Crystal structure of *trans*-2,4-bis(*sec*-butyl)-2,4-bis((2-methyl-1-thioxo)butylsulfanyl)-1,3-dithietane, $C_2S_2(C_4H_9)_2(C_4H_9CS_2)_2$

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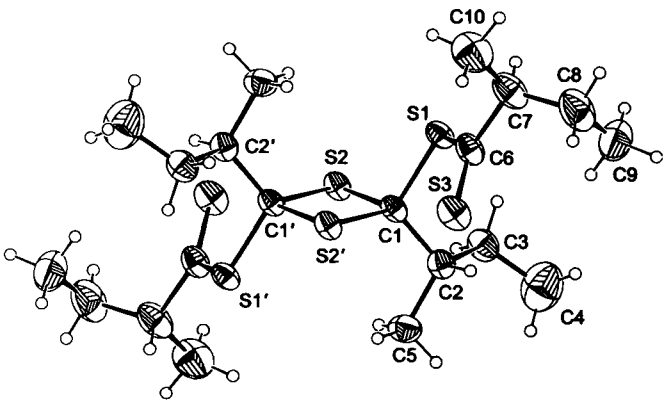
Received August 15, 2005, accepted and available on-line September 5, 2005; CCDC no. 1267/1628



Abstract

$C_{20}H_{36}S_6$, monoclinic, $P12_1/n1$ (no. 14), a = 12.008(3) Å, b = 6.050(2) Å, c = 17.858(5) Å, β = 99.34(2)°, V = 1280.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.050, $wR_{ref}(F)$ = 0.080, T = 293 K.

Source of material

2-methylbutanedithioic acid was prepared according to a previously described procedure [1,2]. Reaction of the dithioacid with $BF_3 \cdot Me_2O$, as it is the case for branched dithioacids [3] caused the evolution of H_2S and produced the dithietane species. So 2.34 g (0.05 mol) of $CH_3CH_2CH(CH_3)CS_2H$ was dissolved in 20 mL hexane. Boron trifluoride dimethyletherate (3.42 g, 0.03 mol) was then added. The mixture was cooled to 253 K. After two weeks, crystals of crude dithietane were collected by filtration. Good quality crystals for X-ray diffraction analysis were obtained by slow evaporation from an acetone solution.

Discussion

The tetrahedral unit C1-S2-C2 located close to the centre of symmetry and shares an edge (S2-S2′) with its centrosymmetrical one. At the centre of the molecule, this condensation creates an almost regular square plane built by S2 and C1 and their centrosymmetrical ones with the S2–C1–S2′ angle being 94.57(3)°. The tetrahedral unit C1-S2-C2 is mainly characterized by three very similar C—S distances ranging from 1.832(2) Å to 1.842(2) Å and one C1—C2 distance of 1.539(3) Å. In spite of this apparent distortion the average angle C2–C1–S in this tetrahedron, 114.3°, is not far from the theoretical value for a regular tetrahedron in the other organic entities of the arrangement. The observed interatomic distances and bond angles are in accordance with all values previously reported [4].

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.40 x 0.45 x 0.44 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	5.36 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	59.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4172, 4061
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2503
$N(param)_{refined}$:	118
Programs:	SIR92 [5], teXsan [6], ORTEP-3 [7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	1.2257	−0.0010	0.9336	0.066
H(2)	4e	1.1951	0.3892	0.9384	0.083
H(3)	4e	1.0983	0.3568	0.8703	0.083
H(4)	4e	1.3258	0.3138	0.8691	0.175
H(5)	4e	1.2410	0.4595	0.8158	0.175
H(6)	4e	1.2398	0.2048	0.8057	0.175
H(7)	4e	1.1301	−0.0819	0.8152	0.084
H(8)	4e	1.0173	−0.0027	0.8378	0.084
H(9)	4e	1.0747	−0.2165	0.8733	0.084
H(10)	4e	1.3506	0.2761	1.1726	0.103
H(11)	4e	1.5378	0.1776	1.1996	0.140
H(12)	4e	1.5114	−0.0319	1.1499	0.140
H(13)	4e	1.5796	0.2293	1.0762	0.141
H(14)	4e	1.4930	0.4019	1.0956	0.141
H(15)	4e	1.4522	0.2005	1.0447	0.141
H(16)	4e	1.4112	0.0705	1.2864	0.133
H(17)	4e	1.3700	−0.1441	1.2430	0.133
H(18)	4e	1.2836	0.0356	1.2572	0.133

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)	4e	1.18385(4)	0.20592(8)	1.07038(3)	0.0508(3)	0.0378(2)	0.0658(3)	0.0006(2)	−0.0043(2)	−0.0061(2)
S(2)	4e	0.95730(4)	0.20034(8)	0.97752(3)	0.0472(2)	0.0305(2)	0.0666(3)	0.0032(2)	−0.0002(2)	0.0038(2)
S(3)	4e	1.30151(6)	−0.2281(1)	1.07217(5)	0.0655(3)	0.0462(3)	0.0998(5)	0.0121(3)	−0.0051(3)	0.0005(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	1.0961(2)	0.0631(3)	0.9916(1)	0.0455(8)	0.0312(8)	0.060(1)	0.0016(7)	–0.0018(7)	–0.0006(8)
C(2)	4*e*	1.1531(2)	0.0636(4)	0.9203(1)	0.054(1)	0.042(1)	0.067(1)	0.0020(9)	0.0067(9)	0.0025(9)
C(3)	4*e*	1.1692(3)	0.3021(4)	0.8947(2)	0.080(2)	0.048(1)	0.077(2)	–0.008(1)	0.009(1)	0.006(1)
C(4)	4*e*	1.2513(5)	0.3218(9)	0.8416(4)	0.155(4)	0.122(4)	0.169(4)	–0.001(3)	0.056(3)	0.032(3)
C(5)	4*e*	1.0876(2)	–0.0723(4)	0.8555(1)	0.089(2)	0.058(1)	0.066(1)	–0.004(1)	0.015(1)	–0.009(1)
C(6)	4*e*	1.2911(2)	0.0226(4)	1.1029(1)	0.050(1)	0.050(1)	0.076(1)	–0.0011(9)	–0.006(1)	0.003(1)
C(7)	4*e*	1.3709(2)	0.1251(6)	1.1692(2)	0.066(1)	0.080(2)	0.099(2)	0.001(1)	–0.025(1)	–0.006(2)
C(8)	4*e*	1.4906(3)	0.1173(8)	1.1566(3)	0.062(2)	0.107(3)	0.171(3)	0.005(2)	–0.011(2)	–0.027(3)
C(9)	4*e*	1.5052(4)	0.2496(9)	1.0868(3)	0.093(2)	0.126(3)	0.145(3)	–0.009(2)	0.050(2)	–0.012(3)
C(10)	4*e*	1.3576(3)	0.0103(8)	1.2467(2)	0.124(3)	0.119(3)	0.083(2)	–0.014(2)	–0.008(2)	0.017(2)

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