Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(4)	1·206 (4)	C(5)—C(6)	1·383 (5)
O(2)—C(4)	1·361 (4)	C(5)—C(10)	1·389 (5)
O(2)—C(5)	1·401 (4)	C(6)—C(7)	1·386 (5)
O(3)—C(11)	1·282 (4)	C(7)—C(8)	1·399 (5)
O(4)—C(11)	1·267 (4)	C(8)—C(9)	1·398 (5)
C(1)—C(2)	1·411 (5)	C(8)—C(11)	1·477 (5)
C(2)—C(3)	1·424 (6)	C(9)—C(10)	1·382 (5)
C(2)—C(4)	1·497 (6)	O(3)—H(O3)	0·950 (2)
		O(3)—O(4′)	2·620 (3)
		H(O3)—O(4′)	1·683 (2)
C(4)—O(2)—C(5)	118·4 (3)	C(5)—C(6)—C(7)	119·0 (3)
C(1)—C(2)—C(3)	124·3 (3)	C(6)—C(7)—C(8)	119·8 (3)
C(1)—C(2)—C(4)	119·7 (3)	C(7)—C(8)—C(9)	120·0 (3)
C(3)—C(2)—C(4)	116·0 (4)	C(7)—C(8)—C(11)	119·6 (3)
O(1)—C(4)—O(2)	124·1 (3)	C(9)—C(8)—C(11)	120·3 (3)
O(1)—C(4)—C(2)	124·7 (3)	C(8)—C(9)—C(10)	120·4 (3)
O(2)—C(4)—C(2)	111·2 (3)	C(5)—C(10)—C(9)	118·4 (3)
O(2)—C(5)—C(6)	116·3 (3)	O(3)—C(11)—O(4)	123·3 (3)
O(2)—C(5)—C(10)	121·1 (3)	O(3)—C(11)—C(8)	117·5 (3)
C(6)—C(5)—C(10)	122·4 (3)	O(4)—C(11)—C(8)	119·2 (3)

Primed atoms are derived from those in Table 1 by the operations $\bar{x}, \bar{y}, \bar{z}$.

Discussion. The molecular structure with the atomic numbering scheme is shown in Fig. 2 in the form of a dimer, consisting of two molecules held together by the carboxyl groups through hydrogen bonding. Such a tendency is quite common for molecules carrying carboxyl groups, even in solutions of non-polar or weakly polar solvents.

The positional parameters of the C and O atoms are listed in Table 1.* Selected distances and angles are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52486 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The arrangement of the molecules in the crystal is depicted in Fig. 3 showing alternating polar and non-polar layers owing to dimer formation. This explains the platelet-like or flat needle-like structure of the grown crystals (Fig. 1). It is possible that the layered nature of the crystals (Fig. 1) causes a small disorder. This disorder probably partly averages the single and double C—C bonds at the non-polar ends of the dimers, because the C(2)—C(3) and C(1)—C(2) bond distances are similar despite the low measurement temperature. Moreover, the difference between these two bonds hardly increased on lowering the temperature from ambient to 140 K which contrasts with previous findings concerning the compound (2,6-diphenyl)phenyl methacrylate (Viersen, Menge, Tan & van Bolhuis, 1988).

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Acetylsaturejol, an *endo*-Peroxide Menthane Derivative from *Satureja gilliessi*

BY VICTOR MANRIQUEZ, C. LABBE AND M. CASTILLO

Departamento de Quimica, Facultad de Ciencias, Universidad de Chile, Casilla 653, *Santiago, Chile*

AND H. G. VON SCHNERING AND K. PETERS

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, *D*-7000 *Stuttgart* 80, *Federal Republic of Germany*

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Abstract. $C_{12}H_{18}O_5$, $M_r = 242{\cdot}27$, monoclinic, $P2_1$, $a = 8{\cdot}187$ (3), $b = 11{\cdot}536$ (6), $c = 7{\cdot}698$ (3) Å, $\beta = 115{\cdot}17$ (3)°, $V = 658{\cdot}0$ (5) Å^3, $Z = 2$, $D_x = 1{\cdot}223$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 236$, $T = 293$ K, $R = 0{\cdot}060$, $wR = 0{\cdot}045$ for 894 unique observed reflections [$F >$

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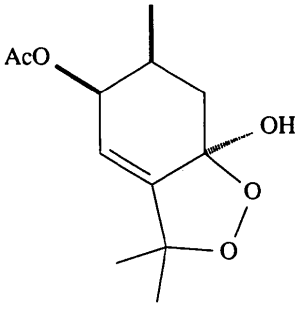
Table 1. *Atomic parameters* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	7887 (6)	3222	6668 (5)	60 (2)
C(1)	7781 (7)	3871 (7)	1908 (8)	47 (3)
O(2)	7179 (6)	2107 (5)	7064 (5)	76 (2)
C(2)	8186 (7)	4110 (7)	3996 (7)	45 (3)
C(3)	8047 (7)	2973 (7)	4929 (7)	44 (3)
C(4)	6315 (7)	2344 (6)	3798 (8)	45 (3)
C(5)	5331 (7)	2542 (7)	1934 (7)	45 (2)
C(6)	5862 (7)	3403 (7)	819 (7)	47 (3)
C(7)	8108 (9)	4949 (7)	915 (8)	67 (3)
C(8)	5829 (9)	1705 (7)	5219 (9)	55 (3)
C(9)	6147 (9)	391 (7)	5204 (9)	69 (3)
C(10)	3949 (8)	1983 (7)	5010 (10)	73 (4)
O(31)	9485 (5)	2215 (6)	5258 (6)	59 (2)
O(61)	4599 (5)	4388 (5)	438 (5)	50 (2)
O(62)	2533 (5)	3421 (6)	−2027 (6)	69 (2)
C(62)	2953 (8)	4260 (7)	−1006 (8)	51 (3)
C(63)	1758 (8)	5269 (7)	−1159 (8)	62 (3)

$3\sigma(F)$]. The bicyclic molecule is built up from a cyclic five-membered *endo*-peroxide fused to a menthane monoterpene skeleton. The acetyl and hydroxyl substituents are quasiaxial in a *trans* position. The molecule shows no unusual geometrical features.

Introduction. In the course of investigations of terpenoids from Chilean Labiatae we isolated the title compound from *Satureja gilliessi* (Labbe, Castillo & Conolly, 1989). Its spectroscopic data led to formula (1). Configurational details, especially of the hydroxyl group, as well as the confirmation of the rare peroxyhemiacetal function, were provided by an X-ray structure analysis.



(1)

Experimental. A plate-shaped crystal of dimensions $0{\cdot}2 \times 0{\cdot}25 \times 0{\cdot}1$ mm was used on a Nicolet *R3m*/V diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions from 22 centred reflections, $3 < \theta < 7{\cdot}5°$. Wyckoff scan used for data collection of 1226 unique reflections of which 894 were observed with $F > 3\sigma(F)$. According to the pre-scan intensity the Wyckoff-scan speed ranged from 1·5 to 19·5° min^{-1}. Absorption correction based on ψ scans of 11 reflections. Diffraction intensities were measured up to $(\sin\theta)/\lambda = 0{\cdot}60$ $Å^{-1}$ in the index range $h = 0$–9, $k = 0$–13 and $l = -9$–8. Three standard reflections (021, 11$\bar{1}$ and 02$\bar{1}$) varied less than 2·5% over 22·8 h of data collection. Solved by direct-phase determination, $E_{min} = 1{\cdot}2$. Full-matrix least squares minimized $w(\sigma F)^2$; H-atom positions calculated geometrically and considered isotropically with $U = 1{\cdot}2$ U of bonded C, the position of the O-bonded H atom was found on difference Fourier maps and refined isotropically. All other atoms refined anisotropically for 157 variables. $R = 0{\cdot}060$, $wR = 0{\cdot}045$, $S = 2{\cdot}73$, where $w^{-1} = \sigma^2(F)$. Final $(\Delta/\sigma)_{max} = 0{\cdot}016$, $\Delta\rho_{max} = 0{\cdot}3$ and $\Delta\rho_{min} = -0{\cdot}3$ e $Å^{-3}$ on final difference Fourier map. Atomic scattering factors taken from *SHELXTL-Plus* (Sheldrick, 1987).

Discussion. Table 1 gives the final positional and equivalent isotropic thermal parameters for all non-H atoms.* Fig. 1 shows a perspective drawing of the molecule with the atoms labelled according to the table.

The molecular structure of the title compound consists of one five-membered ring *endo*-peroxide fused to a menthane monoterpene skeleton with acetyl and hydroxyl substituents at C(6) and C(3), respectively. The molecule shows no unusual geometrical features. The six-membered ring has four atoms, C(3), C(4), C(5), and C(6), which lie exactly in a plane. The deviations of C(1) and C(2) from this plane are −0·25 and 0·51 Å, respectively. The C(3)—C(4)—C(8) plane of the five-membered ring

* Lists of bond lengths and angles, torsion angles, atomic coordinates of H atoms, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52509 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

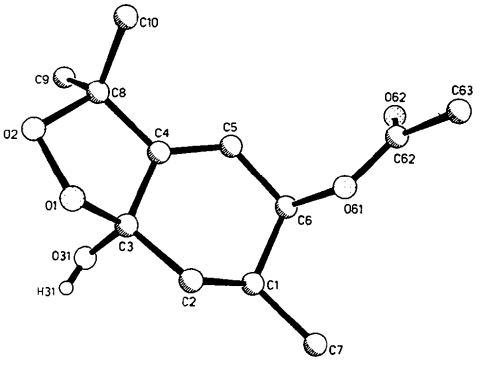


Fig. 1. Perspective drawing of the molecule with the atoms labelled according to the table. White, dotted and hatched circles represent H, C and O atoms, respectively.

forms an angle of 169·1° with the previous plane. The deviations of O(1) and O(2) from this plane of the five-membered ring are 0·73 and 0·15 Å, respectively. Except for C(2) five C atoms of the six-membered ring are nearly coplanar but the tendency toward a boat conformation is indicated by the mean absolute deviation of 7° (Table 4, deposited) of the torsion angles from the ideal boat: 0, 0, 28, −56, 54, −27°, cyclically starting from the double bond (Bucourt & Hainaut, 1965). The acetyl and hydroxyl substituents are quasiaxial in a *trans* position. All observed bond lengths are within the expected ranges.

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Structures of 4,5-Bis(methylthio)-2*H*-1,3-dithiole-2-thione and its 2-Oxo Analogue. Precursors to Organic Multisulfur π Donors

BY OLE SIMONSEN

Department of Chemistry, Odense University, DK-5230 Odense M, Denmark

AND K. S. VARMA, A. CLARK AND A. E. UNDERHILL

Department of Chemistry and Institute for Molecular and Biomolecular Electronics, University College of N Wales, Bangor, Gwynedd LL57 2DG, Wales

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Abstract. 4,5-Bis(methylthio)-2*H*-1,3-dithiole-2-thione, $C_5H_6S_5$ (BTDTT), $M_r = 226{\cdot}40$, monoclinic, $P2_1/c$, $a = 7{\cdot}543\ (2)$, $b = 12{\cdot}480\ (2)$, $c = 9{\cdot}896\ (1)$ Å, $\beta = 99{\cdot}81\ (1)°$, $V = 918{\cdot}0\ (5)$ Å^3, $Z = 4$, $D_m = 1{\cdot}62\ (1)$, $D_x = 1{\cdot}638$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}14$ mm^{-1}, $F(000) = 464$, $T = 295$ K. $R = 0{\cdot}027$ for 1928 unique observed reflections. 4,5-Bis(methylthio)-2*H*-1,3-dithiole-2-one, $C_5H_6OS_4$ (BTDTO), $M_r = 210{\cdot}34$, monoclinic, $P2_1$, $a = 11{\cdot}087\ (2)$, $b = 9{\cdot}198\ (2)$, $c = 4{\cdot}194\ (2)$ Å, $\beta = 96{\cdot}03\ (2)°$, $V = 425{\cdot}2\ (4)$ Å^3, $Z = 2$, $D_m = 1{\cdot}62\ (1)$, $D_x = 1{\cdot}643$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}01$ mm^{-1}, $F(000) = 216$, $T = 295$ K, $R = 0{\cdot}026$ for 1261 unique observed reflections. The dithiole ring in both BTDTT and BTDTO is planar. Torsion angles involving the CH_3—S groups and the C=C bond vary between −121 and −178°. The endocyclic C—S bonds are shorter than the exocyclic C—S single bonds. The two endocyclic C—S bonds in the S—C—S region of the ring in BTDTO are longer than those in BTDTT. The S···S contact distances have values between 3·54 and 3·68 Å.

Introduction. The quest for molecular metals and superconductors has concentrated on multisulfur π donors and ligands over the past five years. This is due to the discovery in 1983 of a new family of organic superconductors based on 3,4;3′,4′-bis(ethylenedithio)-2,2′,5,5′-tetrathiafulvalene (BEDT-TTF) (Parkin, Engler, Schumaker, Lagier, Lee, Scott & Greene, 1983). The compound initially studied, β-(BEDT-TTF)$_2$I$_3$, had a superconductivity transition temperature (T_c) of approximately 1·6 K, but the value of T_c has been raised to 10·4 K in (BEDT-TTF)$_2$ Cu(SCN)$_2$ (Kikuchi, Murata, Honda, Namiki, Saito, Kobayashi, Ishiguro & Ikemoto, 1987). The related ligand, dmit, has also been shown to give rise to a series of molecular metals and superconductors (Kobayashi, Kim, Sasaki, Kato, Kobayashi, Moriyama, Nishio, Kajita & Sasaki, 1987; Clark, Underhill, Parker & Friend, 1989).

As part of an extensive investigation into molecular conductors we have prepared and determined the crystal structure of 4,5-bis(methylthio)-2*H*-1,3-dithiole-2-thione BTDTT and 4,5-bis(methylthio)-2*H*-1,3-dithiole-2-one BTDTO to study the influence of the *exo* heteroatom attached to C(1) on the structures of the multisulfur π systems.

Experimental. BTDTT was prepared by methylating sodium 4,5-dimercapto-1,3-dithiole-2-thione (Poleschner, John, Hoppe & Fanghänel, 1983). BTDTO was prepared by treatment of BTDTT with mercuric acetate (Varma, Bury, Harris

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