7)
H7	1.4411	0.3514	0.3720	0.097*
C8	1.4839 (3)	0.2608 (3)	0.5167 (2)	0.0828 (8)
H8	1.5619	0.1925	0.4949	0.099*
C9	1.4409 (3)	0.2664 (3)	0.6226 (2)	0.0812 (7)
H9	1.4889	0.2011	0.6729	0.097*
C10	1.3264 (3)	0.3687 (2)	0.65499 (18)	0.0679 (6)
H10	1.2990	0.3721	0.7272	0.081*
C11	0.8348 (2)	0.72232 (18)	0.82253 (14)	0.0443 (4)
C15	0.8024 (2)	0.83566 (18)	0.86960 (14)	0.0441 (4)
C16	0.8587 (2)	0.96785 (19)	0.83729 (14)	0.0469 (4)
C17	0.7485 (3)	1.0865 (2)	0.83338 (17)	0.0593 (5)
H17	0.6448	1.0807	0.8596	0.071*
C18	0.7902 (3)	1.2120 (2)	0.79157 (19)	0.0714 (6)
H18	0.7155	1.2900	0.7893	0.086*
C19	0.9425 (3)	1.2200 (2)	0.75369 (19)	0.0728 (7)
H19	0.9705	1.3038	0.7231	0.087*
C20	1.0553 (3)	1.1065 (2)	0.75982 (17)	0.0657 (6)
H20	1.1585	1.1150	0.7339	0.079*
C21	1.0178 (2)	0.9778 (2)	0.80447 (15)	0.0508 (5)
C22	1.1478 (3)	0.7867 (3)	0.9196 (2)	0.0793 (7)
H22A	1.2362	0.8089	0.9475	0.119*
H22B	1.1633	0.6909	0.9149	0.119*
H22C	1.0544	0.8098	0.9657	0.119*
C23	1.2797 (3)	0.8644 (3)	0.7482 (2)	0.0951 (9)
H23A	1.2597	0.9017	0.6767	0.143*
H23B	1.3349	0.7732	0.7493	0.143*
H23C	1.3423	0.9196	0.7744	0.143*
C24	0.8113 (3)	0.9081 (2)	0.59867 (18)	0.0713 (6)
H24A	0.7170	0.9092	0.6466	0.107*
H24B	0.7863	0.9121	0.5273	0.107*
H24C	0.8603	0.9853	0.6036	0.107*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.1159 (14)	0.0358 (8)	0.0717 (11)	−0.0072 (8)	0.0267 (9)	0.0001 (7)
F2	0.0973 (13)	0.0970 (14)	0.0771 (10)	−0.0567 (10)	−0.0090 (10)	−0.0075 (9)
F3	0.1293 (15)	0.0775 (11)	0.0512 (8)	0.0051 (10)	−0.0145 (9)	0.0042 (7)
F4	0.1046 (13)	0.0791 (11)	0.1006 (14)	−0.0419 (10)	0.0504 (11)	−0.0139 (10)

F5	0.0911 (11)	0.0739 (11)	0.0527 (7)	−0.0183 (8)	0.0079 (7)	−0.0247 (7)
F6	0.0487 (8)	0.0675 (9)	0.0908 (12)	−0.0013 (7)	−0.0015 (7)	−0.0114 (8)
C12	0.0710 (14)	0.0430 (12)	0.0522 (11)	−0.0146 (10)	−0.0002 (10)	−0.0020 (9)
C13	0.0684 (14)	0.0555 (12)	0.0522 (12)	−0.0125 (10)	0.0096 (10)	0.0005 (10)
C14	0.0515 (13)	0.0518 (13)	0.0517 (11)	−0.0032 (11)	0.0022 (9)	−0.0130 (9)
F1'	0.1159 (14)	0.0358 (8)	0.0717 (11)	−0.0072 (8)	0.0267 (9)	0.0001 (7)
F2'	0.0973 (13)	0.0970 (14)	0.0771 (10)	−0.0567 (10)	−0.0090 (10)	−0.0075 (9)
F3'	0.1293 (15)	0.0775 (11)	0.0512 (8)	0.0051 (10)	−0.0145 (9)	0.0042 (7)
F4'	0.1046 (13)	0.0791 (11)	0.1006 (14)	−0.0419 (10)	0.0504 (11)	−0.0139 (10)
F5'	0.0911 (11)	0.0739 (11)	0.0527 (7)	−0.0183 (8)	0.0079 (7)	−0.0247 (7)
F6'	0.0487 (8)	0.0675 (9)	0.0908 (12)	−0.0013 (7)	−0.0015 (7)	−0.0114 (8)
C12'	0.0710 (14)	0.0430 (12)	0.0522 (11)	−0.0146 (10)	−0.0002 (10)	−0.0020 (9)
C13'	0.0684 (14)	0.0555 (12)	0.0522 (12)	−0.0125 (10)	0.0096 (10)	0.0005 (10)
C14'	0.0515 (13)	0.0518 (13)	0.0517 (11)	−0.0032 (11)	0.0022 (9)	−0.0130 (9)
S1	0.0825 (4)	0.0630 (4)	0.0377 (3)	−0.0163 (3)	0.0008 (2)	−0.0001 (2)
N1	0.0560 (10)	0.0666 (12)	0.0575 (10)	−0.0097 (9)	−0.0055 (8)	−0.0080 (9)
C1	0.0600 (12)	0.0501 (11)	0.0436 (10)	−0.0186 (9)	−0.0002 (9)	−0.0071 (8)
C2	0.0630 (12)	0.0418 (10)	0.0391 (10)	−0.0116 (9)	−0.0009 (8)	−0.0027 (8)
C3	0.0607 (11)	0.0387 (10)	0.0401 (10)	−0.0134 (8)	−0.0040 (8)	−0.0037 (8)
C4	0.0706 (13)	0.0453 (11)	0.0428 (10)	−0.0145 (9)	−0.0080 (9)	−0.0025 (8)
C5	0.0539 (11)	0.0576 (12)	0.0500 (11)	−0.0197 (9)	0.0044 (9)	−0.0141 (9)
C6	0.0703 (14)	0.0854 (17)	0.0510 (12)	−0.0132 (12)	0.0036 (10)	−0.0197 (11)
C7	0.0742 (16)	0.104 (2)	0.0667 (15)	−0.0126 (15)	0.0144 (13)	−0.0397 (15)
C8	0.0629 (15)	0.091 (2)	0.097 (2)	−0.0090 (13)	0.0114 (14)	−0.0391 (17)
C9	0.0711 (16)	0.0820 (18)	0.0845 (18)	0.0004 (13)	−0.0009 (13)	−0.0116 (14)
C10	0.0705 (14)	0.0729 (16)	0.0556 (13)	−0.0051 (12)	0.0055 (11)	−0.0106 (11)
C11	0.0531 (11)	0.0391 (10)	0.0395 (9)	−0.0071 (8)	−0.0045 (8)	−0.0018 (8)
C15	0.0472 (10)	0.0408 (10)	0.0438 (10)	−0.0056 (8)	−0.0069 (8)	−0.0035 (8)
C16	0.0597 (12)	0.0400 (10)	0.0423 (10)	−0.0095 (8)	−0.0057 (8)	−0.0075 (8)
C17	0.0695 (14)	0.0460 (12)	0.0622 (13)	−0.0052 (10)	−0.0057 (10)	−0.0115 (10)
C18	0.1010 (19)	0.0412 (12)	0.0733 (15)	−0.0069 (12)	−0.0186 (14)	−0.0079 (11)
C19	0.113 (2)	0.0478 (14)	0.0636 (14)	−0.0313 (14)	−0.0174 (14)	−0.0003 (10)
C20	0.0811 (15)	0.0642 (15)	0.0589 (13)	−0.0338 (13)	−0.0087 (11)	−0.0048 (11)
C21	0.0645 (12)	0.0503 (12)	0.0422 (10)	−0.0179 (10)	−0.0082 (9)	−0.0082 (8)
C22	0.0745 (16)	0.0862 (18)	0.0728 (16)	−0.0041 (13)	−0.0220 (13)	0.0054 (13)
C23	0.0667 (16)	0.123 (2)	0.0886 (19)	−0.0079 (16)	0.0093 (14)	−0.0132 (17)
C24	0.0989 (18)	0.0568 (14)	0.0554 (13)	−0.0044 (12)	−0.0202 (12)	0.0030 (10)

Geometric parameters (Å, °)

F1—C12	1.349 (3)	C5—C10	1.385 (3)
F2—C12	1.364 (3)	C5—C6	1.390 (3)
F3—C13	1.350 (3)	C6—C7	1.380 (3)
F4—C13	1.337 (3)	C6—H6	0.9300
F5—C14	1.347 (3)	C7—C8	1.356 (4)
F6—C14	1.365 (3)	C7—H7	0.9300
C12—C11	1.508 (3)	C8—C9	1.368 (4)
C12—C13	1.527 (3)	C8—H8	0.9300
C13—C14	1.525 (3)	C9—C10	1.381 (3)

C14—C15	1.508 (3)	C9—H9	0.9300
F1'—C12'	1.344 (11)	C10—H10	0.9300
F2'—C12'	1.339 (11)	C11—C15	1.344 (3)
F3'—C13'	1.340 (11)	C15—C16	1.474 (3)
F4'—C13'	1.345 (11)	C16—C17	1.398 (3)
F5'—C14'	1.344 (11)	C16—C21	1.410 (3)
F6'—C14'	1.343 (10)	C17—C18	1.379 (3)
C12'—C11	1.490 (12)	C17—H17	0.9300
C12'—C13'	1.492 (12)	C18—C19	1.363 (4)
C13'—C14'	1.493 (11)	C18—H18	0.9300
C14'—C15	1.493 (11)	C19—C20	1.374 (3)
S1—C4	1.722 (2)	C19—H19	0.9300
S1—C1	1.727 (2)	C20—C21	1.403 (3)
N1—C21	1.395 (3)	C20—H20	0.9300
N1—C22	1.444 (3)	C22—H22A	0.9600
N1—C23	1.453 (3)	C22—H22B	0.9600
C1—C2	1.359 (3)	C22—H22C	0.9600
C1—C5	1.477 (3)	C23—H23A	0.9600
C2—C3	1.423 (3)	C23—H23B	0.9600
C2—H2	0.9300	C23—H23C	0.9600
C3—C4	1.370 (3)	C24—H24A	0.9600
C3—C11	1.473 (3)	C24—H24B	0.9600
C4—C24	1.501 (3)	C24—H24C	0.9600
F1—C12—F2	105.9 (2)	C8—C7—C6	121.1 (2)
F1—C12—C11	114.1 (2)	C8—C7—H7	119.5
F2—C12—C11	111.5 (2)	C6—C7—H7	119.5
F1—C12—C13	111.3 (2)	C7—C8—C9	119.5 (2)
F2—C12—C13	109.5 (2)	C7—C8—H8	120.2
C11—C12—C13	104.51 (19)	C9—C8—H8	120.2
F4—C13—F3	107.3 (2)	C8—C9—C10	120.2 (3)
F4—C13—C12	114.3 (2)	C8—C9—H9	119.9
F3—C13—C12	109.0 (2)	C10—C9—H9	119.9
F4—C13—C14	113.4 (2)	C9—C10—C5	121.2 (2)
F3—C13—C14	109.1 (2)	C9—C10—H10	119.4
C12—C13—C14	103.61 (19)	C5—C10—H10	119.4
F5—C14—F6	105.62 (18)	C15—C11—C3	129.46 (16)
F5—C14—C15	113.97 (19)	C15—C11—C12'	113.3 (8)
F6—C14—C15	112.68 (18)	C3—C11—C12'	117.1 (8)
F5—C14—C13	111.62 (19)	C15—C11—C12	110.45 (18)
F6—C14—C13	108.8 (2)	C3—C11—C12	120.03 (17)
C15—C14—C13	104.17 (17)	C11—C15—C16	129.55 (16)
F2'—C12'—F1'	107.2 (10)	C11—C15—C14'	105.0 (8)
F2'—C12'—C11	111.4 (13)	C16—C15—C14'	124.9 (7)
F1'—C12'—C11	110.4 (13)	C11—C15—C14	110.47 (17)
F2'—C12'—C13'	114.7 (16)	C16—C15—C14	119.86 (17)
F1'—C12'—C13'	107.4 (15)	C17—C16—C21	119.19 (18)
C11—C12'—C13'	105.7 (10)	C17—C16—C15	118.44 (18)
F3'—C13'—F4'	106.7 (10)	C21—C16—C15	122.26 (17)
F3'—C13'—C12'	112.5 (15)	C18—C17—C16	121.5 (2)

F4'—C13'—C12'	108.1 (16)	C18—C17—H17	119.2
F3'—C13'—C14'	113.5 (15)	C16—C17—H17	119.2
F4'—C13'—C14'	114.0 (16)	C19—C18—C17	119.0 (2)
C12'—C13'—C14'	102.0 (12)	C19—C18—H18	120.5
F5'—C14'—F6'	106.7 (10)	C17—C18—H18	120.5
F5'—C14'—C15	113.8 (12)	C18—C19—C20	121.2 (2)
F6'—C14'—C15	111.1 (13)	C18—C19—H19	119.4
F5'—C14'—C13'	110.0 (13)	C20—C19—H19	119.4
F6'—C14'—C13'	104.7 (14)	C19—C20—C21	121.3 (2)
C15—C14'—C13'	110.1 (11)	C19—C20—H20	119.4
C4—S1—C1	93.19 (9)	C21—C20—H20	119.4
C21—N1—C22	118.53 (18)	N1—C21—C20	121.9 (2)
C21—N1—C23	118.33 (19)	N1—C21—C16	120.50 (17)
C22—N1—C23	113.6 (2)	C20—C21—C16	117.6 (2)
C2—C1—C5	128.50 (18)	N1—C22—H22A	109.5
C2—C1—S1	109.61 (15)	N1—C22—H22B	109.5
C5—C1—S1	121.88 (14)	H22A—C22—H22B	109.5
C1—C2—C3	114.37 (17)	N1—C22—H22C	109.5
C1—C2—H2	122.8	H22A—C22—H22C	109.5
C3—C2—H2	122.8	H22B—C22—H22C	109.5
C4—C3—C2	112.41 (17)	N1—C23—H23A	109.5
C4—C3—C11	124.94 (18)	N1—C23—H23B	109.5
C2—C3—C11	122.62 (16)	H23A—C23—H23B	109.5
C3—C4—C24	130.03 (19)	N1—C23—H23C	109.5
C3—C4—S1	110.42 (15)	H23A—C23—H23C	109.5
C24—C4—S1	119.52 (15)	H23B—C23—H23C	109.5
C10—C5—C6	117.4 (2)	C4—C24—H24A	109.5
C10—C5—C1	120.80 (18)	C4—C24—H24B	109.5
C6—C5—C1	121.7 (2)	H24A—C24—H24B	109.5
C7—C6—C5	120.6 (2)	C4—C24—H24C	109.5
C7—C6—H6	119.7	H24A—C24—H24C	109.5
C5—C6—H6	119.7	H24B—C24—H24C	109.5
F1—C12—C13—F4	−88.6 (3)	F2'—C12'—C11—C15	136.6 (11)
F2—C12—C13—F4	28.2 (3)	F1'—C12'—C11—C15	−104.5 (13)
C11—C12—C13—F4	147.7 (2)	C13'—C12'—C11—C15	11.4 (15)
F1—C12—C13—F3	31.4 (3)	F2'—C12'—C11—C3	−48.2 (16)
F2—C12—C13—F3	148.2 (2)	F1'—C12'—C11—C3	70.8 (14)
C11—C12—C13—F3	−92.3 (2)	C13'—C12'—C11—C3	−173.3 (10)
F1—C12—C13—C14	147.5 (2)	F2'—C12'—C11—C12	65 (6)
F2—C12—C13—C14	−95.7 (2)	F1'—C12'—C11—C12	−176 (7)
C11—C12—C13—C14	23.8 (3)	C13'—C12'—C11—C12	−60 (5)
F4—C13—C14—F5	86.8 (3)	F1—C12—C11—C15	−135.5 (2)
F3—C13—C14—F5	−32.7 (3)	F2—C12—C11—C15	104.5 (2)
C12—C13—C14—F5	−148.7 (2)	C13—C12—C11—C15	−13.7 (3)
F4—C13—C14—F6	−29.3 (3)	F1—C12—C11—C3	47.0 (3)
F3—C13—C14—F6	−148.9 (2)	F2—C12—C11—C3	−73.0 (3)
C12—C13—C14—F6	95.1 (2)	C13—C12—C11—C3	168.80 (18)
F4—C13—C14—C15	−149.7 (2)	F1—C12—C11—C12'	−24 (6)
F3—C13—C14—C15	90.7 (2)	F2—C12—C11—C12'	−144 (6)

C12—C13—C14—C15	−25.3 (2)	C13—C12—C11—C12'	98 (6)
F2'—C12'—C13'—F3'	96.9 (18)	C3—C11—C15—C16	−1.7 (3)
F1'—C12'—C13'—F3'	−22.2 (17)	C12'—C11—C15—C16	172.9 (9)
C11—C12'—C13'—F3'	−140.0 (14)	C12—C11—C15—C16	−178.9 (2)
F2'—C12'—C13'—F4'	−20.6 (18)	C3—C11—C15—C14'	−173.6 (7)
F1'—C12'—C13'—F4'	−139.7 (15)	C12'—C11—C15—C14'	1.0 (11)
C11—C12'—C13'—F4'	102.5 (15)	C12—C11—C15—C14'	9.2 (7)
F2'—C12'—C13'—C14'	−141.1 (13)	C3—C11—C15—C14	174.39 (19)
F1'—C12'—C13'—C14'	99.9 (15)	C12'—C11—C15—C14	−11.1 (9)
C11—C12'—C13'—C14'	−18.0 (17)	C12—C11—C15—C14	−2.8 (2)
F3'—C13'—C14'—F5'	14.7 (18)	F5'—C14'—C15—C11	110.7 (12)
F4'—C13'—C14'—F5'	137.1 (15)	F6'—C14'—C15—C11	−128.9 (11)
C12'—C13'—C14'—F5'	−106.6 (15)	C13'—C14'—C15—C11	−13.3 (13)
F3'—C13'—C14'—F6'	−99.6 (17)	F5'—C14'—C15—C16	−61.6 (15)
F4'—C13'—C14'—F6'	22.8 (17)	F6'—C14'—C15—C16	58.8 (14)
C12'—C13'—C14'—F6'	139.1 (13)	C13'—C14'—C15—C16	174.3 (9)
F3'—C13'—C14'—C15	140.9 (14)	F5'—C14'—C15—C14	−132 (4)
F4'—C13'—C14'—C15	−96.7 (16)	F6'—C14'—C15—C14	−12 (3)
C12'—C13'—C14'—C15	19.6 (17)	C13'—C14'—C15—C14	104 (4)
C4—S1—C1—C2	0.04 (16)	F5—C14—C15—C11	140.05 (19)
C4—S1—C1—C5	178.88 (16)	F6—C14—C15—C11	−99.6 (2)
C5—C1—C2—C3	−179.16 (18)	C13—C14—C15—C11	18.2 (2)
S1—C1—C2—C3	−0.4 (2)	F5—C14—C15—C16	−43.4 (3)
C1—C2—C3—C4	0.7 (2)	F6—C14—C15—C16	76.9 (3)
C1—C2—C3—C11	178.77 (17)	C13—C14—C15—C16	−165.31 (18)
C2—C3—C4—C24	177.3 (2)	F5—C14—C15—C14'	74 (3)
C11—C3—C4—C24	−0.7 (3)	F6—C14—C15—C14'	−166 (4)
C2—C3—C4—S1	−0.6 (2)	C13—C14—C15—C14'	−48 (3)
C11—C3—C4—S1	−178.65 (15)	C11—C15—C16—C17	130.2 (2)
C1—S1—C4—C3	0.35 (16)	C14'—C15—C16—C17	−59.4 (8)
C1—S1—C4—C24	−177.87 (18)	C14—C15—C16—C17	−45.6 (3)
C2—C1—C5—C10	−12.5 (3)	C11—C15—C16—C21	−46.0 (3)
S1—C1—C5—C10	168.85 (17)	C14'—C15—C16—C21	124.4 (8)
C2—C1—C5—C6	166.1 (2)	C14—C15—C16—C21	138.23 (19)
S1—C1—C5—C6	−12.5 (3)	C21—C16—C17—C18	4.4 (3)
C10—C5—C6—C7	−0.4 (3)	C15—C16—C17—C18	−171.92 (19)
C1—C5—C6—C7	−179.1 (2)	C16—C17—C18—C19	−0.3 (3)
C5—C6—C7—C8	0.3 (4)	C17—C18—C19—C20	−2.3 (4)
C6—C7—C8—C9	0.2 (4)	C18—C19—C20—C21	0.5 (3)
C7—C8—C9—C10	−0.8 (4)	C22—N1—C21—C20	123.1 (2)
C8—C9—C10—C5	0.7 (4)	C23—N1—C21—C20	−21.1 (3)
C6—C5—C10—C9	−0.1 (3)	C22—N1—C21—C16	−57.9 (3)
C1—C5—C10—C9	178.6 (2)	C23—N1—C21—C16	157.9 (2)
C4—C3—C11—C15	−50.8 (3)	C19—C20—C21—N1	−177.4 (2)
C2—C3—C11—C15	131.4 (2)	C19—C20—C21—C16	3.6 (3)
C4—C3—C11—C12'	134.8 (9)	C17—C16—C21—N1	175.04 (18)
C2—C3—C11—C12'	−43.0 (9)	C15—C16—C21—N1	−8.8 (3)
C4—C3—C11—C12	126.2 (2)	C17—C16—C21—C20	−5.9 (3)
C2—C3—C11—C12	−51.6 (3)	C15—C16—C21—C20	170.28 (17)

Fig. 1

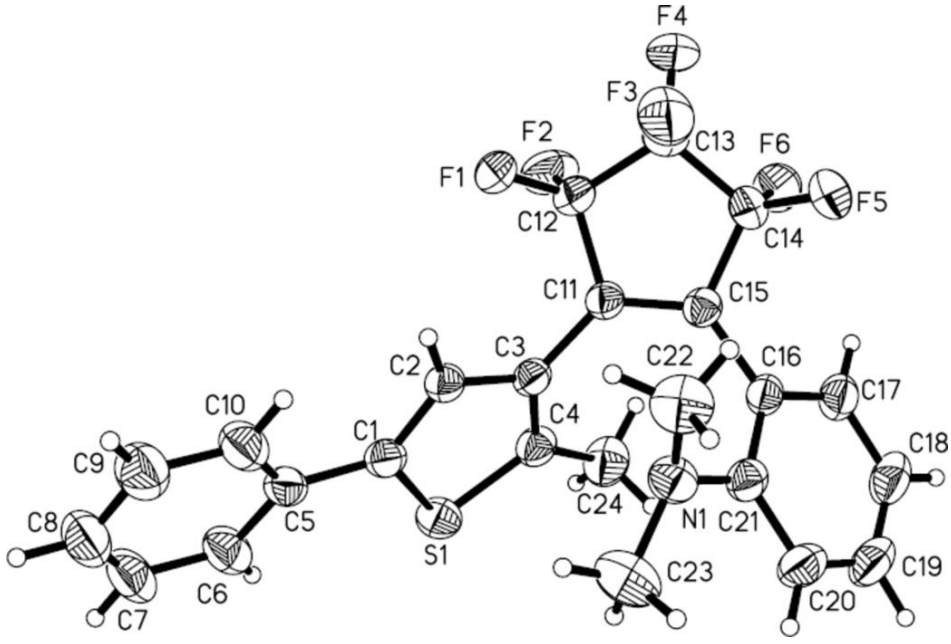

F4
F3
F2
C13
F6
F1
F5
C12
C14
C11
C15
C3
C22
C16
C2
C17
C10
C4
C1
C18
C9
C24
N1
C21
C8
C5
S1
C23
C19
C6
C20
C7

Fig. 2

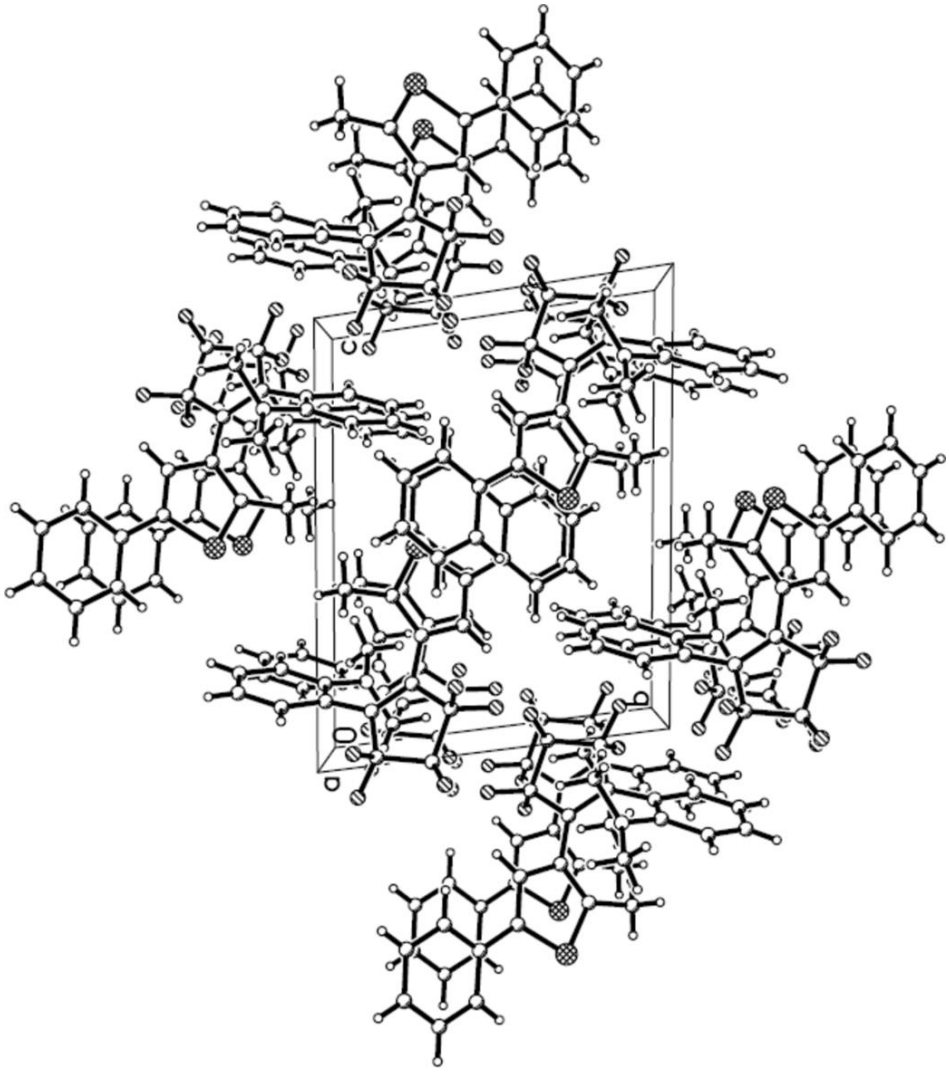


Fig. 3

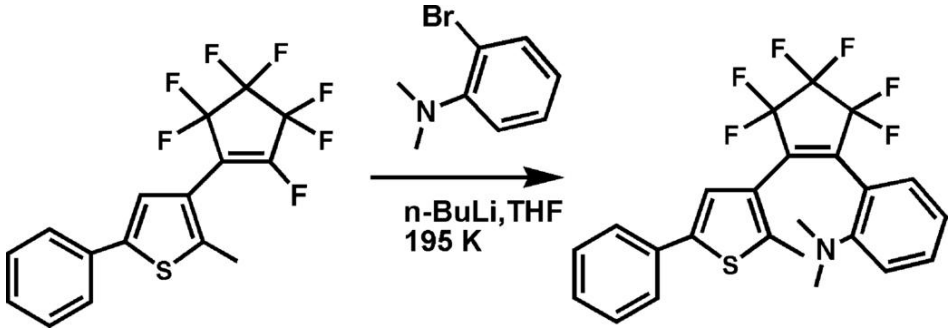


Fig. 4