Crystal structure of 1-oxa-6-thia-7,10-bis(trimethylsilyl)-11-sulfoxide-spiro[4.4.5]undeca-3,8-dien-2-one, $C_{14}H_{22}O_3S_2Si_2$

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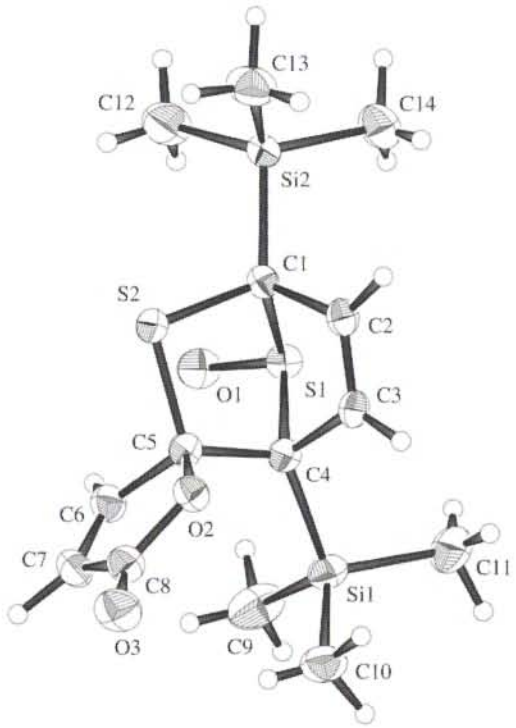

Abstract

$C_{14}H_{22}O_3S_2Si_2$, monoclinic, $P12_1/a1$ (No. 14), a = 12.267(1) Å, b = 11.548(1) Å, c = 13.242(1) Å, β = 91.13(1)°, V = 1875.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.034, $wR(F)$ = 0.036, T = 296 K.

Source of material

The title compound (10% yield) was prepared by irradiating 2,5-bis(trimethylsilyl)-thiophene *S*-oxide with UV from a high pressure Hg lamp for 12 hours. Suitable crystals of the compound were obtained by slow recrystallization from chloroform.

Discussion

This structural analysis is part of the preparation and reactivity study of thiophene monooxides compounds [1-3].The ORTEP plot (30% probability ellipsoids) shows the structure of the unusual thiophene derivative. The bond distances, d(C2—C3) = 1.322(3) A, d(C6—C7) = 1.317(3) Å, d(S1—O1) = 1.490(1) Å and d(O3—C8) = 1.201(2) Å, identifies the respective carbon-carbon, sulfur-oxygen and oxygen-carbon double bonds in the molecules. The newly formed single bonds are represented by larger distances: d(C1—C2) = 1.507(2) Å and d(C3—C4) = 1.510(2) Å. Futhermore, the molecule is the result of the Diels-Alder addition of a thiofurane group to the thiophene S-oxide precursor's 2,5 ring positions. The connecting bond distances are d(S2—C1) = 1.829(2) Å and d(C4—C5) = 1.556(2) Å. All other non-hydrogen bonds are normal.

Table 1. Data collection and handling.

Crystal:	brown, block, size 0.5 × 0.5 × 1.2 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	4.17 cm^{-1}
Diffractometer, scan mode:	Rigaku CAD4, ω/2θ
$2\theta_{max}$:	59.99°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5929, 5711
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 5\ \sigma(I_{obs})$, 2670
$N(param)_{refined}$:	248
Programs:	teXsan [4], DIFABS [5], ORTEPII [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	1.025(1)	0.265(2)	0.244(1)	0.049(5)
H(2)	4e	1.024(1)	0.074(1)	0.310(1)	0.037(5)
H(3)	4e	0.621(2)	0.061(2)	0.339(2)	0.064(6)
H(4)	4e	0.606(2)	0.002(2)	0.534(2)	0.071(7)
H(5)	4e	0.689(3)	−0.127(3)	0.201(3)	0.111
H(6)	4e	0.666(3)	−0.157(4)	0.312(2)	0.111
H(7)	4e	0.726(3)	−0.245(3)	0.245(3)	0.111
H(8)	4e	0.855(2)	−0.155(3)	0.461(3)	0.087
H(9)	4e	0.925(3)	−0.245(3)	0.404(3)	0.087
H(10)	4e	0.972(2)	−0.123(3)	0.428(3)	0.087
H(11)	4e	0.979(4)	−0.232(3)	0.203(4)	0.155
H(12)	4e	1.036(4)	−0.114(4)	0.222(4)	0.155
H(13)	4e	0.941(4)	−0.123(5)	0.144(4)	0.155
H(14)	4e	0.670(4)	0.339(3)	0.066(4)	0.143
H(15)	4e	0.653(4)	0.424(4)	0.155(3)	0.143
H(16)	4e	0.685(4)	0.471(4)	0.051(3)	0.143
H(17)	4e	0.858(4)	0.540(5)	0.253(3)	0.156
H(18)	4e	0.900(4)	0.580(4)	0.149(3)	0.156
H(19)	4e	0.970(3)	0.498(4)	0.216(4)	0.156
H(20)	4e	0.915(3)	0.402(3)	−0.033(3)	0.099
H(21)	4e	0.986(3)	0.332(3)	0.043(3)	0.099
H(22)	4e	0.890(3)	0.273(3)	−0.014(3)	0.099

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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}
(1)	4*e*	0.80069(4)	0.11237(4)	0.16911(3)	0.0401(2)	0.0419(2)	0.0370(2)	–0.0016(2)	–0.0041(2)	–0.0012(2)
S(2)	4*e*	0.76248(4)	0.27045(4)	0.33245(4)	0.0438(2)	0.0398(2)	0.0434(2)	0.0055(2)	0.0077(2)	0.0010(2)
Si(1)	4*e*	0.85593(5)	–0.11109(5)	0.29235(4)	0.0487(3)	0.0361(3)	0.0551(3)	0.0000(3)	–0.0061(2)	0.0004(3)
Si(2)	4*e*	0.83590(4)	0.38073(5)	0.12735(4)	0.0433(3)	0.0453(3)	0.0470(3)	0.0021(3)	0.0083(2)	0.0106(3)
O(1)	4*e*	0.6795(1)	0.1060(1)	0.1616(1)	0.0417(7)	0.0581(8)	0.0613(8)	–0.0084(7)	–0.0181(6)	0.0061(7)
O(2)	4*e*	0.8407(1)	0.1142(1)	0.46815(9)	0.0424(7)	0.0533(8)	0.0356(6)	–0.0029(6)	–0.0018(5)	0.0024(6)
O(3)	4*e*	0.8085(1)	0.0521(1)	0.6256(1)	0.083(1)	0.074(1)	0.0389(7)	0.0024(9)	–0.0003(7)	0.0057(8)
C(1)	4*e*	0.8459(1)	0.2549(1)	0.2203(1)	0.0355(8)	0.0379(8)	0.0376(9)	–0.0015(7)	0.0019(6)	0.0001(7)
C(2)	4*e*	0.9591(1)	0.2177(2)	0.2526(1)	0.0306(8)	0.048(1)	0.043(1)	–0.0027(8)	0.0004(7)	–0.0017(8)
C(3)	4*e*	0.9616(1)	0.1117(2)	0.2905(1)	0.0278(8)	0.0452(9)	0.0426(9)	0.0036(8)	–0.0014(7)	–0.0023(8)
C(4)	4*e*	0.8506(1)	0.0552(1)	0.2920(1)	0.0338(8)	0.0383(8)	0.0364(8)	–0.0005(7)	–0.0022(6)	0.0000(7)
C(5)	4*e*	0.7813(1)	0.1191(2)	0.3719(1)	0.0354(8)	0.0400(8)	0.0364(8)	–0.0004(8)	–0.0027(6)	0.0004(7)
C(6)	4*e*	0.6728(2)	0.0654(2)	0.3959(2)	0.0349(9)	0.053(1)	0.051(1)	–0.0009(9)	0.0009(8)	0.0055(9)
C(7)	4*e*	0.6704(2)	0.0348(2)	0.4917(2)	0.046(1)	0.061(1)	0.053(1)	–0.001(1)	0.0095(8)	0.012(1)
C(8)	4*e*	0.7760(2)	0.0645(2)	0.5402(1)	0.054(1)	0.049(1)	0.0435(9)	0.0019(9)	0.0051(7)	0.0024(9)
C(9)	4*e*	0.7209(2)	–0.1725(2)	0.2577(2)	0.071(2)	0.049(1)	0.117(2)	–0.012(1)	–0.035(2)	–0.005(2)
C(10)	4*e*	0.9027(2)	–0.1638(2)	0.4178(2)	0.066(2)	0.061(1)	0.071(1)	0.004(1)	–0.006(1)	0.022(1)
C(11)	4*e*	0.9589(3)	–0.1479(2)	0.1970(2)	0.096(2)	0.056(1)	0.081(2)	0.020(1)	0.023(1)	–0.012(1)
C(12)	4*e*	0.6920(2)	0.4058(3)	0.0909(3)	0.051(1)	0.117(2)	0.117(2)	0.011(2)	–0.002(1)	0.066(2)
C(13)	4*e*	0.8961(2)	0.5095(2)	0.1914(2)	0.099(2)	0.049(1)	0.075(2)	–0.014(1)	0.017(1)	0.000(1)
C(14)	4*e*	0.9200(2)	0.3399(2)	0.0191(2)	0.088(2)	0.080(2)	0.051(1)	0.003(2)	0.022(1)	0.006(1)

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