Crystal structure of 3,8-di-*tert*-butyl-4,9,10-trimethoxy-1-methyl-dibenzo[*b*,*d*]pyran-6-thione, $C_6H(OCH_3)_2[C(CH_3)_3]CSO(C_6H)(CH_3)(OCH_3)[C(CH_3)_3]$

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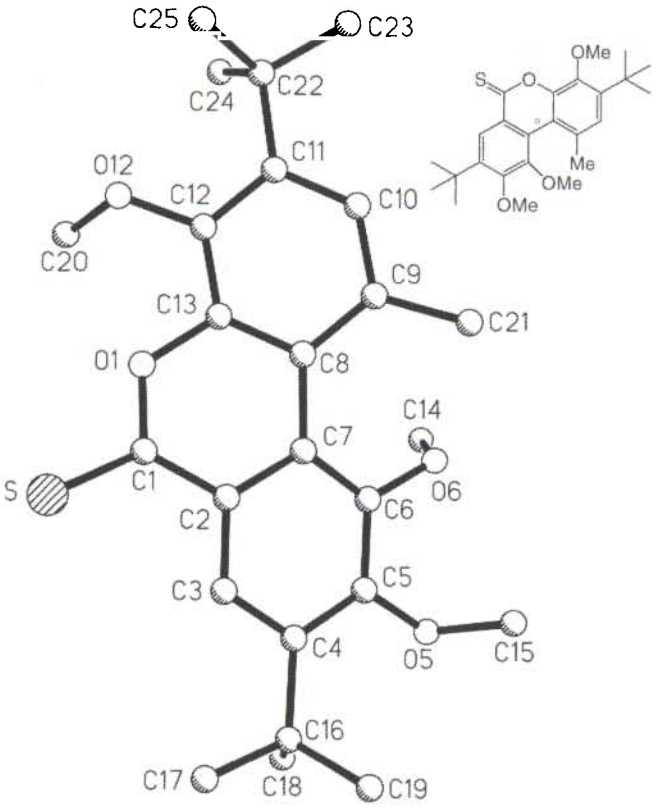

Abstract

$C_{25}H_{32}O_4S$, monoclinic, $P12_1/c1$ (No. 14), a = 13.704(1) Å, b = 10.530(1) Å, c = 16.248(1) Å, β = 92.37(1)°, V = 2342.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.062, $wR(F)$ = 0.058, T = 293 K.

Source of material:

The title compound, a potential substrate for the atroposelective ring cleavage reaction according to the 'lactone method' [1], was synthesized by treating the corresponding oxo-compound [2] with Lawesson's reagent.

Table 1. Data collection and handling.

Crystal:	yellow plate, size 0.2 × 0.35 × 0.65 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.70 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5380, 5380
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 3700
$N(param)_{refined}$:	271
Program:	SHELXTL-plus [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4*e*	0.7359(2)	−0.0679(3)	0.2775(2)	0.08
H(10)	4*e*	0.3920(2)	−0.2040(2)	−0.1026(2)	0.08
H(14A)	4*e*	0.6181(3)	−0.5369(3)	0.0148(2)	0.08
H(14B)	4*e*	0.5627(3)	−0.4589(3)	0.0804(2)	0.08
H(14C)	4*e*	0.6630(3)	−0.5233(3)	0.1046(2)	0.08
H(15A)	4*e*	0.9249(2)	−0.4532(4)	0.0421(2)	0.08
H(15B)	4*e*	0.9125(2)	−0.3053(4)	0.0372(2)	0.08
H(15C)	4*e*	0.8262(2)	−0.3944(4)	0.0080(2)	0.08
H(17A)	4*e*	0.8479(3)	−0.1047(4)	0.3600(2)	0.08
H(17B)	4*e*	0.8996(3)	−0.0215(4)	0.2947(2)	0.08
H(17C)	4*e*	0.9621(3)	−0.1052(4)	0.3568(2)	0.08
H(18A)	4*e*	0.8966(3)	−0.4040(4)	0.2727(2)	0.08
H(18B)	4*e*	0.8464(3)	−0.3395(4)	0.3469(2)	0.08
H(18C)	4*e*	0.9605(3)	−0.3394(4)	0.3432(2)	0.08
H(19A)	4*e*	0.9817(2)	−0.2680(4)	0.1693(2)	0.08
H(19B)	4*e*	1.0433(2)	−0.2037(4)	0.2413(2)	0.08
H(19C)	4*e*	0.9808(2)	−0.1199(4)	0.1793(2)	0.08
H(20A)	4*e*	0.2244(2)	−0.0709(4)	0.2392(2)	0.08
H(20B)	4*e*	0.3352(2)	−0.1085(4)	0.2425(2)	0.08
H(20C)	4*e*	0.2570(2)	−0.2022(4)	0.2041(2)	0.08
H(21A)	4*e*	0.6426(2)	−0.2299(3)	−0.0545(2)	0.08
H(21B)	4*e*	0.5779(2)	−0.1588(3)	−0.1216(2)	0.08
H(21C)	4*e*	0.5630(2)	−0.3045(3)	−0.1069(2)	0.08
H(23A)	4*e*	0.2495(2)	−0.1427(4)	−0.1532(2)	0.08
H(23B)	4*e*	0.1464(2)	−0.2005(4)	−0.1380(2)	0.08
H(23C)	4*e*	0.2405(2)	−0.2831(4)	−0.1224(2)	0.08
H(24A)	4*e*	0.1678(2)	−0.2163(4)	0.0795(2)	0.08
H(24B)	4*e*	0.1910(2)	−0.3275(4)	0.0192(2)	0.08
H(24C)	4*e*	0.0969(2)	−0.2448(4)	0.0036(2)	0.08
H(25A)	4*e*	0.1805(2)	0.0118(3)	0.0278(2)	0.08
H(25B)	4*e*	0.1096(2)	−0.0233(3)	−0.0470(2)	0.08
H(25C)	4*e*	0.2120(2)	0.0362(3)	−0.0625(2)	0.08

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	4*e*	0.57356(6)	0.07562(8)	0.28823(5)	0.0599(5)	0.0656(5)	0.0562(5)	–0.0004(4)	0.0008(4)	–0.0252(4)
O(1)	4*e*	0.4700(1)	–0.0537(2)	0.1848(1)	0.038(1)	0.052(1)	0.041(1)	0.0036(9)	0.0004(8)	–0.0114(9)
C(1)	4*e*	0.5616(2)	–0.0327(2)	0.2161(2)	0.042(2)	0.043(2)	0.038(1)	–0.001(1)	0.001(1)	–0.002(1)
C(2)	4*e*	0.6373(2)	–0.1131(2)	0.1843(2)	0.036(1)	0.041(1)	0.039(1)	–0.002(1)	0.002(1)	–0.002(1)
C(3)	4*e*	0.7259(2)	–0.1222(3)	0.2305(2)	0.042(2)	0.054(2)	0.039(1)	–0.005(1)	–0.001(1)	–0.001(1)
C(4)	4*e*	0.7987(2)	–0.2055(3)	0.2111(2)	0.037(1)	0.050(2)	0.044(2)	–0.001(1)	–0.000(1)	0.008(1)
C(5)	4*e*	0.7777(2)	–0.2872(3)	0.1449(2)	0.035(1)	0.043(2)	0.047(2)	0.005(1)	0.002(1)	0.008(1)
O(5)	4*e*	0.8435(1)	–0.3818(2)	0.1284(1)	0.045(1)	0.059(1)	0.063(1)	0.015(1)	0.0036(9)	0.003(1)
C(6)	4*e*	0.6892(2)	–0.2809(2)	0.0984(2)	0.036(1)	0.039(1)	0.041(1)	–0.001(1)	0.004(1)	0.002(1)
O(6)	4*e*	0.6735(1)	–0.3699(2)	0.0381(1)	0.055(1)	0.040(1)	0.051(1)	0.0024(9)	0.0003(9)	–0.0073(9)
C(7)	4*e*	0.6198(2)	–0.1875(2)	0.1136(1)	0.034(1)	0.035(1)	0.037(1)	–0.004(1)	0.003(1)	0.003(1)
C(8)	4*e*	0.5265(2)	–0.1693(2)	0.0662(1)	0.034(1)	0.028(1)	0.039(1)	–0.000(1)	0.001(1)	0.001(1)
C(9)	4*e*	0.5037(2)	–0.1959(2)	–0.0174(1)	0.039(1)	0.030(1)	0.038(1)	–0.001(1)	0.003(1)	0.001(1)
C(10)	4*e*	0.4068(2)	–0.1857(2)	–0.0455(2)	0.040(1)	0.037(1)	0.037(1)	–0.005(1)	0.001(1)	–0.001(1)
C(11)	4*e*	0.3291(2)	–0.1500(2)	0.0029(2)	0.037(1)	0.034(1)	0.044(1)	–0.004(1)	–0.001(1)	0.005(1)
C(12)	4*e*	0.3544(2)	–0.1106(2)	0.0828(2)	0.034(1)	0.037(1)	0.042(1)	0.002(1)	0.005(1)	0.003(1)
O(12)	4*e*	0.2848(1)	–0.0597(2)	0.1318(1)	0.037(1)	0.058(1)	0.042(1)	0.0103(9)	0.0074(8)	0.0020(9)
C(13)	4*e*	0.4520(2)	–0.1150(2)	0.1103(2)	0.038(1)	0.036(1)	0.034(1)	–0.000(1)	0.001(1)	0.000(1)
C(14)	4*e*	0.6258(3)	–0.4812(3)	0.0613(2)	0.108(3)	0.063(2)	0.081(3)	–0.025(2)	0.012(2)	–0.011(2)
C(15)	4*e*	0.8799(2)	–0.3841(4)	0.0473(2)	0.052(2)	0.088(3)	0.084(3)	0.015(2)	0.017(2)	–0.006(2)
C(16)	4*e*	0.8954(2)	–0.2108(3)	0.2629(2)	0.040(2)	0.069(2)	0.054(2)	–0.005(2)	–0.011(1)	0.009(2)
C(17)	4*e*	0.9020(3)	–0.1003(4)	0.3244(2)	0.060(2)	0.121(3)	0.084(3)	0.003(2)	–0.033(2)	–0.019(2)
C(18)	4*e*	0.9002(3)	–0.3347(4)	0.3110(2)	0.058(2)	0.109(3)	0.089(3)	–0.008(2)	–0.023(2)	0.041(2)
C(19)	4*e*	0.9837(2)	–0.1994(4)	0.2083(2)	0.042(2)	0.105(3)	0.086(3)	–0.012(2)	–0.007(2)	0.017(2)
C(20)	4*e*	0.2746(2)	–0.1145(4)	0.2109(2)	0.052(2)	0.104(3)	0.048(2)	0.016(2)	0.013(1)	0.008(2)
C(21)	4*e*	0.5788(2)	–0.2249(3)	–0.0809(2)	0.045(2)	0.055(2)	0.039(1)	0.006(1)	0.007(1)	0.001(1)
C(22)	4*e*	0.2224(2)	–0.1530(3)	–0.0297(2)	0.033(1)	0.057(2)	0.050(2)	–0.005(1)	–0.001(1)	–0.000(1)
C(23)	4*e*	0.2137(2)	–0.1991(4)	–0.1192(2)	0.042(2)	0.091(3)	0.061(2)	–0.005(2)	–0.012(1)	–0.014(2)
C(24)	4*e*	0.1639(2)	–0.2437(4)	0.0231(2)	0.054(2)	0.081(2)	0.084(2)	–0.027(2)	0.001(2)	0.006(2)
C(25)	4*e*	0.1767(2)	–0.0196(3)	–0.0278(2)	0.043(2)	0.072(2)	0.064(2)	0.014(2)	–0.009(1)	0.004(2)

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