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The crystal structure of (Z)-2-(tert-butyl)-6-(7-(tert-butyl)-5-methylbenzo[d][1,3]oxathiol-2-ylidene)-4-methylcyclohexa-2,4-dien-1-one, $C_{23}H_{28}O_2S$

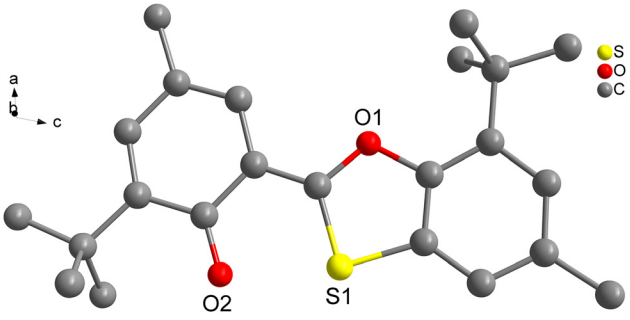

Table 1: Data collection and handling.

Crystal:	Clear light yellow needle
Size:	0.13 × 0.12 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	1.58 mm^{-1}
Diffractometer, scan mode:	Rigaku synergy, *ω* scans
θ_{max}, completeness:	77.6°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6475, 2074, 0.020
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1981
$N(param)_{refined}$:	156
Programs:	Rigaku[1] Olex2,[2,3] SHELX.[4]

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Abstract

$C_{23}H_{28}O_2S$, monoclinic, $P2_1/m$ (no. 11), a = 9.3198(3) Å, b = 7.0131(2) Å, c = 15.4566(4) Å, β = 106.236(3)°, V = 969.96(5) $Å^3$, Z = 2, $R_{gt}(F)$ = 0.0335, $wR_{ref}(F^2)$ = 0.0922, T = 100 K.

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

1 Source of materials

The 2-(*tert*-butyl)-6-((3-(*tert*-butyl)-2-hydroxy-5-methylbenzyl) thio)-4-methylphenol (3.59 g, 10 mmol), sodium methoxide (5.04 g, 100 mmol), and lanthanum chloride hexahydrate (1.77 g, 5 mmol) were dissolved in tetrahydrofuran (15 g) and stirred. The mixture was stirred at 66 °C for 3 h, then cooled to room temperature. The solution was evaporated to dryness under reduced pressure by rotary evaporator, and chloroform (50 g) and water (50 g) were added. After stirring for 10 min, the layers were separated, and the organic layer was retained. The organic layer was evaporated by rotary evaporator to obtain a brown solid. The brown solid was dissolved in a chloroform/methanol = 1/2 mixture and slowly evaporated at room temperature to obtain crystals.

2 Experimental details

Data were collected with CrysAlis^{PRO}.[1] The structure was solved with the olex2. solve structure solution program using Charge Flipping and refined with the SHELXL refinement package.[2,3] Hydrogen atoms were placed in their geometrically idealized positions. Hydrogen atoms were constrained to ride on their parent atoms.

3 Comment

The title molecule in the crystal structure is a rigid molecule. Each title molecule contains one benzoyl group, one ether group and one thioether group. The C–S distances are in the range of 1.7217(17)–1.7547(16) Å.[5] The C–O distances are in the range of 1.258(2)–1.3983(18) Å.[6] The C–C distances are in the range of 1.359(2) to 1.5403(16) Å.[7] The bond lengths are all in the expected ranges.

The crystals were mainly stabilized by the intramolecular hydrogen bonds and C–H···π interactions. Only two hydrogens are involved in the creation of intramolecular hydrogen bonds, which bind together adjacent atoms within a molecule.[8,9]

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

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