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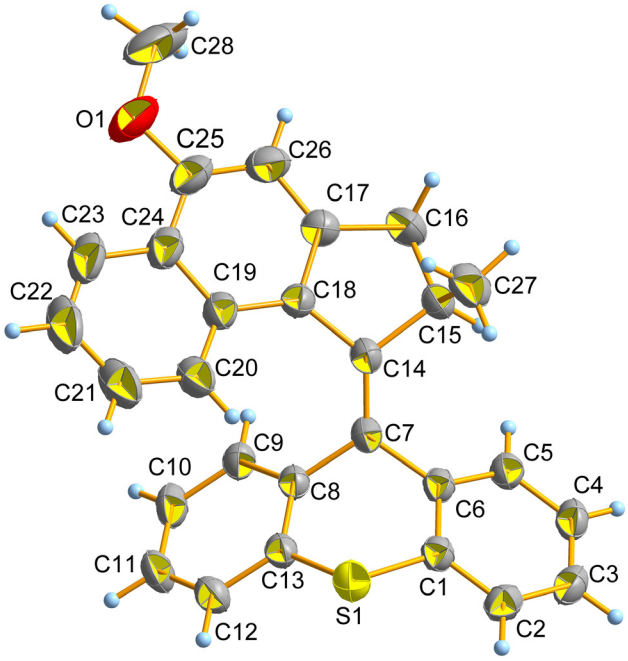
The crystal structure of (*R*)-9-(5-methoxy-2-methyl-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-1-ylidene)-9*H*-thioxanthene, $C_{28}H_{22}OS$



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Abstract

$C_{28}H_{22}OS$, monoclinic, $P2_1/c$ (no. 14), a = 21.690(3) Å, b = 5.9871(10) Å, c = 17.587(3) Å, β = 112.993(2)°, V = 2102.4(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0449, $wR_{ref}(F^2)$ = 0.1171, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Block
Size:	0.48 × 0.43 × 0.37 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22,226, 4726, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	3500
$N(param)_{refined}$:	273
Programs:	Bruker [1], SHELX [2, 3]

1 Source of material

The (9*H*-thioxanthen-9-ylidene)hydrazine (0.45 g, 2 mmol) was dissolved in THF (20 mL) and the solution cooled in an icebath. MnO_2 (1.75 g, 20 mmol) was added and the mixture stirred at 0 °C for 1 h. After warming to RT, the mixture was filtered over a silica plug and concentrated under reduced pressure. The resulting diazo compound was used without further purification. (*R*)-5-methoxy-2-methyl-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalene-1-thione (0.48 g, 2 mmol) was redissolved in THF (20 mL) and added to a solution of the diazo compound in THF (20 mL). The mixture was heated at reflux overnight. After cooling to room temperature, the mixture was concentrated under reduced pressure and the residue purified by flash column chromatography (SiO_2, n-pentane:EtOAc 6:1) yielding the title compound. The crystals of the product can be obtained by recrystallization in ethanol.

2 Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the methyl groups were set to $1.5U_{eq}$(C), and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$(C).

3 Comment

Thioxanthene is a sulfur-containing polycyclic aromatic hydrocarbon compound, and its derivatives can be used as

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.09262 (8)	0.3163 (3)	0.52215 (10)	0.0367 (4)
C2	0.03862 (8)	0.2603 (3)	0.44980 (10)	0.0443 (4)
H2	0.0176	0.1227	0.4454	0.053*
C3	0.01628 (9)	0.4084 (3)	0.38483 (11)	0.0485 (4)
H3	−0.0187	0.3690	0.3358	0.058*
C4	0.04610 (9)	0.6161 (3)	0.39275 (11)	0.0464 (4)
H4	0.0301	0.7180	0.3495	0.056*
C5	0.09949 (8)	0.6730 (3)	0.46456 (10)	0.0397 (4)
H5	0.1183	0.8145	0.4694	0.048*
C6	0.12581 (7)	0.5216 (3)	0.53021 (9)	0.0343 (3)
C7	0.18379 (8)	0.5693 (2)	0.60932 (9)	0.0331 (3)
C8	0.16835 (7)	0.5177 (3)	0.68260 (9)	0.0333 (3)
C9	0.18126 (8)	0.6714 (3)	0.74654 (9)	0.0392 (4)
H9	0.1998	0.8094	0.7435	0.047*
C10	0.16675 (9)	0.6209 (3)	0.81439 (10)	0.0491 (4)
H10	0.1752	0.7251	0.8565	0.059*
C11	0.13952 (10)	0.4144 (4)	0.81974 (11)	0.0544 (5)
H11	0.1314	0.3786	0.8665	0.065*
C12	0.12443 (9)	0.2627 (3)	0.75639 (10)	0.0464 (4)
H12	0.1053	0.1258	0.7597	0.056*
C13	0.13795 (8)	0.3151 (3)	0.68708 (9)	0.0367 (4)
C14	0.24354 (8)	0.6499 (3)	0.61486 (9)	0.0354 (3)
C15	0.25879 (9)	0.7482 (3)	0.54395 (10)	0.0432 (4)
H15	0.2179	0.8111	0.5021	0.052*
C16	0.30851 (10)	0.9368 (3)	0.58619 (12)	0.0547 (5)
H16A	0.3409	0.9552	0.5612	0.066*
H16B	0.2854	1.0772	0.5832	0.066*
C17	0.34162 (9)	0.8578 (3)	0.67370 (11)	0.0457 (4)
C18	0.30588 (8)	0.6863 (3)	0.68915 (10)	0.0381 (4)
C19	0.33353 (8)	0.5711 (3)	0.76654 (10)	0.0426 (4)
C20	0.30894 (9)	0.3658 (3)	0.78285 (12)	0.0508 (5)
H20	0.2731	0.2969	0.7413	0.061*
C21	0.33698 (11)	0.2668 (4)	0.85886 (14)	0.0714 (7)
H21	0.3201	0.1316	0.8684	0.086*
C22	0.39090 (12)	0.3680 (5)	0.92243 (15)	0.0846 (9)
H22	0.4080	0.3045	0.9748	0.102*
C23	0.41821 (11)	0.5580 (5)	0.90776 (13)	0.0741 (7)
H23	0.4548	0.6211	0.9500	0.089*
C24	0.39215 (9)	0.6625 (4)	0.82945 (11)	0.0537 (5)
C25	0.42458 (9)	0.8497 (4)	0.81064 (13)	0.0602 (5)
C26	0.40122 (10)	0.9409 (4)	0.73378 (13)	0.0580 (5)
H26	0.4243	1.0564	0.7212	0.070*
C27	0.28803 (11)	0.5740 (4)	0.50471 (13)	0.0633 (6)
H27A	0.3266	0.5066	0.5465	0.095*
H27B	0.3010	0.6441	0.4641	0.095*
H27C	0.2550	0.4612	0.4787	0.095*
C28	0.51688 (13)	1.1039 (6)	0.86103 (19)	0.1084 (11)
H28A	0.5357	1.0600	0.8222	0.163*
H28B	0.5522	1.1469	0.9121	0.163*
H28C	0.4871	1.2279	0.8392	0.163*
O1	0.48061 (8)	0.9208 (3)	0.87543 (10)	0.0895 (6)
S1	0.11780 (2)	0.12333 (7)	0.60497 (3)	0.04599 (14)

an important class of antipsychotic drugs for the treatment of psychiatric patients [4, 5]. At the same time, thioxanthene derivatives can also be used for chiral molecular switches, molecular motors, and organic light-emitting diodes [6–8]. Because of its wide range of uses and special structure, thioxanthene has always attracted the attention of scientists [9, 10].

In the crystal structure, six rings make up two sets of fused rings, which connect to each other with C(7)–C(14). The bond lengths of S(1)–C(1), S(1)–C(13), C(7)–C(8), C(7)–C(6) and C(7)–C(14) are 1.7702(17), 1.7611(17), 1.485(2), 1.494(2) and 1.350(2) Å, respectively. The dihedral angle between the plane through C(1)–C(6) and the plane through C(8)–C(13) is 50.8°. The bond lengths of C(15)–C(14), C(15)–C(16) and C(15)–C(27) are 1.527(2), 1.537(3) and 1.519(3) Å, respectively. Meanwhile, the C(15) atom is an R-type chiral carbon. The atoms of C(14), C(16), C(17) and C(18) are almost coplanar.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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