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Crystal structure of 4-(2-(benzo[*b*]thiophen-2-yl)-3,3,4,4,5,5-hexafluorocyclopent-1-en-1-yl)-1,5-dimethyl-1*H*-pyrrole-2-carbonitrile, C₂₀H₁₂F₆N₂S

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.28 × 0.26 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.24 mm ⁻¹
Diffractometer, scan mode:	CCD area detector, φ and ω
$\theta_{\max}$, completeness:	27.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18010, 4356, 0.018
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3687
$N(\text{param})_{\text{refined}}$:	301
Programs:	CrysAlis ^{PRO} [1], SHELX [2–4], Bruker [5]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	−0.1755(3)	0.86114(17)	0.07026(11)	0.0677(6)
C2	−0.1269(2)	0.82242(13)	0.14828(9)	0.0476(4)
C3	−0.0162(2)	0.85753(11)	0.20697(10)	0.0437(3)
H3	0.0455	0.9133	0.2042	0.052*
C4	−0.01451(17)	0.79227(10)	0.27235(8)	0.0365(3)
C5	−0.12736(18)	0.72070(11)	0.25162(9)	0.0416(3)
C6	−0.1802(3)	0.63793(15)	0.29988(14)	0.0671(5)
H6A	−0.2918	0.6469	0.3122	0.101*
H6B	−0.1122	0.6335	0.3494	0.101*
H6C	−0.1710	0.5798	0.2692	0.101*
C7	−0.3168(3)	0.68110(18)	0.12987(14)	0.0750(6)
H7A	−0.2733	0.6182	0.1211	0.112*
H7B	−0.3455	0.7111	0.0785	0.112*
H7C	−0.4122	0.6757	0.1600	0.112*
C8	0.08297(18)	0.79983(10)	0.34993(8)	0.0383(3)
C9	0.0515(3)	0.88028(13)	0.40666(11)	0.0582(5)
C10	0.1807(3)	0.87134(16)	0.47714(13)	0.0750(7)
C11	0.2611(2)	0.77387(13)	0.46559(10)	0.0537(4)
C12	0.20136(18)	0.74064(10)	0.38180(8)	0.0370(3)
C13	0.27349(17)	0.65632(10)	0.34747(8)	0.0357(3)
C14	0.38676(19)	0.59696(11)	0.38594(9)	0.0433(3)
H14	0.4274	0.6058	0.4394	0.052*
C15	0.43647(17)	0.52046(10)	0.33646(9)	0.0401(3)
C16	0.35896(17)	0.52418(10)	0.25837(9)	0.0397(3)
C17	0.5482(2)	0.44610(13)	0.35670(12)	0.0537(4)
H17	0.6033	0.4438	0.4078	0.064*
C18	0.5746(2)	0.37725(13)	0.29989(14)	0.0600(5)
H18	0.6470	0.3273	0.3130	0.072*
C19	0.4946(2)	0.38108(13)	0.22288(15)	0.0642(5)

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Abstract

C₂₀H₁₂F₆N₂S, monoclinic, *P*2₁/*c* (no. 14), *a* = 8.2091(9) Å, *b* = 13.9215(16) Å, *c* = 16.5280(19) Å, β = 94.0670(10)°, *V* = 1884.1(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0421, *wR*_{ref}(*F*²) = 0.1226, *T* = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Based on the literature method [6, 7], the title compound was obtained in 42.21% yield. The title compound was crystallized by volatilization of hexane at room temperature to get the colorless crystals for X-ray analysis.

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H19	0.5135	0.3330	0.1856	0.077*
C20	0.3877(2)	0.45464(13)	0.20036(12)	0.0562(4)
H20	0.3367	0.4576	0.1483	0.067*
F1 ^a	−0.0996(9)	0.8656(6)	0.4382(4)	0.0839(14)
F2 ^a	0.0404(7)	0.9665(5)	0.3717(4)	0.0759(12)
F3 ^a	0.1399(15)	0.8904(7)	0.5478(3)	0.111(3)
F4 ^a	0.3043(10)	0.9414(3)	0.4606(7)	0.103(2)
F5	0.2187(2)	0.71229(10)	0.52347(7)	0.0913(5)
F6	0.42441(16)	0.78170(11)	0.47524(8)	0.0873(5)
F1 ^b	−0.090(2)	0.896(2)	0.4246(13)	0.116(7)
F2 ^b	0.107(5)	0.9656(10)	0.3704(11)	0.115(6)
F3 ^b	0.0709(18)	0.8475(15)	0.5473(8)	0.089(4)
F4 ^b	0.2552(17)	0.9358(8)	0.5043(14)	0.100(5)
N1	−0.19367(16)	0.73944(10)	0.17574(8)	0.0472(3)
N2	−0.2168(3)	0.8918(2)	0.00819(12)	0.1047(8)
S1	0.22644(5)	0.62123(3)	0.24677(2)	0.04313(13)

^aOccupancy: 0.70(2), ^bOccupancy: 0.30(2).

Experimental details

The hydrogen atoms were located by geometrical calculations, and their positions and thermal parameters were fixed during the structure refinement. The occupancies of the disorder fluorine atoms at the central cyclopentene ring are refined to a 0.70(2):0.30(2) ratio for F1:F1', F2:F2', F3:F3', and F4:F4', respectively.

Comment

The functional organic optical switch materials have received more and more attention for their promising business applications in many fields, such as optical memories and switches [8–10], solar energy [11], separation [12, 13], sensors [14, 15]. Among these functional optical switch compounds, diarylethenes compounds are regarded as the best promising candidates for their excellent characteristics of reversible cyclization/cycloreversion intramolecule upon alternating irradiation with UV and visible light [16, 17]. Up to date, scientists have designed and synthesized many new diarylethenes. It is reported that some of them can exhibit the properties of photochromism in solid state and in solution [18]. However, some of them cannot show the photochromism in solid state, but they can represent good photochromism characteristics in solution [19]. Herein, a new diarylethene crystal without alkyl group at the active carbon was designed and it was shown that it cannot show photochromism in solid state and solution.

In order to investigate the structural and photochemical properties of the title diarylethene, the structure was analyzed in detail. The benzo[*b*]thiophenyl and pyrrole ring are linked by the C8=C12 double bond (1.3522(19) Å), which is shorter than the formal single bond (such as C11–C12, 1.509(2) Å and

C8–C9, 1.495(2) Å. The dihedral angle between the perfluorocyclopentene ring and the benzo[*b*]thiophenyl ring was 8.2°. The angle between the perfluorocyclopentene ring and the pyrrole ring was 63.4°.

The distance between the photoactive carbon atoms (C5–C14) was 4.938 Å. Based on the empirical rule, the parallel crystal could not display color change upon irradiation of UV light [20]. When the colorless crystals were dissolved in hexane, irradiation with UV irradiation, the colorless solution can't change the color. Compared with the other photochromic diarylethene [21, 22], the methyl group at the active atom plays a very important role for triggering the process of photochromism. Geometric parameters are all in the expected ranges [23, 24].

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