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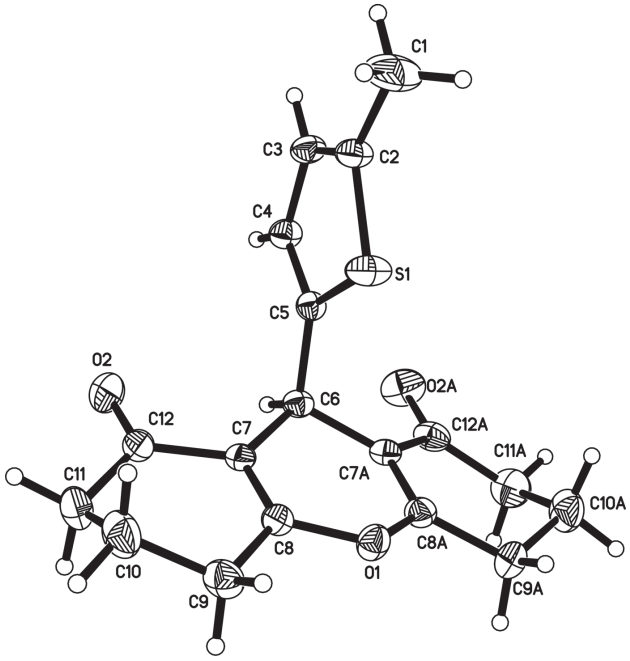
Crystal structure of 9-(5-methylthiophen-2-yl)-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione, $C_{18}H_{18}O_3S$



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Abstract

$C_{18}H_{18}O_3S$, orthorhombic, $Cmc2_1$ (no. 36), $a = 14.463(3)$ Å, $b = 9.780(5)$ Å, $c = 10.848(4)$ Å, $V = 1534.4(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0416$, $wR_{ref}(F^2) = 0.1132$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.36 × 0.29 × 0.24 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.22 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}:	4404, 1524, 0.051
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1386
$N(param)_{refined}$:	113
Programs:	Olex2 [1], SHELX [2], Bruker [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.500000	0.84044(10)	0.61604(10)	0.0493(4)
O1	0.500000	1.2222(3)	0.6784(3)	0.0412(7)
O2	0.32316(19)	0.9455(3)	0.9404(3)	0.0635(8)
C1	0.500000	0.5858(7)	0.5035(6)	0.080(2)
H1A	0.500000	0.647578	0.434735	0.120*
H1B[a]	0.554196	0.529214	0.500124	0.120*
H1C[a]	0.445804	0.529214	0.500124	0.120*
C2	0.500000	0.6648(4)	0.6197(6)	0.0411(8)
C3	0.500000	0.6197(4)	0.7358(4)	0.0398(9)
H3	0.500000	0.527588	0.757069	0.048*
C4	0.500000	0.7274(4)	0.8230(4)	0.0369(8)
H4	0.500000	0.712561	0.907691	0.044*
C5	0.500000	0.8530(4)	0.7726(4)	0.0300(7)
C6	0.500000	0.9894(4)	0.8367(3)	0.0325(8)
H6	0.500000	0.972182	0.925708	0.039*
C7	0.41586(17)	1.0727(3)	0.8072(3)	0.0323(6)
C8	0.41896(17)	1.1767(3)	0.7290(3)	0.0355(6)
C9	0.3389(2)	1.2602(3)	0.6873(4)	0.0487(8)
H9A	0.344353	1.277658	0.599589	0.058*
H9B	0.339481	1.347458	0.729732	0.058*
C10	0.2489(2)	1.1880(4)	0.7128(4)	0.0598(10)
H10A	0.197936	1.251359	0.701983	0.072*
H10B	0.240906	1.113568	0.654602	0.072*
C11	0.2477(2)	1.1325(4)	0.8420(4)	0.0587(10)
H11A	0.248637	1.208155	0.899779	0.070*
H11B	0.190638	1.082229	0.854862	0.070*
C12	0.3278(2)	1.0403(3)	0.8681(3)	0.0431(7)

[a]Occupancy: 0.5.

Source of material

The title compound was synthesized according to a reported procedure. A mixture of 3,5-cyclohexanedione (20 mmol) and 5-methyl-2-thiophenecarboxaldehyde (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model, with C—H = 0.93/0.96/0.97 Å with U_{iso}(H) = 1.2 times U_{eq}(C).

Comment

Many enone derivatives have received considerable attention in the past few years for their versatile biological and medical activities [4]. Different substituents at the aromatic ring lead to multiple chemical modifications which may generates antioxidant, anti-inflammatory, antibacterial and anticancer activities for these compounds. Recognizing the considerable importance of the compounds, research focused on the synthesis of enone derivatives [5].

In the crystal structure of the title compound (figure) the molecule show a crystallographically imposed mirror symmetry. The bond lengths presented in the title compound are all in their normal ranges and they are similar with the known compounds [6–9].

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