Crystal structure of 1-(2,5-dimethylthien-3-yl)-2-[3-methyl-5-(*p*-methoxyphenyl)thien-2-yl]perfluorocyclopentene, $C_{23}H_{18}F_6OS_2$

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Abstract

$C_{23}H_{18}F_6OS_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 14.225(2)$ Å, $b = 17.710(3)$ Å, $c = 8.962(3)$ Å, $\beta = 101.23(2)^\circ$, $V = 2214.5$ Å³, $Z = 4$, $R_{gt}(F) = 0.067$, $wR_{ref}(F^2) = 0.182$, $T = 293$ K.

Source of material

Synthesis of the title compound was referred to the method in [1,2]. To a stirred anhydrous tetrahydrofuran solution containing 3-iodo-2,5-dimethylthiophene was added a BuLi hexane solution under nitrogen atmosphere at 195 K. The solution was stirred for 15 min. Then, 3-methyl-5-(*p*-methoxyphenyl)thien-2-ylperfluorocyclopentene was added. The reaction mixture was stirred for 3 h at that temperature. Methanol was added to stop the reaction. The reaction mixture was extracted with ether and evaporated in vacuo, then purified by column chromatography to give 1-(2,5-dimethylthien-3-yl)-2-[3-methyl-5-(*p*-methoxyphenyl)thien-2-yl]perfluorocyclopentene. Pale yellow prismatic crystals were obtained by recrystallization from hexane in 41% yield (mp 346 K). ¹H-NMR results are included in the deposited CIF-file.

Discussion

Photochromic diarylethenes that exhibit excellent properties such as thermal and chemical stability, as well as remarkable fatigue resistance, have potential capabilities for optoelectronic devices [3,4]. In solution, diarylethenes have two conformations, parallel

and antiparallel, and they interconvert. The conrotatory cyclization can take place only from the antiparallel conformation. On the other hand, two conformations are not interconvertible in the crystals. Their crystals are obtained depending on recrystallization solvents [5]. The crystals obtained by recrystallization from hexane was found to be packed in the photoactive antiparallel conformation by X-ray crystallographic analysis. The distance between the reacting atoms (C20, C22) was determined to be 3.965 Å, which is close enough for cyclization reaction [6].

Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.1 × 0.2 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	3.04 cm ⁻¹
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5023, 3903
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1705
$N(param)_{refined}$:	290
Program:	SHELXTL [7]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(8A)	4e	0.0205	−0.0371	0.2540	0.107
H(11A)	4e	−0.1233	0.0227	0.1142	0.114
H(12A)	4e	−0.2350	0.0698	−0.0817	0.118
H(14A)	4e	−0.0571	0.2377	−0.1383	0.117
H(15A)	4e	0.0534	0.1926	0.0629	0.113
H(18A)	4e	0.3906	−0.0883	0.8381	0.124
H(20A)	4e	0.3160	0.0681	0.3929	0.170
H(20B)	4e	0.3064	−0.0008	0.2812	0.170
H(20C)	4e	0.4057	0.0393	0.3321	0.170
H(21A)	4e	−0.2790	0.2627	−0.3874	0.183
H(21B)	4e	−0.1680	0.2500	−0.3624	0.183
H(21C)	4e	−0.2121	0.2930	−0.2395	0.183
H(22A)	4e	0.1864	−0.0715	0.6146	0.146
H(22B)	4e	0.1644	−0.1136	0.4578	0.146
H(22C)	4e	0.0817	−0.098	0.5466	0.146
H(23A)	4e	0.4756	−0.2173	0.8294	0.253
H(23B)	4e	0.5449	−0.2138	0.7133	0.253
H(23C)	4e	0.4455	−0.2544	0.6684	0.253

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
S(1)	4e	0.1167(1)	0.13622(9)	0.3684(2)	0.111(1)	0.086(1)	0.095(1)	−0.007(1)	0.0169(9)	0.0002(9)
S(2)	4e	0.4288(1)	−0.1129(1)	0.4760(2)	0.107(1)	0.133(2)	0.132(2)	−0.005(1)	0.032(1)	−0.045(1)
F(1)	4e	0.3672(4)	0.0278(2)	0.9663(4)	0.230(5)	0.120(3)	0.080(2)	0.017(3)	0.023(3)	0.001(2)
F(2)	4e	0.4402(3)	0.1057(3)	0.8471(5)	0.114(3)	0.178(4)	0.143(4)	−0.029(3)	0.025(3)	−0.060(3)
F(3)	4e	0.2361(4)	0.1156(3)	0.9820(5)	0.181(4)	0.210(5)	0.117(3)	0.033(4)	0.067(3)	0.006(3)
F(4)	4e	0.3280(4)	0.2021(3)	0.9287(6)	0.168(4)	0.120(3)	0.179(4)	−0.010(3)	−0.010(3)	−0.050(3)
F(5)	4e	0.1251(3)	0.1557(3)	0.7463(5)	0.120(3)	0.155(4)	0.147(4)	0.015(3)	0.036(3)	−0.048(3)
F(6)	4e	0.2350(4)	0.2133(2)	0.6567(5)	0.229(5)	0.083(3)	0.125(3)	−0.011(3)	0.038(3)	−0.002(2)
O(1)	4e	−0.2320(3)	0.1841(3)	−0.2392(5)	0.116(3)	0.118(4)	0.092(3)	−0.013(3)	0.006(3)	0.008(3)
C(1)	4e	0.2306(5)	0.0802(3)	0.6319(7)	0.115(5)	0.082(4)	0.078(4)	−0.011(4)	0.032(4)	−0.005(3)
C(2)	4e	0.3090(5)	0.0422(3)	0.6999(6)	0.105(4)	0.079(4)	0.069(4)	−0.008(3)	0.025(3)	−0.005(3)
C(3)	4e	0.3528(6)	0.0770(4)	0.8469(7)	0.112(5)	0.103(5)	0.084(4)	−0.013(4)	0.017(4)	−0.006(4)
C(4)	4e	0.2843(6)	0.1405(4)	0.8742(8)	0.128(6)	0.106(5)	0.097(5)	−0.019(5)	0.036(5)	−0.016(4)
C(5)	4e	0.2158(5)	0.1482(4)	0.7245(8)	0.114(6)	0.097(5)	0.095(5)	−0.009(4)	0.024(4)	−0.003(4)
C(6)	4e	0.1614(4)	0.0636(3)	0.4952(7)	0.101(4)	0.083(4)	0.079(4)	−0.001(3)	0.023(3)	−0.002(3)
C(7)	4e	0.1201(4)	−0.0042(3)	0.4418(7)	0.093(4)	0.083(4)	0.087(4)	−0.002(3)	0.029(3)	−0.005(3)
C(8)	4e	0.0543(4)	0.0036(4)	0.3035(7)	0.093(4)	0.085(4)	0.093(4)	−0.011(3)	0.028(3)	−0.008(4)
C(9)	4e	0.0439(4)	0.0756(4)	0.2474(7)	0.091(4)	0.089(4)	0.087(4)	−0.012(3)	0.020(3)	−0.005(3)
C(10)	4e	−0.0220(4)	0.1029(4)	0.1144(7)	0.091(4)	0.090(4)	0.080(4)	−0.005(3)	0.020(3)	−0.003(3)
C(11)	4e	−0.1099(5)	0.0667(4)	0.0657(7)	0.113(5)	0.094(4)	0.076(4)	−0.015(4)	0.016(4)	0.001(3)
C(12)	4e	−0.1768(5)	0.0947(4)	−0.0520(7)	0.108(5)	0.106(5)	0.081(4)	−0.014(4)	0.020(4)	−0.009(4)
C(13)	4e	−0.1586(5)	0.1603(4)	−0.1278(7)	0.101(5)	0.100(5)	0.065(4)	−0.001(4)	0.011(3)	−0.003(3)
C(14)	4e	−0.0713(5)	0.1953(4)	−0.0857(7)	0.107(5)	0.099(5)	0.087(4)	−0.011(4)	0.022(4)	0.000(4)
C(15)	4e	−0.0048(5)	0.1676(4)	0.0342(7)	0.095(4)	0.097(5)	0.092(5)	−0.007(4)	0.019(4)	0.000(4)
C(16)	4e	0.3715(4)	−0.0282(4)	0.4959(7)	0.094(4)	0.097(5)	0.091(4)	−0.012(4)	0.016(3)	−0.016(4)
C(17)	4e	0.3562(4)	−0.0219(4)	0.6401(7)	0.090(4)	0.091(4)	0.082(4)	−0.003(3)	0.015(3)	−0.001(3)
C(18)	4e	0.3936(5)	−0.0852(4)	0.7356(8)	0.109(5)	0.097(5)	0.107(5)	0.003(4)	0.028(4)	−0.018(4)
C(19)	4e	0.4333(5)	−0.1393(4)	0.6613(9)	0.117(6)	0.109(6)	0.134(6)	0.003(5)	0.030(5)	−0.014(5)
C(20)	4e	0.3478(5)	0.0243(4)	0.3637(7)	0.113(5)	0.144(6)	0.087(4)	−0.011(5)	0.026(4)	−0.006(4)
C(21)	4e	−0.2220(5)	0.2529(4)	−0.3130(8)	0.139(6)	0.111(5)	0.108(5)	0.000(5)	0.006(5)	0.013(5)
C(22)	4e	0.1400(5)	−0.0785(3)	0.5225(7)	0.111(5)	0.080(4)	0.103(5)	−0.005(4)	0.027(4)	0.003(4)
C(23)	4e	0.4790(6)	−0.2130(4)	0.724(1)	0.158(8)	0.115(6)	0.24(1)	0.035(6)	0.058(7)	0.007(7)

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