Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A dithietane of 2-methylpropanedithioic acid

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Under the action of $BF_3{\cdot}Me_2O$, 2-methylpropanedithioic acid does not give the expected adamantanoid product but the *trans*-2,4-diisopropyl-2,4-bis[(2-methyl-1-thioxo)propylsulfanyl]-1,3-dithietane. The X-ray structure of the centrosymmetric title compound confirms the *trans* conformation of the dithietane molecule.

Received 8 December 2002
Accepted 7 February 2003
Online 31 March 2003

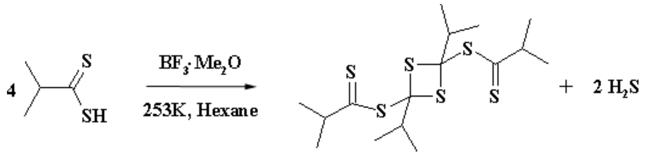
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.061
Data-to-parameter ratio = 20.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We recently described the action of $BF_3{\cdot}Me_2O$ on propanedithioic acid (Mahjoub & Zantour, 2000) and butanedithioic acid (Mahjoub & Zantour, 2001). Another linear aliphatic dithioacid (ethanedithioic acid) was also treated in the same manner. In all cases we obtained a tetraalkyl hexathia-adamantanoid compound.



(I)

The aim of the present work is to investigate the action of this Lewis acid $BF_3{\cdot}Me_2O$, using the same conditions, on a branched aliphatic dithioacid (2-methylpropanedithioic acid). This reaction occurs with a large evolution of H_2S and the formation of the dithietane, (I), as shown in the reaction scheme. A similar compound was obtained by reaction of the same dithioacid with dicyclohexylcarbodiimide (Kato *et al.*, 1982). The crystal structure determination was undertaken to establish the conformation of the reaction product, (I), and to understand the process of its formation.

The atomic arrangement of the title compound (Fig. 1) is centrosymmetric about the mid-point of the S3—S3^{i} vector, creating a planar, almost square central ring [(i): $1 - x$, $-y$, $1 - z$]. The angle S3—C5—S3^{i} is 95.54 (6)°. The tetrahedral unit C5,S2,S3,C6,S3^{i} is mainly characterized by three very similar C—S distances ranging from 1.827 to 1.839 Å and one C5—C6 distance of 1.539 Å. In spite of this apparent distortion, the average angle C6—C5—S in this tetrahedron, 108.7°, is close to the theoretical value for a regular tetrahedron. The other interatomic distances and bond angles are in accordance with expected values (Allen *et al.*, 1987).

Experimental

2-Methylpropanedithioic acid (1.8 g, 0.0015 mol), prepared according to a method described by Beiner & Thuillier (1972), was dissolved in

10 ml of hexane. Boron trifluoride dimethyletherate (3.42 g, 0.03 mol) was then added. The mixture was cooled down to 253 K. After two weeks, crystals of the crude dithietane were collected by filtration. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from an acetone solution.

Crystal data

$C_{16}H_{28}S_6$
$M_r = 412.76$
Monoclinic, $P2_1/c$
$a = 8.923$ (2) Å
$b = 6.082$ (2) Å
$c = 19.828$ (5) Å
$\beta = 93.29$ (2)°
$V = 1074.3$ (4) Å^3
$Z = 2$
$D_x = 1.276$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 25 reflections
$\theta = 10.0$–15.5°
$\mu = 0.63$ mm^{-1}
$T = 293$ K
Square prism, yellow
0.22 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: none
3473 measured reflections
3084 independent reflections
2261 reflections with $I > 2.5\sigma(I)$
$R_{\rm int} = 0.014$
$\theta_{\rm max} = 30.0°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 27$
2 standard reflections every 120 minutes intensity decay: 0.1%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.061$
$S = 1.49$
2261 reflections
109 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00036|F_o|^2]$
$(\Delta/\sigma)_{\rm max} = 0.006$
$\Delta\rho_{\rm max} = 0.39$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C4	1.630 (2)	C1—C3	1.483 (5)
S2—C4	1.721 (2)	C1—C4	1.529 (3)
S2—C5	1.827 (2)	C2—C2′	0.92 (1)
S3—C5	1.827 (2)	C5—C6	1.541 (2)
S3—C5$^{\rm i}$	1.839 (2)	C6—C7	1.518 (3)
C1—C2	1.460 (8)	C6—C8	1.516 (3)
C1—C2′	1.68 (1)		
C4—S2—C5	105.24 (9)	S1—C4—C1	125.4 (2)
C5—S3—C5$^{\rm i}$	85.40 (8)	S2—C4—C1	109.1 (2)
C2—C1—C2′	33.2 (4)	S2—C5—S3	111.51 (9)
C2—C1—C3	124.2 (4)	S2—C5—S3$^{\rm i}$	106.14 (8)
C2—C1—C4	113.4 (4)	S2—C5—C6	110.8 (1)
C2′—C1—C3	102.4 (4)	S3—C5—S3$^{\rm i}$	94.60 (8)
C2′—C1—C4	105.3 (4)	S3—C5—C6	118.2 (1)
C3—C1—C4	109.9 (3)	S3$^{\rm i}$—C5—C6	114.1 (1)
C1—C2—C2′	86.6 (9)	C5—C6—C7	113.0 (2)
C1—C2′—C2	60.3 (9)	C5—C6—C8	110.5 (2)
S1—C4—S2	125.5 (1)	C7—C6—C8	109.8 (2)

Symmetry code: (i) $1 - x, -y, 1 - z$.

The H atoms were located in a difference Fourier map and introduced in calculated positions. Owing to the large displacement ellipsoid components, atom C2 was considered disordered and split into two positions, C2 and C2′. The refinement of the occupancy factors led to almost equipartition, with occupancy factors of 0.55 and 0.45 for C2 and C2′, respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *teXsan* (MSC, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *teXsan* (Molecular Structure Corporation, 1995); software used to prepare material for publication: *ORTEP*III (Burnett & Johnson, 1996).

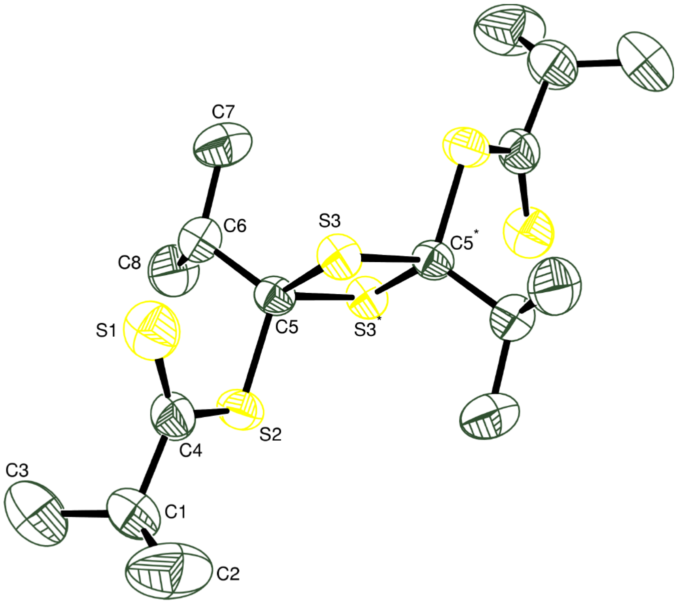


Figure 1
*ORTEP*III drawing (Burnett & Johnson, 1996), showing the molecular structure of the title compound and the numbering scheme (35% probability displacement ellipsoids).

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