Crystal structure of 3-(bis(benzylthio)methylene)pentane-2,4-dione, $C_{20}H_{20}O_2S_2$

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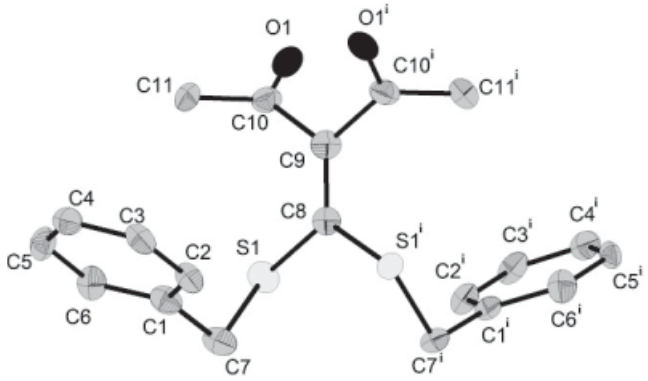

Abstract

$C_{20}H_{20}O_2S_2$, monoclinic, *C*2/*c* (no. 15), a = 11.478(8) Å, b = 11.757(8) Å, c = 14.39(1) Å, β = 101.08(2)°, V = 1905.8 Å^3, Z = 4, $R_{gt(F)}$ = 0.0448, $wR_{ref(F^2)}$ = 0.1433, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless parallelepipeds, size 0.15×0.18×0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.88 cm^{-1}
Diffractometer, scan mode:	Rigaku Mercury CCD area-detector, ω
$2\theta_{max}$:	55.22°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9240, 2214
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1935
$N(param)_{refined}$:	111
Programs:	RAPID AUTO [7], SHELX [8]

Source of material

K_2CO_3 (5.50 g, 0.04 mol) was dissolved in the mixed-solvent of vacetylacetone (2.1 ml) and dimethylformamide (30 ml). The mixture was stirred for 0.5 h, and then the the CS_2 was added dropwise within 10 min. and kept stirring. After reacting for 1 h, benzyl bromide (7.5 g, 0.044 mol) was added at once. The reaction was allowed to proceed for 12 h at room temperature, and then was poured into water and resulted in lots of white precipitate. The precipitate was filtered, washed with water and dried. Colourless crystals of the title compound, which were suitable for X-ray analysis, were obtained by recrystallizing the white deposit from an anhydrous ethanol solution at room temperature over a period of several days.

Experimental details

All non-H atoms were refined with anisotropic displacement parameters. All H atoms were generated geometrically and refined as riding atoms with d(C–H) = 0.93-0.98 Å, and U_{iso}(H) = 1.2U_{eq}(C) for C atoms of CH_2 and CH groups and U_{iso}(H) = 1.5U_{eq}(C) for C atoms of CH_3 groups.

Discussion

Thiols are versatile reagents and have been widely utilized in organic synthesis [1, 2]. However, most of thiols, such as methanethiol, ethanethiol and benzyl mercaptan, having toxicity and a foul smell, can lead to environmental and safety problems. To develop green chemistry, some new reagents substituting for thiols should be developed in organic synthesis [3-6]. Herein, a new substitute material for thiols was prepared and characterized by single-crystal X-ray diffraction. Within the crystal structure, the geometric parameters are in the usual ranges. The distance of C8 and C9 is 1.331(3) Å, revealing the typical C=C double bond. The S1–C7 and S1–C8 bond lengths are 1.824(2) and 1.759(1) Å, respectively. The torsion angles of C1–C7–S1–C8 and S1–C8–Sl(i)–C7(i) are 70.25(12)° and 49.36(9)°. The C10–O1 distance is 1.188(3) Å, showing the bond between the two atoms is a double bond. The molecules are held together by the van der Waals forces to form the title structure.

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

Atom	Site	x	y	z	U_{iso}
H(2A)	8*f*	0.6443	0.1384	0.4500	0.072
H(3A)	8*f*	0.7055	0.2242	0.5941	0.096
H(4A)	8*f*	0.5692	0.2763	0.6840	0.101
H(5A)	8*f*	0.3707	0.2354	0.6311	0.105
H(6A)	8*f*	0.3095	0.1472	0.4874	0.083
H(7A)	8*f*	0.3693	0.0249	0.3621	0.062
H(7B)	8*f*	0.4969	0.0404	0.3401	0.062
H(11A)	8*f*	0.3254	0.4937	0.3841	0.176
H(11B)	8*f*	0.4622	0.4726	0.4155	0.176
H(11C)	8*f*	0.3747	0.3691	0.3963	0.176

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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(1)	8*f*	0.37242(3)	0.18325(3)	0.26539(3)	0.0362(3)	0.0522(3)	0.0463(3)	–0.0038(1)	0.0008(2)	0.0047(1)
O(1)	8*f*	0.3497(2)	0.5061(2)	0.2182(1)	0.091(1)	0.116(1)	0.113(1)	0.057(1)	0.006(1)	0.006(1)
C(1)	8*f*	0.4703(1)	0.1350(1)	0.4523(1)	0.0532(9)	0.0434(8)	0.0457(7)	–0.0035(6)	0.0035(6)	0.0096(6)
C(2)	8*f*	0.5885(2)	0.1581(2)	0.4862(1)	0.058(1)	0.071(1)	0.0494(9)	–0.0170(8)	0.0045(7)	0.0086(7)
C(3)	8*f*	0.6252(2)	0.2097(2)	0.5724(1)	0.087(2)	0.092(2)	0.054(1)	–0.039(1)	–0.005(1)	0.008(1)
C(4)	8*f*	0.5445(3)	0.2399(2)	0.6262(1)	0.124(2)	0.075(1)	0.049(1)	–0.016(1)	0.003(1)	–0.0032(9)
C(5)	8*f*	0.4261(3)	0.2160(2)	0.5943(2)	0.109(2)	0.101(2)	0.056(1)	0.026(2)	0.022(1)	0.003(1)
C(6)	8*f*	0.3896(2)	0.1636(2)	0.5082(1)	0.062(1)	0.086(1)	0.059(1)	0.009(1)	0.0085(9)	0.0073(9)
C(7)	8*f*	0.4304(2)	0.0804(1)	0.3576(1)	0.0554(9)	0.0439(8)	0.0518(8)	–0.0080(7)	0.0024(7)	0.0063(6)
C(8)	4*e*	$\frac{1}{2}$	0.2583(2)	$\frac{1}{4}$	0.0358(9)	0.043(1)	0.0369(9)	0	0.0013(7)	0
C(9)	4*e*	$\frac{1}{2}$	0.3715(2)	$\frac{1}{4}$	0.041(1)	0.044(1)	0.048(1)	0	0.0056(8)	0
C(10)	8*f*	0.4027(2)	0.4439(2)	0.2764(2)	0.055(1)	0.0478(9)	0.082(1)	0.0037(8)	0.0175(8)	–0.0047(8)
C(11)	8*f*	0.3902(3)	0.4449(2)	0.3769(2)	0.185(3)	0.071(1)	0.124(2)	0.028(2)	0.099(2)	0.002(2)

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