Supporting Information

1-Adamantyl-*t*-butyltetrathiolane 2,3-Dioxide: The First Isolable *vic*-Disulfoxide and an Efficient Precursor of S_2O

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Characterization data (^1H and ^{13}C NMR, IR, MS, and elemental analyses) for **3**, **4**, and **5**; structure determination summaries and tables of X-ray structure data for **3**.

1-Adamantyl-*t*-butyltetrathiolane 2,3-dioxide (**3**)

Pale-yellow, fine plates, m.p. 65—66 °C decomp (CH_2Cl_2-EtOH). ^1H NMR (400 MHz, $CDCl_3$, 263 K) δ 1.45 (br s, 9H, *t*-Bu), 1.65 (br s, 6H), 2.09 (br s, 9H); ^{13}C NMR (100.6 MHz, $CDCl_3$, 263 K) δ 29.2 (CH), 33.4 (br s, CH_3), 35.9 (CH_2), 42.2 (CH_2), 44.5 (C), 46.9 (C), 153.9 (C); IR (KBr, 298 K) 1134, 1104 cm^{-1}. Anal. Calcd for $C_{15}H_{24}O_2S_4$: C, 49.42; H, 6.63. Found: C, 48.84; H, 6.57.

Structure determination summaries and tables of X-ray structure data for **3**

For yellow prisms:

$C_{15}H_{24}O_2S_4$, M_w 364.59. Monoclinic, space group $P2_1/n$, a = 6.4150(3), b = 18.878(1), c = 13.7410(9) Å, β = 97.349(4), V = 1650.4(2) $Å^3$, Z = 4, ρ_{calcd} = 1.467 g cm^{-3}, $\mu(Mo_{K\alpha})$ = 5.54 mm^{-1}. A yellow prism with dimensions 0.30 × 0.12 × 0.10 mm was mounted on a Mac Science DIP3000 diffractometer with a graphite-monochromater. Oscillation and nonscreen Weissenberg photographs were recorded on the imaging plates of the diffractometer by using $Mo_{K\alpha}$ radiation (λ = 0.71073 Å) at 153 K and the data reduction was made by the MAC DENZO program system. Intensity data of 4111

independent reflections were collected in the range of $0 \le h \le 7$, $0 \le k \le 26$, $-19 \le l \le 19$. Cell parameters were determined and refined by using the MAC DENZO for all observed reflections. The structure was solved by direct methods using SIR[1] in the CRYSTAN-GM program system. The atomic coordinates and anisotropic thermal parameters of the non-H atoms were refined by full-matrix least squares[2] to minimize the functions, $\Sigma(|F_o|-|F_c|)^2$, for 3683 reflections [$I \ge 2\sigma(I)$] (286 parameters). The final R (R_w) = 0.077 (0.086) and GOF = 3.761; max/min residual electron density = 2.06/–1.97 e Å^{-3}.

Crystal and molecular structure of 1–Adamantyl-t-butyltetrathiolane 2,3–Dioxide

Abstract

We present the crystal and molecular structure of 1–Adamantyl-t-butyltetrathiolane 2,3–Dioxide.

Comment

The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All Diagrams and calculations were performed using *CRYSTAN* (MacScience, Japan).

Experimental

Crystal data

$C_{15}H_{24}O_2S_4$
$M_r = 364.59$
Monoclinic
$P2_1/n$
$a = 6.4150\ (3)$ Å
$b = 18.878\ (1)$ Å
$c = 13.7410\ (9)$ Å
$\beta = 97.349\ (4)°$
$V = 1650.4\ (2)$ Å^3
$Z = 4$
$D_x = 1.467$ Mg m^{-3}
Mo K-alpha radiation
$\lambda = 0.71073$ Å
$\mu = 5.54$ mm^{-1}
$T = 153$ K
Yellow prism
$0.30 \times 0.12 \times 0.10$ mm
Crystal source: Local laboratory

Data collection

Mac Science DIP3000 diffractometer

theta/2theta scans

Absorption correction:

none

4313 measured reflections

4111 independent reflections

3683 observed reflections

$[I > 2.00 \text{ sigma}(I)]$

$\theta_{max} = 28.91°$

$h = 0 \rightarrow 7$

$k = 0 \rightarrow 26$

$l = -19 \rightarrow 19$

Refinement

Refinement on F

$R = 0.077$

$wR = 0.086$

$S = 3.761$

3683 reflections

286 parameters

Only coordinates of H atoms refined

Count statistics

$(\Delta/\sigma)_{max} = 0.0286$

$\Delta\rho_{max} = 2.06$ e Å^{-3}

$\Delta\rho_{min} = -1.97$ e Å^{-3}

Extinction correction: None

Atomic scattering factors from *International Tables* (1974)

Data collection: MXC(MAC Science). Cell refinement: MXC(MAC Science). Data reduction: *CRYSTAN*. Program(s) used to solve structure: *CRYSTAN*. Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

FRACTIONAL ATOMIC COORDINATES & U(iso)

Atom	x/a	y/b	z/c	U(iso)
S(1)	-0.07900(7)	0.14729(2)	0.91299(3)	0.0265(2)
S(2)	-0.04618(9)	0.13545(3)	1.06279(4)	0.0379(2)
S(3)	0.27814(10)	0.08210(3)	1.07151(4)	0.0476(3)
S(4)	0.38697(7)	0.15473(2)	0.97902(3)	0.0255(2)
O(1)	-0.0026(3)	0.2001(1)	1.1202(1)	0.0507(9)
O(2)	0.2531(3)	0.0111(1)	1.0403(1)	0.060(1)
C(1)	0.1794(3)	0.1732(1)	0.8746(1)	0.0201(6)
C(2)	0.2037(3)	0.1217(1)	0.7836(1)	0.0191(6)
C(3)	0.2381(3)	0.0446(1)	0.8196(1)	0.0219(7)
C(4)	0.2647(3)	-0.0067(1)	0.7352(1)	0.0299(8)
C(5)	0.0687(3)	-0.0053(1)	0.6595(2)	0.0331(9)
C(6)	0.0346(3)	0.0700(1)	0.6212(1)	0.0297(8)
C(7)	0.0055(3)	0.1209(1)	0.7068(1)	0.0240(7)
C(8)	0.4580(3)	0.0158(1)	0.6877(2)	0.0305(8)
C(9)	0.4207(3)	0.0912(1)	0.6474(1)	0.0278(8)
C(10)	0.3938(3)	0.1428(1)	0.7318(1)	0.0230(7)
C(11)	0.2226(3)	0.0934(1)	0.5720(1)	0.0332(9)
C(12)	0.1912(3)	0.2572(1)	0.8612(1)	0.0256(7)
C(13)	0.0502(4)	0.2827(1)	0.7676(2)	0.0358(9)
C(14)	0.4168(3)	0.2842(1)	0.8579(2)	0.0336(8)
C(15)	0.1105(4)	0.2952(1)	0.9482(2)	0.0387(9)
H(3A)	0.342(3)	0.046(1)	0.868(2)	0.020(5)
H(3B)	0.138(3)	0.028(1)	0.856(1)	0.016(4)
H(4)	0.293(4)	-0.049(1)	0.756(2)	0.051(7)
H(5A)	0.083(4)	-0.037(1)	0.605(2)	0.033(6)
H(5B)	-0.055(4)	-0.017(1)	0.681(2)	0.032(6)
H(6)	-0.089(4)	0.073(1)	0.574(2)	0.033(6)
H(7A)	-0.112(3)	0.111(1)	0.737(2)	0.023(5)
H(7B)	-0.030(4)	0.164(1)	0.681(2)	0.038(6)
H(8A)	0.464(4)	-0.012(2)	0.630(2)	0.059(8)
H(8B)	0.581(4)	0.014(1)	0.732(2)	0.049(7)
H(9)	0.518(4)	0.103(1)	0.622(2)	0.023(5)
H(10A)	0.374(3)	0.187(1)	0.702(2)	0.025(5)
H(10B)	0.520(3)	0.142(1)	0.779(1)	0.015(4)
H(11A)	0.214(3)	0.134(1)	0.550(1)	0.016(5)
H(11B)	0.234(4)	0.064(1)	0.522(2)	0.030(5)
H(13A)	-0.102(5)	0.262(1)	0.756(2)	0.054(8)
H(13B)	0.066(5)	0.325(2)	0.775(2)	0.062(9)
H(13C)	0.105(4)	0.270(1)	0.708(2)	0.029(5)
H(14A)	0.410(4)	0.329(1)	0.849(2)	0.041(6)
H(14B)	0.491(4)	0.265(1)	0.806(2)	0.050(7)
H(14C)	0.506(4)	0.267(1)	0.921(2)	0.046(7)
H(15A)	-0.042(5)	0.287(1)	0.952(2)	0.052(8)
H(15B)	0.129(5)	0.343(2)	0.940(2)	0.065(9)
H(15C)	0.207(4)	0.281(1)	1.016(2)	0.035(6)

$$T = exp[2\pi^2 U]; \; U = U_{iso} \; or \; U_{eqv}; \; U_{eqv} = \frac{1}{3}\sum_{i=1}^{3}\sum_{j=1}^{3} U_{ij} a_i a_j \mathbf{a}_i . \mathbf{a}_j$$

ANISOTROPIC THERMAL PARAMETERS

Atom	U11	U22	U33	U12	U13	U23
S(1)	0.0250(3)	0.0301(2)	0.0249(2)	-.0020(2)	0.0096(2)	-.0040(2)
S(2)	0.0467(4)	0.0404(3)	0.0276(2)	0.0039(2)	0.0189(2)	0.0029(2)
S(3)	0.0572(4)	0.0455(3)	0.0411(3)	0.0154(3)	0.0195(3)	0.0207(2)
S(4)	0.0265(3)	0.0283(2)	0.0207(2)	-.0009(2)	-.0007(2)	-.0010(1)
O(1)	0.068(1)	0.054(1)	0.030(1)	0.010(1)	0.016(1)	-0.009(1)
O(2)	0.073(1)	0.053(1)	0.053(1)	0.001(1)	0.004(1)	0.016(1)
C(1)	0.0223(9)	0.0172(7)	0.0208(7)	-.0006(5)	0.0051(6)	0.0007(5)
C(2)	0.021(1)	0.018(1)	0.019(1)	0.000(1)	0.005(1)	-0.002(1)
C(3)	0.023(1)	0.019(1)	0.023(1)	-0.001(1)	0.003(1)	-0.002(1)
C(4)	0.034(1)	0.020(1)	0.036(1)	0.001(1)	0.012(1)	-0.007(1)
C(5)	0.030(1)	0.031(1)	0.038(1)	-0.006(1)	0.009(1)	-0.016(1)
C(6)	0.027(1)	0.039(1)	0.023(1)	-0.002(1)	0.001(1)	-0.008(1)
C(7)	0.019(1)	0.028(1)	0.024(1)	0.000(1)	0.003(1)	-0.003(1)
C(8)	0.023(1)	0.035(1)	0.033(1)	0.003(1)	0.007(1)	-0.011(1)
C(9)	0.024(1)	0.037(1)	0.023(1)	-0.004(1)	0.010(1)	-0.005(1)
C(10)	0.018(1)	0.027(1)	0.024(1)	-0.002(1)	0.004(1)	0.000(1)
C(11)	0.034(1)	0.044(1)	0.021(1)	-0.002(1)	0.003(1)	-0.003(1)
C(12)	0.030(1)	0.019(1)	0.027(1)	0.001(1)	0.003(1)	-0.001(1)
C(13)	0.042(1)	0.020(1)	0.043(1)	0.005(1)	0.001(1)	0.007(1)
C(14)	0.043(1)	0.021(1)	0.036(1)	-0.010(1)	0.008(1)	-0.003(1)
C(15)	0.049(1)	0.023(1)	0.044(1)	0.003(1)	0.014(1)	-0.010(1)

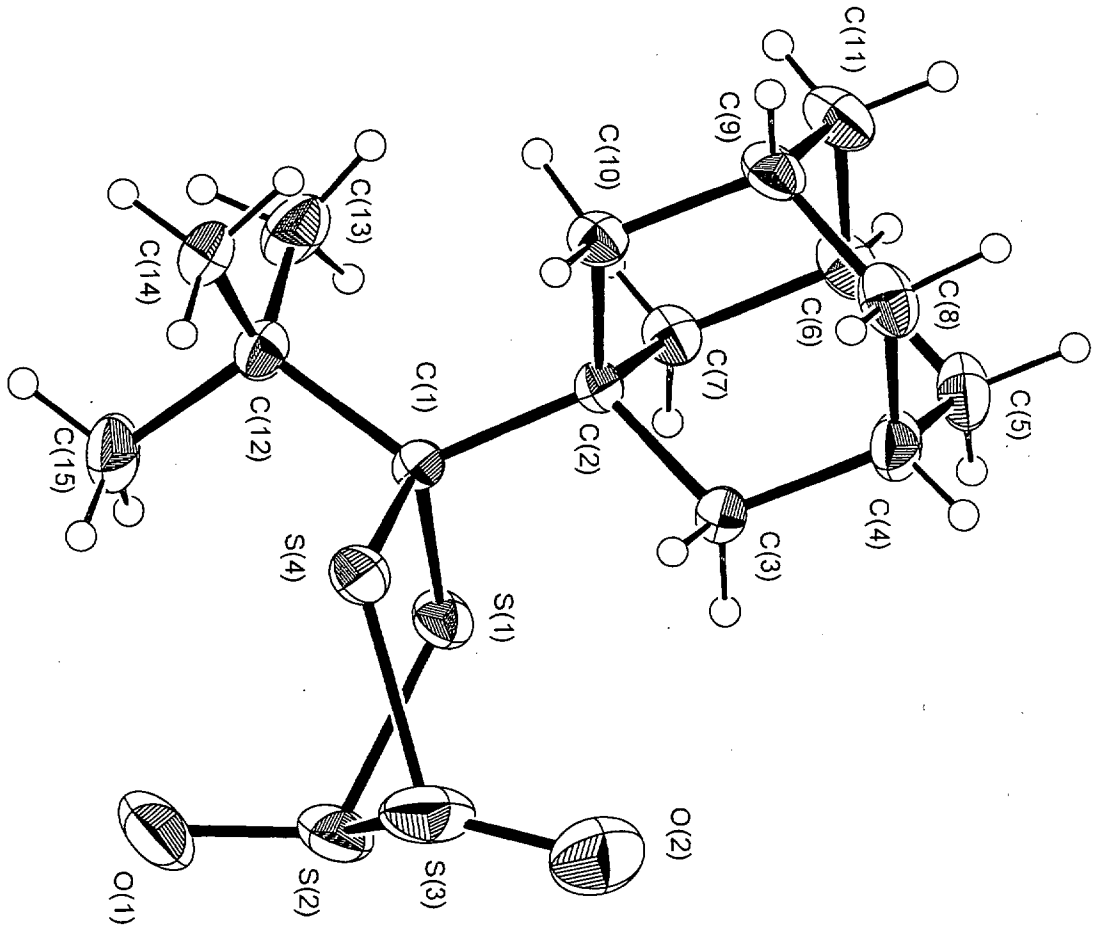
$$T=\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{23}klb^*c^*+2U_{13}hla^*c^*)]$$

INTRAMOLECULAR BOND LENGTHS

Minimum bond length= 0.80A		Maximum bond length= 2.31A	
S(1)-S(2)	2.054(1)	S(1)-C(1)	1.868(2)
S(2)-S(3)	2.301(1)	S(2)-O(1)	1.461(2)
S(3)-S(4)	2.052(1)	S(3)-O(2)	1.409(2)
S(4)-C(1)	1.861(2)	C(1)-C(2)	1.606(3)
C(1)-C(12)	1.599(3)	C(2)-C(3)	1.545(3)
C(2)-C(7)	1.545(3)	C(2)-C(10)	1.540(3)
C(3)-C(4)	1.537(3)	C(4)-C(5)	1.526(3)
C(4)-C(8)	1.533(3)	C(5)-C(6)	1.523(3)
C(6)-C(7)	1.549(3)	C(6)-C(11)	1.522(3)
C(8)-C(9)	1.534(3)	C(9)-C(10)	1.541(3)
C(9)-C(11)	1.534(3)	C(12)-C(13)	1.551(3)
C(12)-C(14)	1.540(3)	C(12)-C(15)	1.539(3)
C(2)-H(3A)	1.98(3)	C(2)-H(3B)	2.098(19)
C(2)-H(7A)	2.05(3)	C(2)-H(7B)	2.07(3)
C(2)-H(10A)	2.07(3)	C(2)-H(10B)	2.07(3)
C(3)-H(3A)	0.88(3)	C(3)-H(3B)	0.92(2)
C(3)-H(4)	2.03(3)	C(4)-H(3A)	2.08(3)
C(4)-H(3B)	2.05(2)	C(4)-H(4)	0.86(3)
C(4)-H(5A)	2.09(3)	C(4)-H(5B)	2.10(3)
C(4)-H(8A)	2.05(3)	C(4)-H(8B)	2.07(3)
C(5)-H(4)	2.00(3)	C(5)-H(5A)	0.97(3)
C(5)-H(5B)	0.91(3)	C(5)-H(6)	2.07(3)
C(6)-H(5A)	2.06(3)	C(6)-H(5B)	1.96(3)
C(6)-H(6)	0.96(3)	C(6)-H(7A)	2.10(3)
C(6)-H(7B)	2.02(3)	C(6)-H(11A)	2.00(3)
C(6)-H(11B)	1.99(3)	C(7)-H(6)	2.06(3)
C(7)-H(7A)	0.93(3)	C(7)-H(7B)	0.90(3)
C(8)-H(4)	1.94(3)	C(8)-H(8A)	0.96(3)
C(8)-H(8B)	0.93(3)	C(8)-H(9)	1.94(3)
C(9)-H(8A)	1.99(3)	C(9)-H(8B)	2.05(3)
C(9)-H(10A)	1.99(3)	C(9)-H(10B)	2.07(2)
C(9)-H(11A)	1.93(2)	C(9)-H(11B)	2.04(3)
C(10)-H(9)	1.95(3)	C(10)-H(10A)	0.93(3)
C(10)-H(10B)	0.98(3)	C(11)-H(6)	2.04(3)
C(11)-H(9)	1.94(3)	C(11)-H(11A)	0.82(3)
C(11)-H(11B)	0.90(3)	C(12)-H(13A)	2.23(3)
C(12)-H(13B)	1.86(4)	C(12)-H(13C)	2.12(3)
C(12)-H(14A)	1.98(3)	C(12)-H(14B)	2.16(3)
C(12)-H(14C)	2.09(3)	C(12)-H(15A)	2.15(3)
C(12)-H(15B)	2.02(4)	C(12)-H(15C)	2.16(3)
C(13)-H(13A)	1.04(3)	C(13)-H(13B)	0.82(4)
C(13)-H(13C)	0.96(3)	C(14)-H(14A)	0.86(3)
C(14)-H(14B)	0.98(3)	C(14)-H(14C)	1.02(3)
C(15)-H(15A)	1.00(3)	C(15)-H(15B)	0.92(4)
C(15)-H(15C)	1.08(3)		

INTRAMOLECULAR BOND ANGLES (H omitted)

Minimum bond length= 0.80A	: Maximum bond length= 2.31A		
S(2)-S(1)-C(1)	109.3(1)	S(1)-S(2)-S(3)	94.4(1)
S(1)-S(2)-O(1)	116.0(1)	S(3)-S(2)-O(1)	103.2(1)
S(2)-S(3)-S(4)	93.0(1)	S(2)-S(3)-O(2)	109.5(1)
S(4)-S(3)-O(2)	118.8(1)	S(3)-S(4)-C(1)	109.6(1)
S(1)-C(1)-S(4)	107.8(1)	S(1)-C(1)-C(2)	103.9(2)
S(1)-C(1)-C(12)	110.4(2)	S(4)-C(1)-C(2)	110.8(2)
S(4)-C(1)-C(12)	103.6(2)	C(2)-C(1)-C(12)	120.0(2)
C(1)-C(2)-C(3)	110.2(2)	C(1)-C(2)-C(7)	112.6(2)
C(1)-C(2)-C(10)	111.4(2)	C(3)-C(2)-C(7)	106.3(2)
C(3)-C(2)-C(10)	107.6(2)	C(7)-C(2)-C(10)	108.6(2)
C(2)-C(3)-C(4)	112.1(2)	C(3)-C(4)-C(5)	110.0(2)
C(3)-C(4)-C(8)	108.7(2)	C(5)-C(4)-C(8)	110.2(2)
C(4)-C(5)-C(6)	108.8(2)	C(5)-C(6)-C(7)	110.0(2)
C(5)-C(6)-C(11)	109.7(2)	C(7)-C(6)-C(11)	109.7(2)
C(2)-C(7)-C(6)	110.9(2)	C(4)-C(8)-C(9)	108.3(2)
C(8)-C(9)-C(10)	110.0(2)	C(8)-C(9)-C(11)	110.3(2)
C(10)-C(9)-C(11)	109.2(2)	C(2)-C(10)-C(9)	110.8(2)
C(6)-C(11)-C(9)	109.1(2)	C(1)-C(12)-C(13)	111.8(2)
C(1)-C(12)-C(14)	112.9(2)	C(1)-C(12)-C(15)	110.4(2)
C(13)-C(12)-C(14)	108.9(2)	C(13)-C(12)-C(15)	106.3(2)
C(14)-C(12)-C(15)	106.2(2)		



For pale-yellow plates:

orthorhombic, space group *P2nn*, a = 6.5200(6), b = 13.970(1), c = 18.386(2) Å, V = 1665.6(3) Å^3, Z = 4, ρ_{calcd} = 1.454 g cm^{-3}, $\mu(Mo_{K\alpha})$ = 5.49 mm^{-1}. A pale-yellow plate with dimensions 0.20 × 0.14 × 0.05 mm was mounted on a Mac Science DIP3000 diffractometer with a graphite-monochromater. Oscillation and nonscreen Weissenberg photographs were recorded on the imaging plates of the diffractometer by using $Mo_{K\alpha}$ radiation (λ = 0.71073 Å) at 153 K and the data reduction was made by the MAC DENZO program system. Intensity data of 2565 independent reflections were collected in the range of $0 \le h \le 9$, $0 \le k \le 19$, $0 \le l \le 25$. Cell parameters were determined and refined by using the MAC DENZO for all observed reflections. The structure was solved by direct methods using SIR[1] in the CRYSTAN-GM program system. The atomic coordinates and anisotropic thermal parameters of the non-H atoms were refined by full-matrix least squares[2] to minimize the functions, $\Sigma(|F_o|-|F_c|)^2$, for 1992 reflections [$I \ge 2\sigma(I)$] (210 parameters). The structure was solved as a mixture consisted of 86% of one enantiomer (2*R*,3*R* or 2*S*,3*S*) and 14% of the other, leading unsatisfactory results. The final R (R_w)œ = 0.090 (0.094) and GOF = 3.422; max/min residual electron density = 2.37/–0.83 e Å^{-3}.

Crystal and molecular structure of 1–Adamantyl-t-butyltetrathiolane 2,3–Dioxide

Abstract

We present the crystal and molecular structure of 1–Adamantyl-t-butyltetrathiolane 2,3–Dioxe.

Comment

The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All Diagrams and calculations were performed using *CRYSTAN* (MacScience, Japan).

Experimental

Crystal data

$C_{15}H_{24}O_2S_4$
$M_r = 364.59$
Orthorhombic
$P2nn$
$a = 6.5220\ (7)$ Å
$b = 13.965\ (1)$ Å
$c = 18.279\ (2)$ Å
$V = 1664.8\ (3)$ Å^3
$Z = 4$

$D_x = 1.454$ Mg m^{-3}
Mo K-alpha radiation
$\lambda = 0.71073$ Å
$\mu = 5.492$ mm^{-1}
$T = 123$ K
Pale-yellow Plates
$0.20 \times 0.20 \times 0.09$ mm
Crystal source: Local laboratory

Data collection

Mac Science DIP3000 diffractometer
theta/2theta scans
Absorption correction:
none
2805 measured reflections
2578 independent reflections

2208 observed reflections
$[I > 2.00 \text{ sigma}(I)]$
$\theta_{max} = 28.95°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 25$

Refinement

Refinement on F
$R = 0.108$
$wR = 0.113$
$S = 4.753$
2208 reflections
223 parameters
Only coordinates of H atoms refined

Count statistics
$(\Delta/\sigma)_{max} = 0.2929$
$\Delta\rho_{max} = 3.65$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}
Extinction correction: None
Atomic scattering factors from *International Tables* (1974)

Data collection: MXC(MAC Science). Cell refinement: MXC(MAC Science). Data reduction: *CRYSTAN*. Program(s) used to solve structure: *CRYSTAN*. Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

FRACTIONAL ATOMIC COORDINATES & U(iso)

Atom	x/a	y/b	z/c	U(iso)
S(1)	0.59800	0.11157(10)	0.84533(7)	0.0275(6)
S(2)	0.1466(3)	0.1430(1)	0.8262(1)	0.0332(7)
S(3)	0.4524(4)	-0.0156(1)	0.8782(1)	0.0395(7)
S(4)	0.1692(4)	0.0581(1)	0.9172(1)	0.0324(8)
S(22)	0.6566(13)	0.0604(4)	0.9162(3)	0.022(3)
O(5)	0.3993(7)	-0.0795(2)	0.8184(2)	0.029(2)
O(8)	0.2332(10)	0.1065(3)	0.9846(2)	0.047(3)
O(23)	0.590(3)	0.112(1)	0.985(1)	0.015(3)
C(6)	0.4080(8)	0.1769(3)	0.7136(2)	0.017(2)
C(7)	0.4074(8)	0.1905(3)	0.7998(2)	0.018(2)
C(9)	0.4342(10)	0.2928(3)	0.8361(2)	0.034(3)
C(10)	0.4171(9)	0.0692(3)	0.6927(2)	0.024(2)
C(11)	0.2119(9)	0.2169(4)	0.6774(3)	0.031(2)
C(12)	0.5967(9)	0.2261(3)	0.6779(2)	0.024(2)
C(13)	0.5957(11)	0.2107(4)	0.5954(3)	0.042(3)
C(14)	0.4012(13)	0.2551(3)	0.5612(2)	0.043(3)
C(15)	0.3629(17)	0.2914(6)	0.9151(4)	0.037(4)
C(16)	0.6700(13)	0.3194(4)	0.8357(3)	0.037(3)
C(17)	0.2235(14)	0.0969(5)	0.5754(3)	0.052(4)
C(18)	0.2182(11)	0.2052(4)	0.5929(3)	0.042(3)
C(19)	0.4218(12)	0.0574(3)	0.6094(2)	0.041(3)
C(21)	0.3265(12)	0.3725(5)	0.7950(3)	0.035(3)
C(22)	0.6104(13)	0.1043(4)	0.5757(3)	0.049(3)
C(50)	0.495(5)	0.374(2)	0.795(1)	0.026(5)
C(48)	0.133(7)	0.319(3)	0.847(2)	0.047(8)
C(49)	0.442(4)	0.302(2)	0.917(1)	0.003(5)
H(11A)	0.19480	0.28473	0.69318	0.020
H(13)	0.70684	0.23193	0.58105	0.02(1)
H(10)	0.31432	0.04437	0.70453	0.020
H(12)	0.71248	0.19285	0.69789	0.02(1)
H(48)	0.15743	0.34690	0.80107	0.17(6)
H(22)	0.73326	0.06648	0.60289	0.02(1)
H(18)	0.09(3)	0.25(1)	0.56(1)	0.050
H(14A)	0.41(2)	0.25(1)	0.52(1)	0.05(2)
H(16)	0.70574	0.32731	0.79066	0.03(1)
H(11B)	0.13(3)	0.19(1)	0.70(1)	0.02(1)
H(14B)	0.362(16)	0.333(7)	0.564(5)	0.02(1)
H(17)	0.23617	0.09516	0.51213	0.06(2)

$$T = exp[2\pi^2 U];\ U = U_{iso}\ or\ U_{eqv};\ U_{eqv} = \frac{1}{3}\sum_{i=1}^{3}\sum_{j=1}^{3} U_{ij} a_i a_j \mathbf{a}_i . \mathbf{a}_j$$

ANISOTROPIC THERMAL PARAMETERS

Atom	U11	U22	U33	U12	U13	U23
S(1)	0.0291(7)	0.0227(6)	0.0305(6)	0.0094(6)	-.0143(5)	-.0056(5)
S(2)	0.0372(7)	0.0335(6)	0.0289(5)	-.0032(6)	0.0104(5)	-.0008(4)
S(3)	0.074(1)	0.017(1)	0.027(1)	0.005(1)	-0.006(1)	0.001(0)
S(4)	0.047(1)	0.027(1)	0.024(1)	0.005(1)	0.013(1)	0.003(1)
S(22)	0.043(4)	0.011(2)	0.013(2)	-0.001(3)	-0.012(3)	0.004(2)
O(5)	0.043(2)	0.014(2)	0.029(2)	-0.004(2)	0.007(2)	-0.005(1)
O(8)	0.093(5)	0.028(2)	0.021(2)	0.006(3)	0.007(2)	-0.006(2)
C(6)	0.021(2)	0.018(2)	0.013(1)	0.003(2)	0.003(2)	-0.001(1)
C(7)	0.018(2)	0.013(2)	0.022(2)	0.003(2)	-0.004(2)	0.000(1)
C(9)	0.063(4)	0.018(2)	0.022(2)	0.016(3)	-0.007(2)	-0.005(1)
C(10)	0.034(3)	0.014(2)	0.024(2)	0.003(2)	0.006(2)	-0.003(1)
C(11)	0.033(3)	0.029(3)	0.031(2)	0.004(2)	-0.009(2)	0.007(2)
C(12)	0.035(3)	0.017(2)	0.021(2)	-0.005(2)	0.006(2)	0.003(1)
C(13)	0.062(4)	0.031(3)	0.033(3)	-0.011(3)	0.031(3)	-0.004(2)
C(14)	0.084(5)	0.025(2)	0.021(2)	0.006(3)	0.004(3)	0.008(2)
C(15)	0.052(6)	0.037(4)	0.023(3)	-0.005(4)	-0.013(3)	-0.004(2)
C(16)	0.052(4)	0.025(3)	0.034(3)	-0.021(3)	-0.024(3)	0.003(2)
C(17)	0.095(6)	0.036(3)	0.026(2)	-0.010(4)	-0.026(3)	0.004(2)
C(18)	0.066(4)	0.029(3)	0.031(3)	-0.001(3)	-0.028(3)	0.006(2)
C(19)	0.080(5)	0.023(2)	0.020(2)	-0.014(3)	0.006(3)	-0.007(2)
C(21)	0.052(5)	0.018(3)	0.034(3)	0.013(3)	-0.009(3)	0.000(2)
C(22)	0.091(6)	0.028(3)	0.027(2)	0.003(3)	0.026(3)	-0.004(2)

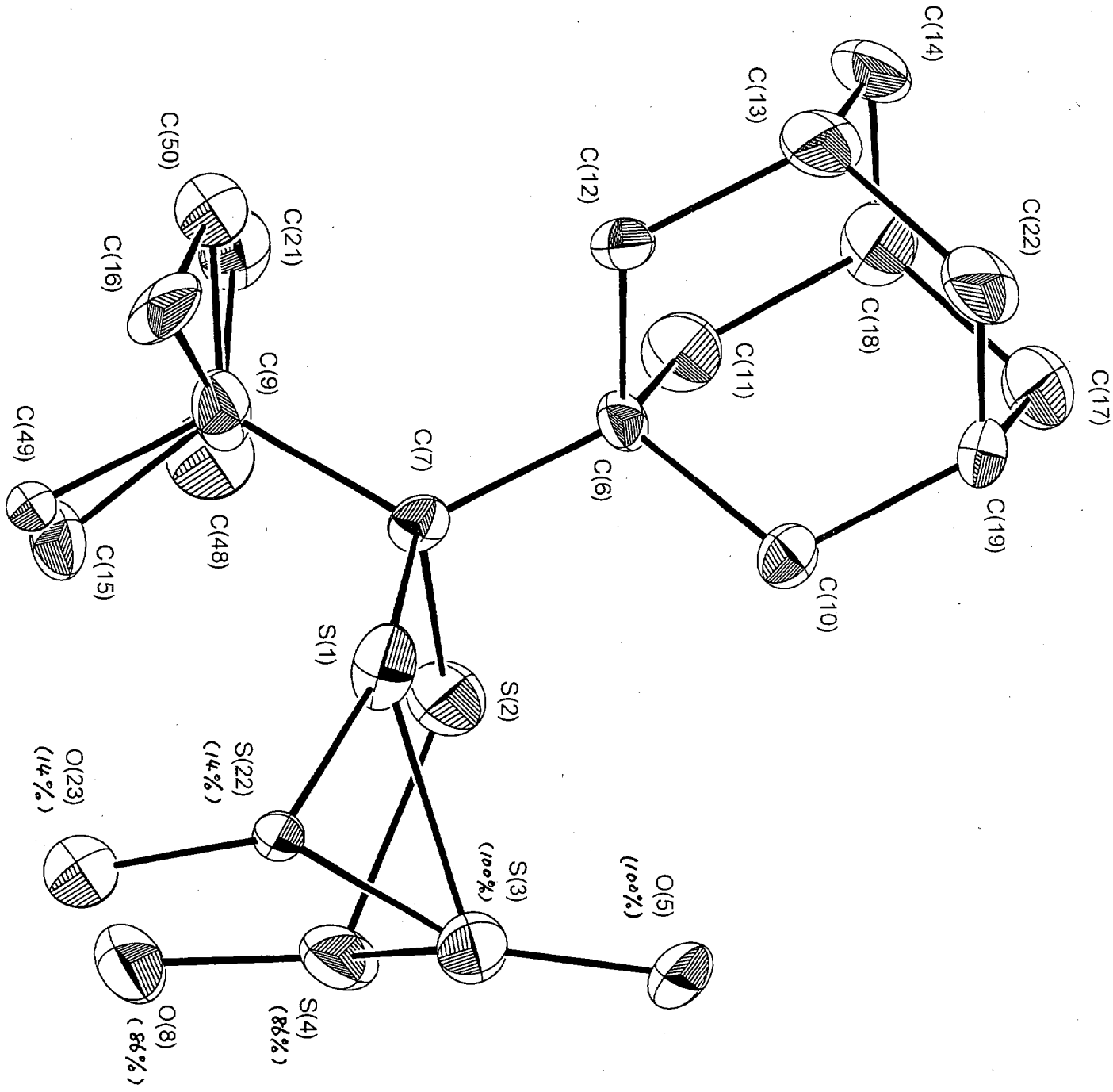
$T=\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{12}hka^*b^*+2U_{23}klb^*c^*+2U_{13}hla^*c^*)]$

INTRAMOLECULAR BOND LENGTHS

Minimum bond length= 0.70A	: Maximum bond length= 2.25A		
S(1)-S(3)	2.102(2)	S(1)-S(22)	1.528(6)
S(1)-C(7)	1.858(5)	S(2)-S(4)	2.048(2)
S(2)-C(7)	1.888(6)	S(3)-S(4)	2.231(4)
S(3)-S(22)	1.840(8)	S(3)-O(5)	1.453(4)
S(4)-O(8)	1.466(5)	S(22)-O(23)	1.510(17)
C(6)-C(7)	1.588(5)	C(6)-C(10)	1.553(6)
C(6)-C(11)	1.545(8)	C(6)-C(12)	1.553(7)
C(7)-C(9)	1.585(6)	C(9)-C(15)	1.517(9)
C(9)-C(16)	1.583(11)	C(9)-C(21)	1.515(9)
C(9)-C(50)	1.42(3)	C(9)-C(48)	2.01(5)
C(9)-C(49)	1.49(3)	C(10)-C(19)	1.531(6)
C(11)-C(18)	1.553(8)	C(12)-C(13)	1.522(7)
C(13)-C(14)	1.545(11)	C(13)-C(22)	1.533(8)
C(14)-C(18)	1.500(10)	C(15)-C(48)	1.98(5)
C(16)-C(50)	1.56(3)	C(16)-C(49)	2.12(3)
C(17)-C(18)	1.547(9)	C(17)-C(19)	1.538(11)
C(19)-C(22)	1.524(11)	C(21)-C(50)	1.10(4)
C(21)-C(48)	1.75(5)	C(6)-H(11A)	2.083(5)
C(6)-H(10)	1.956(4)	C(6)-H(12)	2.019(5)
C(6)-H(11B)	1.81(17)	C(9)-H(48)	2.058(7)
C(9)-H(16)	2.014(7)	C(10)-H(10)	0.785(6)
C(11)-H(11A)	0.996(6)	C(11)-H(11B)	0.83(16)
C(12)-H(13)	1.912(5)	C(12)-H(12)	0.959(6)
C(13)-H(13)	0.826(7)	C(13)-H(12)	2.037(6)
C(13)-H(22)	2.209(6)	C(13)-H(14A)	1.93(13)
C(14)-H(13)	2.052(9)	C(14)-H(18)	2.03(20)
C(14)-H(14A)	0.82(12)	C(14)-H(14B)	1.12(10)
C(16)-H(16)	0.862(7)	C(17)-H(17)	1.160(6)
C(18)-H(11A)	2.148(6)	C(18)-H(18)	1.17(19)
C(18)-H(14A)	1.97(13)	C(18)-H(11B)	2.12(15)
C(18)-H(14B)	2.09(10)	C(18)-H(17)	2.134(6)
C(19)-H(10)	1.883(5)	C(19)-H(22)	2.039(9)
C(19)-H(17)	2.216(6)	C(21)-H(48)	1.165(8)
C(22)-H(13)	1.893(7)	C(22)-H(22)	1.081(8)
C(50)-H(48)	2.24(3)	C(50)-H(16)	1.52(3)
C(48)-H(48)	0.95(4)		

INTRAMOLECULAR BOND ANGLES (H omitted)

Minimum bond length= 0.70A : Maximum bond length= 2.25A			
S(3)-S(1)-S(22)	58.4(3)	S(3)-S(1)-C(7)	109.1(2)
S(22)-S(1)-C(7)	145.5(4)	S(4)-S(2)-C(7)	110.2(2)
S(1)-S(3)-S(4)	94.3(1)	S(1)-S(3)-S(22)	45.0(2)
S(1)-S(3)-O(5)	114.3(2)	S(4)-S(3)-S(22)	102.3(3)
S(4)-S(3)-O(5)	109.0(3)	S(22)-S(3)-O(5)	144.2(3)
S(2)-S(4)-S(3)	93.9(1)	S(2)-S(4)-O(8)	115.9(3)
S(3)-S(4)-O(8)	104.2(3)	S(1)-S(22)-S(3)	76.6(3)
S(1)-S(22)-O(23)	114.3(8)	S(3)-S(22)-O(23)	112.4(9)
C(7)-C(6)-C(10)	111.1(3)	C(7)-C(6)-C(11)	112.3(4)
C(7)-C(6)-C(12)	111.5(4)	C(10)-C(6)-C(11)	106.1(4)
C(10)-C(6)-C(12)	107.1(4)	C(11)-C(6)-C(12)	108.4(4)
S(1)-C(7)-S(2)	106.3(2)	S(1)-C(7)-C(6)	111.8(3)
S(1)-C(7)-C(9)	105.9(4)	S(2)-C(7)-C(6)	102.3(3)
S(2)-C(7)-C(9)	108.0(4)	C(6)-C(7)-C(9)	121.5(4)
C(7)-C(9)-C(15)	110.6(5)	C(7)-C(9)-C(16)	108.5(5)
C(7)-C(9)-C(21)	113.8(5)	C(7)-C(9)-C(50)	122.1(12)
C(7)-C(9)-C(48)	95.5(12)	C(7)-C(9)-C(49)	119.8(11)
C(15)-C(9)-C(16)	107.8(6)	C(15)-C(9)-C(21)	109.9(6)
C(15)-C(9)-C(50)	126.9(12)	C(15)-C(9)-C(48)	66.7(12)
C(15)-C(9)-C(49)	20.6(11)	C(16)-C(9)-C(21)	106.0(5)
C(16)-C(9)-C(50)	62.5(13)	C(16)-C(9)-C(48)	155.4(12)
C(16)-C(9)-C(49)	87.2(11)	C(21)-C(9)-C(50)	43.8(13)
C(21)-C(9)-C(48)	57.8(12)	C(21)-C(9)-C(49)	116.5(11)
C(50)-C(9)-C(48)	100.7(17)	C(50)-C(9)-C(49)	116.7(16)
C(48)-C(9)-C(49)	85.1(15)	C(6)-C(10)-C(19)	110.4(4)
C(6)-C(11)-C(18)	111.5(5)	C(6)-C(12)-C(13)	110.5(5)
C(12)-C(13)-C(14)	110.4(5)	C(12)-C(13)-C(22)	111.7(5)
C(14)-C(13)-C(22)	110.2(6)	C(13)-C(14)-C(18)	108.1(5)
C(9)-C(15)-C(48)	68.6(13)	C(9)-C(16)-C(50)	53.6(12)
C(9)-C(16)-C(49)	44.5(7)	C(50)-C(16)-C(49)	83.0(13)
C(18)-C(17)-C(19)	106.6(6)	C(11)-C(18)-C(14)	110.9(5)
C(11)-C(18)-C(17)	108.0(5)	C(14)-C(18)-C(17)	110.9(6)
C(10)-C(19)-C(17)	110.3(6)	C(10)-C(19)-C(22)	111.9(5)
C(17)-C(19)-C(22)	111.2(5)	C(9)-C(21)-C(50)	63.4(16)
C(9)-C(21)-C(48)	75.3(14)	C(50)-C(21)-C(48)	136.7(20)
C(13)-C(22)-C(19)	105.7(6)	C(9)-C(50)-C(16)	63.9(12)
C(9)-C(50)-C(21)	72.8(17)	C(16)-C(50)-C(21)	136.1(24)
C(9)-C(48)-C(15)	44.7(10)	C(9)-C(48)-C(21)	46.9(11)
C(15)-C(48)-C(21)	83.1(18)	C(9)-C(49)-C(16)	48.2(8)



4,5-Dimethyl-3*H*, 6*H*-1,2-dithiin 1-oxide (**4**)

Pale-yellow oil. ^{1}H NMR (400 MHz) δ 1.96 (s, 3H), 2.02 (s, 3H), 3.18 (d, 1H, J = 13.9 Hz), 3.24 (d, 1H, J = 13.5 Hz), 3.76 (d, 1H, 13.8 Hz), 3.93 (d, 1H, J = 13.4 Hz); ^{13}C NMR (100.6 MHz) δ 19.71, 21.88, 33.99, 60.20, 123.66, 131.16; IR (KBr) 1080 cm^{-1}. Anal. Calcd for $C_6H_{10}OS_2$: C, 44.41; H, 6.21. Found: C, 43.99; H, 6.09.

1-Adamantyl *t*-butyl thioketone (**5**)

Purple crystals, m.p. 45—46 °C (EtOH). ^{1}H NMR (400 MHz) δ 1.45 (s, 9H), 1.71 (br s, 6H), 2.07 (br s, 3H), 2.18 (br s, 6H); ^{13}C NMR (100.6 MHz) δ 29.17, 32.73, 36.50, 43.71, 53.87, 56.74, 278.98; MS *m/z* 236 (M^+). Anal. Calcd for $C_{15}H_{24}S$: C, 76.21; H, 10.23. Found: C, 76.46; H, 10.44.

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