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The crystal structure of thioxanthen-9-one-10,10-dioxide, $C_{13}H_8O_3S$ – a second polymorph

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.56 \times 0.55 \times 0.43$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.31 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	28.0° , 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4481, 1212, 0.012
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1161
$N(\text{param})_{\text{refined}}$:	79
Programs:	Bruker [1, 2], SHELX [3], WinGX/ORTEP [4], Mercury [5], PLATON [6]

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Abstract

$C_{13}H_8O_3S$, monoclinic, $C2/c$ (no. 15), $a = 9.7499(3)$ Å, $b = 12.9758(4)$ Å, $c = 8.2681(3)$ Å, $\beta = 103.784(2)^\circ$, $V = 1015.90(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0318$, $wR_{\text{ref}}(F^2) = 0.0927$, $T = 200$ 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.

Source of material

Thioxanthen-9-one 10,10-dioxide was obtained in low yield from the reaction of 9-phenylthioxanthen-9-ol with hydrogen peroxide in acid medium according to a

published procedure [7]. Crystals were obtained upon recrystallization of the compound from chloroform.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$.

Comment

Saturated six-membered ring-systems adopt, in general, energetically-favourable *chair* conformations. Endowing the parent ring with sterically-demanding or rigid substituents or entering the realm of partial unsaturation, other conformations may become feasible. In our continuous interest in the conformational behaviour of five-, six-, seven- and eight-membered non-aromatic heterocyclic ring-systems [8–19], we determined the crystal structure of the title compound to enable comparisons of metric parameters with related ring-systems. Although the crystal structure of the title compound has already been reported in the literature [20] – the refinement of the model also included the hydrogen atoms – the 3D coordinates of the latter atoms were not deposited. Additionally, a different space group – $P2_1/n$ – was reported by these authors. Furthermore, the crystal structures of asymmetric thioxanthen-9-one-10,10-dioxides with methyl

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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}
S1	0.000000	0.19855 (3)	0.750000	0.02738 (17)
O1	0.000000	−0.14230 (12)	0.750000	0.0498 (5)
O2	−0.06422 (12)	0.25645 (9)	0.60344 (15)	0.0421 (3)
C1	0.000000	−0.04917 (15)	0.750000	0.0281 (4)
C11	0.12694 (13)	0.11582 (10)	0.70096 (15)	0.0232 (3)
C12	0.11444 (13)	0.00847 (10)	0.69697 (15)	0.0232 (3)
C13	0.21766 (15)	−0.04847 (11)	0.64499 (17)	0.0276 (3)
H13	0.210461	−0.121457	0.639172	0.033*
C14	0.33007 (15)	−0.00015 (12)	0.60193 (18)	0.0319 (3)
H14	0.399167	−0.039970	0.566258	0.038*
C15	0.34253 (15)	0.10669 (12)	0.61059 (19)	0.0333 (3)
H15	0.420793	0.139679	0.582694	0.040*
C16	0.24079 (14)	0.16460 (11)	0.65985 (18)	0.0299 (3)
H16	0.248663	0.237548	0.665604	0.036*

as well as cyano groups on the aromatic system are apparent in the literature [21, 22].

The title crystal structure shows the presence of the expected functionalized derivative of anthracene. The asymmetric unit is comprised of half a molecule. The S=O bond length of 1.4364(11) Å and the C=O bond length of 1.209(2) Å are in good agreement with data found for other thioxanthene-10,10-dioxides and ketones whose crystal structure data has been deposited with the Cambridge Structural Database [23]. The least-squares planes defined by the six atoms of the central heterocycle on the one hand and the C atoms of the aromatic moiety on the other hand enclose an angle of 3.52(5)° while the least-squares planes of both aromatic moieties intersect at an angle of 7.04(6)°. A conformational analysis of the six-membered heterocycle according to Cremer & Pople [24] is precluded by the small puckering amplitude ($\tau = 3.9^\circ$).

In the crystal structure, C–H⋯O contacts whose range falls slightly below the sum of van-der-Waals radii of the atoms involved are present. These are apparent between hydrogen atoms of the phenyl groups in *meta*-position to the connection points of the central heterocycle and exclusively apply sulfur-bonded oxygen atoms as acceptor. In terms of graph-set analysis [25, 26], the description of these contacts necessitate a $C_1^1(6)C_1^1(7)$ descriptor on the unary level. Potential C–O⋯ π interactions can be ruled out on grounds of the acuteness of the pertaining angles. In total, the molecules are connected to a three-dimensional network. The shortest intercentroid distance between two centers of gravity was found at 3.8871(7) Å with the shortest distance between two π -systems measured at 4.3980(8) Å.

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