Crystal structure of 2′-methoxyphenyl-2-ethylsulfonyl acetate, $C_{11}H_{14}O_5S$

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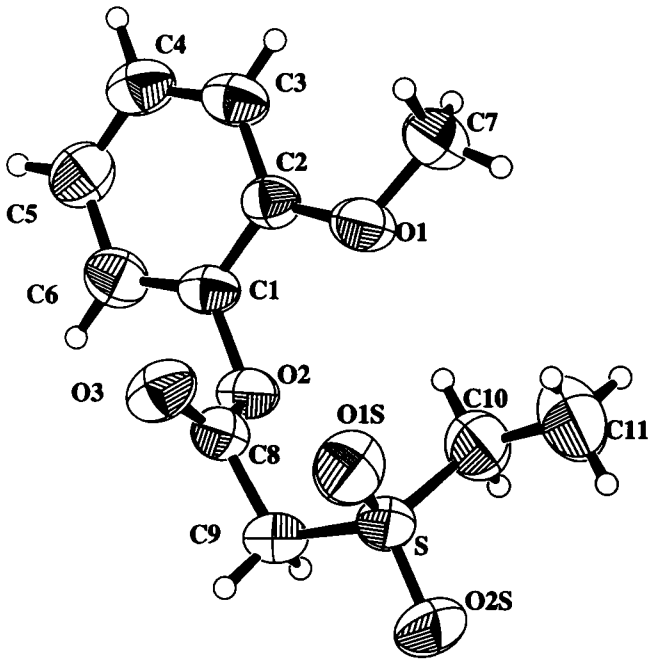

Abstract

$C_{11}H_{14}O_5S$, monoclinic, $P12_1/c1$ (No. 14), a = 8.3068(9) Å, b = 7.966(1) Å, c = 18.599(3) Å, β = 96.304(9)°, V = 1223.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.053, $wR_{all}(F^2)$ = 0.160, T = 291 K

Source of material

A mixture of 2-ethylthio-2′-methoxyacetophenone (0.45 g; 2.15 mmol) and 30% by weight of hydrogen peroxide (0.8 ml; 8.8 mmol) was kept under stirring in 10 ml of acetic acid solution at room temperature during 24 h. The reaction was carried until the substrate disappeared (TLC monitoring). The reaction mixture was diluted with cold water, extracted with chloroform and washed with 10% aqueous solution of sodium bicarbonate. The organic solution was dried over anhydrous magnesium sulphate. After solvent rotoevaporation the obtained oil was kept in a refrigerator. The amorphous solid was crystallized from carbon tetrachloride at 277 K giving 0.42 g of the title compound (1.63 mmol, 75.7% yield; mp 343-346 K). The 2-ethylthio-2′-methoxyacetophenone was obtained following essentially the same procedure described for the 2-ethylthio-4′-substituted acetophenones [1].

Discussion

The S atom is tetrahedrally bonded to two C and two O atoms, with angles: ∠O2S–S–C10 = 108.4(2)°, ∠O1S–S–C10 = 112.1(3)°, ∠O2S–S–C9 = 107.3(2)°, ∠O1S–S–C9 = 107.7(2)° and ∠C9–S–C10 = 102.8(2)°; except for the ∠O1S–S–O2S which is 117.5(2)°.

Table 1. Data collection and handling.

Crystal:	colourless, irregular, size 0.05 x 0.10 x 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.71 cm^{-1}
Diffractometer, scan mode:	CAD-4 Mach 3, ω/2θ
$2\theta_{max}$:	50.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2395, 2235
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1170
$N(param)_{refined}$:	156
Programs:	MolEn [2], SHELXS-86 [3], SHELXL-97 [4], ZORTEP [5]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.9861	0.3267	0.4045	0.072
H(4)	4e	0.7292	0.2190	0.3754	0.077
H(5)	4e	0.5995	0.0834	0.4611	0.081
H(6)	4e	0.7253	0.0551	0.5782	0.073
H(7A)	4e	1.2637	0.2925	0.4414	0.109
H(7B)	4e	1.3663	0.3968	0.5015	0.109
H(7C)	4e	1.2116	0.4760	0.4588	0.109
H(9A)	4e	1.1315	0.1357	0.7512	0.071
H(9B)	4e	1.0400	0.2804	0.7869	0.071
H(10A)	4e	1.4150	0.1756	0.6954	0.113
H(10B)	4e	1.3195	0.2936	0.6381	0.113
H(11A)	4e	1.5076	0.5000	0.6649	0.189
H(11B)	4e	1.5911	0.3367	0.6401	0.189
H(11C)	4e	1.6027	0.3828	0.7224	0.189

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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*	1.2791(1)	0.3781(2)	0.75291(5)	0.0576(6)	0.0803(9)	0.0438(6)	0.0045(7)	0.0053(5)	-0.0018(6)
O(1)	4*e*	1.1626(4)	0.3109(4)	0.5316(1)	0.072(2)	0.073(2)	0.046(2)	-0.008(2)	0.009(1)	0.005(2)
O(2)	4*e*	1.0115(3)	0.1528(3)	0.6299(1)	0.067(2)	0.053(2)	0.042(2)	0.009(2)	0.008(1)	0.004(1)
O(3)	4*e*	0.9100(4)	0.3972(4)	0.6642(2)	0.081(2)	0.063(2)	0.060(2)	0.019(2)	-0.005(2)	-0.007(2)
O(1S)	4*e*	1.2313(4)	0.5462(4)	0.7380(2)	0.078(2)	0.070(2)	0.102(3)	-0.011(2)	0.002(2)	0.011(2)
O(2S)	4*e*	1.3679(4)	0.3442(5)	0.8213(2)	0.080(2)	0.116(3)	0.049(2)	0.004(2)	-0.014(2)	-0.002(2)
C(1)	4*e*	0.9294(5)	0.1736(5)	0.5599(2)	0.066(3)	0.043(2)	0.037(2)	0.006(2)	0.008(2)	0.000(2)
C(2)	4*e*	1.0114(5)	0.2533(5)	0.5086(2)	0.058(2)	0.049(3)	0.042(2)	0.003(2)	0.007(2)	-0.005(2)
C(3)	4*e*	0.9343(6)	0.2714(5)	0.4395(2)	0.079(3)	0.060(3)	0.043(2)	0.006(3)	0.014(2)	-0.001(2)
C(4)	4*e*	0.7804(6)	0.2073(6)	0.4222(2)	0.078(3)	0.065(3)	0.047(2)	0.008(3)	-0.002(2)	-0.003(2)
C(5)	4*e*	0.7029(6)	0.1268(6)	0.4733(2)	0.067(3)	0.066(3)	0.066(3)	-0.005(3)	-0.004(2)	-0.006(3)
C(6)	4*e*	0.7778(6)	0.1096(6)	0.5432(2)	0.069(3)	0.055(3)	0.060(3)	-0.006(3)	0.012(2)	0.003(2)
C(7)	4*e*	1.2589(6)	0.3742(6)	0.4791(2)	0.076(3)	0.081(3)	0.064(3)	-0.002(3)	0.021(2)	0.009(3)
C(8)	4*e*	0.9954(5)	0.2792(6)	0.6770(2)	0.056(3)	0.053(3)	0.045(2)	-0.004(2)	0.008(2)	0.001(2)
C(9)	4*e*	1.1011(5)	0.2531(6)	0.7469(2)	0.069(3)	0.064(3)	0.043(2)	0.001(2)	0.004(2)	0.008(2)
C(10)	4*e*	1.3877(6)	0.2916(9)	0.6839(3)	0.065(3)	0.153(6)	0.064(3)	-0.019(4)	0.002(3)	-0.010(3)
C(11)	4*e*	1.5347(7)	0.386(1)	0.6772(3)	0.085(4)	0.192(8)	0.107(5)	-0.006(5)	0.033(4)	-0.002(5)

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