Duohua Jiang*

Crystal structure of 1-(2-methylphenyl)-2-(2-methylbenzo[*b*]thienyl)-3,3,4,4,5,5-hexafluorocyclopent-ene, C₂₁H₁₄F₆S

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.36 × 0.32 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.23 mm ⁻¹
Diffractometer, scan mode:	φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14,820, 3318, 0.016
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2740
$N(\text{param})_{\text{refined}}$:	301
Programs:	CrysAlis ^{PRO} [1], SHELX [2–4], Bruker [5]

Source of material

Based on the literature method [6], the title compound was synthesized in 41.4% yield. The title compound crystallized from hexane-dichloromethane co-solvent at room temperature.

Experimental details

The hydrogen atoms were located by geometrically 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 0.722(14):0.278(14) ratio for F1:F1', F2:F2', F3:F3', F4:F4', and F5:F5'. All H atoms attached to C were fixed geometrically and treated as riding with C–H = 0.96 Å (methyl) or 0.93 Å (phenyl and thienyl) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ (ethyl and thienyl) or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$ (methyl).

Comment

It is well known that multifunctional materials have been vastly investigated due to their promising applications in special fields. Photochromic diarylethene compounds with photoresponse characteristics are a class of multifunctional materials that can undergo fatigueresistant electrocyclicization and cycloreversion between two stable isomers. The difference in their electronic or geometric structures

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Abstract

C₂₁H₁₄F₆S, monoclinic, $P2_1/n$ (no. 14), $a = 9.9344(11)$ Å, $b = 16.7106(18)$ Å, $c = 11.6207(13)$ Å, $\beta = 101.0080(10)^\circ$, $V = 1893.7(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0491$, $wR_{\text{ref}}(F^2) = 0.1541$, $T = 296(2)$ K.

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

*Corresponding author: Duohua Jiang, Electronic and Information Engineering, Nanchang Normal College of Applied Technology, 1599 Ming Yueshan Avenue, Nanchang 330038, People's Republic of China, E-mail: 517258115@qq.com. <https://orcid.org/0000-0003-2410-1044>

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.2919 (4)	0.2165 (2)	0.1952 (3)	0.0960 (10)
H1	0.2062	0.2353	0.2038	0.115*
C2	0.2985 (3)	0.1503 (2)	0.1314 (3)	0.0954 (10)
H2	0.2189	0.1237	0.0964	0.115*
C3	0.4235 (2)	0.12272 (18)	0.1187 (3)	0.0730 (7)
H3	0.4290	0.0767	0.0747	0.088*
C4	0.5460 (2)	0.16286 (13)	0.17137 (18)	0.0504 (5)
C5	0.5393 (3)	0.23013 (16)	0.2357 (2)	0.0709 (7)
C6	0.6623 (4)	0.2745 (2)	0.2896 (4)	0.1120 (12)
H6A	0.7134	0.2438	0.3530	0.168*
H6B	0.6360	0.3247	0.3191	0.168*
H6C	0.7181	0.2842	0.2321	0.168*
C7	0.4050 (4)	0.2573 (2)	0.2476 (3)	0.0901 (8)
H7	0.3959	0.3029	0.2914	0.108*
C8	0.6773 (2)	0.12904 (11)	0.14905 (16)	0.0416 (4)
C9	0.7122 (2)	0.13644 (15)	0.0291 (2)	0.0576 (6)
C10	0.8550 (3)	0.10295 (18)	0.0399 (2)	0.0726 (8)
C11	0.8831 (3)	0.05995 (15)	0.1578 (2)	0.0621 (6)
C12	0.77315 (19)	0.08805 (11)	0.22065 (17)	0.0427 (4)
C13	0.7749 (2)	0.06330 (12)	0.34280 (17)	0.0462 (5)
C14	0.8585 (2)	0.09500 (15)	0.4377 (2)	0.0612 (6)
C15	0.9618 (3)	0.16077 (19)	0.4447 (3)	0.0870 (9)
H15A	0.9623	0.1809	0.3674	0.131*
H15B	1.0511	0.1404	0.4782	0.131*
H15C	0.9383	0.2032	0.4930	0.131*
C16	0.6903 (2)	−0.00101 (13)	0.37175 (18)	0.0499 (5)
C17	0.7130 (3)	−0.01466 (15)	0.4928 (2)	0.0646 (6)
C18	0.6404 (4)	−0.07443 (19)	0.5400 (3)	0.0872 (10)
H18	0.6546	−0.0829	0.6205	0.105*
C19	0.5491 (4)	−0.11969 (19)	0.4655 (3)	0.0927 (10)
H19	0.5012	−0.1599	0.4958	0.111*
C20	0.5258 (4)	−0.10737 (18)	0.3464 (3)	0.0859 (9)
H20	0.4621	−0.1390	0.2975	0.103*
C21	0.5956 (3)	−0.04866 (14)	0.2984 (2)	0.0624 (6)
H21	0.5796	−0.0410	0.2177	0.075*
F1 ^a	0.6945 (6)	0.2072 (4)	−0.0196 (5)	0.0827 (17)
F1 ^b	0.743 (2)	0.2217 (9)	0.0215 (17)	0.112 (5)
F2 ^a	0.6287 (10)	0.0853 (4)	−0.0493 (7)	0.0920 (17)
F2 ^b	0.621 (2)	0.1239 (17)	−0.0543 (15)	0.108 (8)
F3 ^a	0.9418 (4)	0.1714 (3)	0.0556 (6)	0.0972 (16)
F3 ^b	0.9435 (12)	0.123 (2)	−0.0027 (17)	0.147 (11)
F4 ^a	0.8823 (6)	0.0666 (4)	−0.0500 (4)	0.1025 (19)
F4 ^b	0.810 (3)	0.0245 (12)	−0.0296 (16)	0.166 (10)
F5 ^a	1.0077 (5)	0.0654 (4)	0.2135 (6)	0.098 (2)
F5 ^b	1.0104 (13)	0.0901 (14)	0.2141 (17)	0.121 (6)
F6	0.8716 (3)	−0.01986 (10)	0.1372 (2)	0.1201 (8)
S1	0.83501 (9)	0.05003 (5)	0.56699 (5)	0.0820 (3)

^aOccupancy: 0.722 (14), ^bOccupancy: 0.278 (14).

between open- and closed-ring isomers have attracted significant interest for their distinctive performance which can be used in the fields of optical memories and switches [7], solar energy [8], gas separation [9], sensors [10, 11], and so on. However, the reports focusing on the photochromic

diarylethene systems is composed of five-membered heterocyclic rings or the combination of five-membered aryl ring [12, 13]. Photochromic diarylethene compounds bearing both five- and six-membered moieties have rarely been investigated on the structure-activity relationship. For a broad understanding of the structure-activity relationship of photochromic diarylethene compounds a new 3,3,4,4,5,5-hexafluorocyclopent-ene derivative was synthesized and its crystal structure was analyzed in detail.

The molecular structure of the title compound is shown in the Figure. In the perfluorocyclopentene ring, the benzothienyl and phenyl rings are linked by the C12=C8 double bond (1.329(3) Å), which is shorter than the formal single bond (such as C12–C11, 1.501(3) Å and C8–C4, 1.489(3) Å). The molecule includes three kinds of planar rings and they can form three dihedral angles. The dihedral angle between the perfluorocyclopentene ring and the adjacent benzothiophene ring was 105.7°. The dihedral angles between the perfluorocyclopentene ring and the adjacent six-membered phenyl ring was 104.5°. And the dihedral angle between the benzothiophene ring and the phenyl ring is 77.38°. Bond lengths are all in the expected ranges [12, 14, 15].

It is found that the molecule is packed in a parallel conformation, and the intramolecular distance between the two reactive carbon atoms (C5...C14) was 4.222(2) Å [16, 17]. The former researcher concluded that in the case that the distance of reactive carbon atoms is larger than 4.2 Å, its crystal cannot display color change upon irradiation by UV light [16]. When the colorless crystals were dissolved in hexane, irradiation with UV irradiation, the colorless solution did not changed its color. Compared with the other photochromic diarylethene [7, 18], the anti-parallel formation and the distance of reactive carbon atoms play a very important role in triggering the process of photochromism.

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