C3—C4—S5	116.2 (8)	C34—C35—C36	119.8 (9)
C4—S5—S6	103.7 (3)	C35—C36—C31	120.1 (9)
C7—S6—S5	102.1 (4)	C82—C81—C86	118.6 (8)
C8—C7—S6	114.9 (6)	C82—C81—C8	121.8 (7)
N9—C8—C7	110.5 (7)	C86—C81—C8	119.5 (7)
N9—C8—C81	111.0 (7)	O82—C82—C83	119.2 (8)
C7—C8—C81	108.6 (6)	O82—C82—C81	120.6 (8)
C10—N9—C8	112.7 (8)	C83—C82—C81	120.2 (8)
C32—C31—C36	118.0 (8)	C82—C83—C84	120.5 (9)
C32—C31—C3	123.1 (7)	C85—C84—C83	120.2 (10)
C36—C31—C3	118.7 (7)	C84—C85—C86	120.0 (10)
O32—C32—C31	121.0 (8)	C81—C86—C85	120.5 (10)

Symmetry codes: (i) $x - 1, y - 1, z$; (ii) $1 + x, y, z$.

23 of 25 reflections found on a polaroid photograph could be used by the indexing procedure to obtain a preliminary cell. The indices of the remaining reflections ($-1\ -1\ -1.75$ and $-2\ 0\ -2.5$) gave a strong indication of twinning. The following conditions (confirmed by scanning the reciprocal space) for the additional reflections could be derived: for $h = 4m + 1$ there were reflections with $l = n + 0.75$, for $h = 4m + 2$ there were reflections with $l = n + 0.5$ and for $h = 4m + 3$ there were reflections with $l = n + 0.25$ (with m, n integers). These reflections of the two twin components were separated sufficiently for a satisfactory data collection, but overlap occurred for the reflections with $h = 4m$. The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1985). For refinement the data were read in *via HKLF*5 and an additional variable was introduced (using the *BASF* command) describing the fractional contributions of the two twin components for the reflections with $h = 4m$; the ratio refined to 0.324 (6). Comparing this refinement with two other procedures leads to the following results: omitting all reflections with $h = 4m$ from refinement gives $wR(F^2) = 0.500$ and a mean e.s.d. of a C—C bond (σ) = 0.018 Å, while treating the reflections with $h = 4m$ as normal data, *i.e.* as if there was no twinning, gives $wR(F^2) = 0.4794$ and $\sigma = 0.02$ Å; for comparison, the twin refinement results in $wR(F^2) = 0.4129$ and $\sigma = 0.013$ Å.

All H atoms were located by a difference Fourier synthesis and refined with fixed individual displacement parameters [$U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C_{methyl}},\mathrm{O})$ or $U(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C},\mathrm{N})$] using a riding model with C—H(non aromatic) = 0.96 or C—H(aromatic) = 0.93 Å, respectively. The N—H distance was refined and the O—H distance was fixed to 0.82 Å but the torsion angle about the O—C bond was refined. Data collection: *SDP* (Enraf-Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL XP* (Sheldrick, 1991).

I thank Professor Dr A. Berkessel (University of Heidelberg) for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1127). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C50, 1370–1372

Configuration of (*E*)-2-(1-Hydroxy-1-methyl-2-oxopropyl)-2-(methylthio)thiolane *S*-Oxide†

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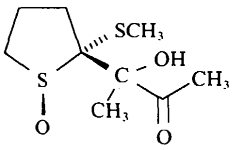
Abstract

The title compound, $C_9H_{16}O_3S_2$ (1), exhibits an (*E*)-configuration with a quasi-diaxial (*trans*) arrangement of the 1-oxido and 2-methylthio substituents. This clearly shows that the carbanion exhibits configurational stability under the reaction conditions. Pairs of molecules of (1) form dimers through two hydrogen bonds, with OH groups as donors and SO groups as acceptors.

Comment

The title hydroxyketone, (1), was formed by the reaction of 2-(methylthio)thiolane 1-oxide with butyllithium, followed by 2,3-butanedione (Brunck, 1993). Two diastereomers of (1) were obtained in a ratio of 3:1 and could be separated by column chromatography. Since their configuration could not be determined by spectroscopic methods, we have performed an X-ray analysis of the main diastereomer, which exhibits the (*E*)-configuration.



(1)

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† C,C-Coupling with Sulfur-Stabilized Carbanions. 5. Part 4: Böge, Schwär & Voss (1993).

No intramolecular O—H···O=C or O—H···O=S hydrogen bonds were found. However, associations (dimers) are formed in the crystal through intermolecular hydrogen bonds of the latter type (Table 2).

The molecular structures of thiolane *S*-oxide and 2-methylthiolane *S*-oxide (Forgacs, Schultz, Hargittai, Jalsovszky & Kucsman, 1989), as well as (*E*)-2-methylthio-2-(3-oxobutyl)thiolane 1-oxide (Brunck, Voss, Olbrich & Viebrock, 1993) have been reported. The latter compound, which is closely related to (1), also exhibits the quasi-diaxial (*trans*) configuration of the oxido and methylthio substituents. This seems to be a result of compensation for the S—O and C—SMe dipoles.

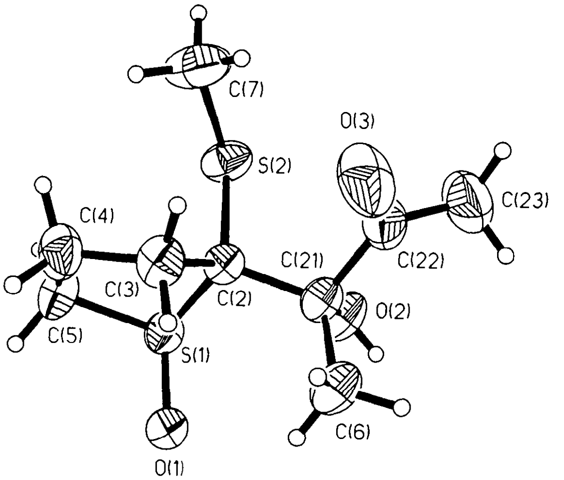

Fig. 1. Structure of (1) showing 50% probability displacement ellipsoids (*SHELXTL-Plus*; Sheldrick, 1990*b*)

Experimental

A crystal (m.p. 372 K) suitable for X-ray analysis was obtained by slow crystallization from ethyl acetate/petroleum ether/trichloromethane.

Crystal data

$C_9H_{16}O_3S_2$
$M_r = 236.34$
Monoclinic
$P2_1/a$
$a = 9.027\ (4)$ Å
$b = 10.561\ (4)$ Å
$c = 11.768\ (4)$ Å
$\beta = 98.81\ (3)°$
$V = 1108.7\ (7)$ Å^3
$Z = 4$
$D_x = 1.416$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 15 reflections
$\theta = 4$–19°
$\mu = 0.460$ mm^{-1}
$T = 293\ (2)$ K
Transparent blocks
$0.7 \times 0.7 \times 0.3$ mm
Colourless

Data collection

Hilger & Watts (Y290) diffractometer
$2\theta/\omega$ scans
Absorption correction: none
3543 measured reflections
3239 independent reflections
2698 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.0078$
$\theta_{\rm max} = 30.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 14$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 97 reflections intensity variation: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0516$
$wR(F^2) = 0.1418$
$S = 1.257$
3227 reflections
132 parameters
H atoms riding
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.4031P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.098$
$\Delta\rho_{\rm max} = 0.761$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.742$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

$U_{\rm iso}$ for H; $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for other atoms.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
S1	0.23178 (6)	0.87676 (5)	0.41472 (5)	0.0407 (2)
S2	0.25640 (7)	0.83861 (6)	0.18399 (5)	0.0488 (2)
O2	0.5019 (2)	0.9562 (2)	0.3497 (2)	0.0494 (4)
C2	0.2455 (2)	0.9689 (2)	0.2843 (2)	0.0346 (4)
O1	0.2504 (2)	0.9642 (2)	0.51521 (15)	0.0543 (5)
C21	0.3937 (2)	1.0425 (2)	0.2994 (2)	0.0401 (4)
C4	−0.0247 (3)	0.9601 (3)	0.2878 (2)	0.0533 (6)
C3	0.1018 (2)	1.0437 (2)	0.2640 (2)	0.0451 (5)
C6	0.3954 (3)	1.1617 (2)	0.3710 (3)	0.0544 (6)
C5	0.0332 (3)	0.8616 (3)	0.3752 (2)	0.0513 (6)
C7	0.1318 (4)	0.8778 (4)	0.0579 (3)	0.0773 (10)
O3	0.3320 (3)	1.1387 (3)	0.1176 (2)	0.0822 (8)
C22	0.4258 (3)	1.0833 (3)	0.1813 (2)	0.0548 (6)
C23	0.5765 (4)	1.0591 (5)	0.1524 (3)	0.0881 (12)
H2	0.567 (3)	0.988 (3)	0.387 (3)	0.058 (9)

Table 2. *Selected geometric parameters* (Å, °)

S1—O1	1.489 (2)	C2—C21	1.534 (3)
S1—C5	1.789 (3)	C21—C6	1.514 (3)
S1—C2	1.837 (2)	C21—C22	1.525 (4)
S2—C7	1.768 (3)	C4—C5	1.499 (4)
S2—C2	1.826 (2)	C4—C3	1.503 (4)
O2—C21	1.399 (3)	O3—C22	1.194 (4)
C2—C3	1.506 (3)	C22—C23	1.475 (4)
O1—S1—C5	104.38 (12)	O2—C21—C22	110.6 (2)
O1—S1—C2	108.72 (11)	C6—C21—C22	106.5 (2)
C5—S1—C2	91.33 (11)	O2—C21—C2	105.0 (2)
C7—S2—C2	106.01 (14)	C6—C21—C2	114.8 (2)
C21—O2—H2	112.9 (25)	C22—C21—C2	108.8 (2)
C3—C2—C21	117.9 (2)	C5—C4—C3	109.9 (2)
C3—C2—S2	115.01 (15)	C4—C3—C2	108.8 (2)
C21—C2—S2	108.91 (14)	C4—C5—S1	110.3 (2)
C3—C2—S1	104.11 (15)	O3—C22—C23	122.0 (3)
C21—C2—S1	109.92 (14)	O3—C22—C21	119.7 (3)
S2—C2—S1	99.09 (10)	C23—C22—C21	118.2 (3)
O2—C21—C6	111.1 (2)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1$^{\rm i}$	0.75 (3)	1.93 (3)	2.673 (3)	168 (3)

Symmetry code: (i) $1 - x, 2 - y, 1 - z$.

Data collection and cell refinement: *Diffractometer Control Program for the Hilger & Watts* (Y290) (Kopf & Abeln, 1993). Data reduction: *WATSHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93 *CIFTAB*; *FCF2FOC* (Kopf, 1992).

This work was supported by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1150). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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ADDENDA AND ERRATA

Acta Cryst. (1994). C**50**, 1372

The structures of alkalides and electrides. II. Structure of caesium bis(15-crown-5) electride. Erratum.

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(*Received* 15 *June* 1994)

Abstract

An error in printing is corrected. In Table 1 of the paper by Ward, Huang, Kuchenmeister & Dye [*Acta Cryst.* (1990), C**46**, 1831–1833] the y coordinate of atom O1 is printed incorrectly as −0.1501 (2). The correct value of this coordinate is +0.1501 (2).

All relevant information is given in the *Abstract*.

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