Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–S(2)	1·746 (6)	C(1)–C(6)	1·337 (8)
C(1)–S(7)	1·747 (6)	S(2)–C(3)	1·804 (7)
C(3)–H(3*A*)	0·960 (1)	C(3)–H(3*B*)	0·960 (1)
C(3)–C(4)	1·462 (10)	C(4)–H(4*A*)	0·960 (1)
C(4)–H(4*B*)	0·960 (1)	C(4)–S(5)	1·781 (6)
S(5)–C(6)	1·767 (5)	C(6)–S(9)	1·720 (5)
S(7)–C(8)	1·748 (6)	C(8)–S(9)	1·741 (6)
C(8)–O	1·226 (7)		
S(2)–C(1)–C(6)	129·1 (4)	S(2)–C(1)–S(7)	114·6 (3)
C(6)–C(1)–S(7)	116·3 (4)	C(1)–S(2)–C(3)	100·9 (3)
S(2)–C(3)–H(3*A*)	108·3 (2)	S(2)–C(3)–H(3*B*)	108·3 (2)
H(3*A*)–C(3)–H(3*B*)	109·5 (1)	S(2)–C(3)–C(4)	114·1 (5)
H(3*A*)–C(3)–C(4)	108·3 (4)	H(3*B*)–C(3)–C(4)	108·3 (4)
C(3)–C(4)–H(4*A*)	107·5 (4)	C(3)–C(4)–H(4*B*)	107·6 (4)
H(4*A*)–C(4)–H(4*B*)	109·5 (1)	C(3)–C(4)–S(5)	117·0 (5)
H(4*A*)–C(4)–S(5)	107·5 (2)	H(4*B*)–C(4)–S(5)	107·6 (2)
C(4)–S(5)–C(6)	102·2 (3)	C(1)–C(6)–S(5)	127·1 (4)
C(1)–C(6)–S(9)	118·0 (4)	S(5)–C(6)–S(9)	115·0 (3)
C(1)–S(7)–C(8)	95·9 (3)	S(7)–C(8)–S(9)	113·5 (3)
S(7)–C(8)–O	122·2 (5)	S(9)–C(8)–O	124·3 (5)
C(6)–S(9)–C(8)	96·3 (3)		

Related literature. We have published the synthesis of 5,6-dihydro-1,4-dithiin-2,3-dithiolate (DDDT) and the crystal structure of $[N(C_2H_5)_4]^+.[Ni(DDDT)_2]^-$ (Vance, Bereman, Bordner, Hatfield & Helms, 1985).

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Structure of 7-Phenylthio-1,4-dioxaspiro[4.5]decan-8-one

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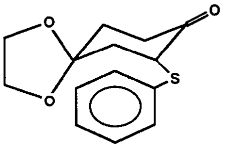
Abstract. $C_{14}H_{16}O_3S$, $M_r = 264{\cdot}34$, monoclinic, $P2_1/c$, $a = 14{\cdot}291$ (7), $b = 5{\cdot}665$ (2), $c = 15{\cdot}872$ (5) Å, $\beta = 94{\cdot}63$ (3)°, $V = 1280{\cdot}8$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}37$ (163 K), $D_m = 1{\cdot}32$ g cm^{-3} (295 K), λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}38$ cm^{-1}, $F(000) = 560$, $T = 163$ K, $R = 0{\cdot}0450$ for 2005 unique observed reflections. The cyclohexanone ring is in the chair conformation. The phenylthio group is forced to be in the equatorial position by the presence of the dioxolane moiety. There is a close, non-bonded intramolecular S···O contact [2·802 (2) Å] involving the carbonyl O atom.

Experimental. Title compound synthesized by extension of the methods described by Trost, Salzmann & Hiroi (1976) to monoketalized 1,4-cyclohexanedione prepared according to Haslanger & Lawton (1974).



Colorless plate, cut from a larger crystal, 0·06 × 0·31 × 0·50 mm from hexane. Crystal density by flotation method in aqueous $ZnCl_2$. Syntex $P2_1$ diffractometer, graphite monochromator, Syntex LT-1 low-temperature delivery system (163 K). Lattice parameters from least-squares refinement of 45 reflections with $18{\cdot}0 < 2\theta < 25{\cdot}2°$. ω-scan technique (5079 reflections, 2578 unique, $R_{\rm int} = 0{\cdot}038$), 2θ range 4·0–52·5°, 1° ω scan at 2·5–5·0° min^{-1} ($h = -4\rightarrow17$, $k = -7\rightarrow6$, $l = -19\rightarrow19$). The space group was determined from systematic absences. Four reflections ($\bar{3}00$, $\bar{1}\bar{1}2$, 211, $1\bar{1}\bar{1}$) remeasured every 96 reflections to monitor instrument and crystal stability. Analysis of these data indicated that no such corrections were necessary. Data corrected for Lp effects and absorption (based on crystal shape; transmission factors 0·932–0·987). Data reduction described in Riley & Davis (1976). Reflections having $F_o < 4\sigma(F_o)$ considered unobserved (573 reflections). Structure solved by the heavy-atom method and refined by full-matrix least-squares procedures (Sheldrick, 1976) with anisotropic thermal parameters for the non-H atoms. H atoms from a ΔF map and refined with isotropic thermal parameters; 227 parameters refined. $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = [0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}04I)^2\}^{1/2}]$. Intensity, I, given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate); 0·04 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp

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Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters* (Å²) *for* $C_{14}H_{16}O_3S$

	x	*y*	*z*	*U**
O(1)	0·32605 (11)	−0·1642 (3)	−0·02364 (9)	0·0234 (5)
C(2)	0·3247 (2)	−0·3183 (4)	−0·09540 (15)	0·0272 (8)
C(3)	0·2893 (2)	−0·5459 (5)	−0·0613 (2)	0·0288 (8)
O(4)	0·33131 (12)	−0·5470 (3)	0·02361 (10)	0·0286 (6)
C(5)	0·3431 (2)	−0·3076 (4)	0·05026 (14)	0·0222 (7)
C(6)	0·2718 (2)	−0·2424 (4)	0·1128 (2)	0·0233 (8)
C(7)	0·2853 (2)	0·0135 (4)	0·14171 (13)	0·0203 (7)
C(8)	0·3856 (2)	0·0564 (4)	0·17732 (14)	0·0243 (7)
C(9)	0·4596 (2)	−0·0250 (4)	0·1220 (2)	0·0254 (8)
C(10)	0·4424 (2)	−0·2768 (4)	0·0895 (2)	0·0242 (7)
O(11)	0·40400 (13)	0·1511 (3)	0·24512 (11)	0·0401 (6)
S(12)	0·20830 (4)	0·11008 (11)	0·22122 (4)	0·0261 (2)
C(13)	0·0961 (2)	0·0736 (4)	0·16528 (13)	0·0214 (7)
C(14)	0·0417 (2)	−0·1226 (4)	0·18042 (14)	0·0248 (7)
C(15)	−0·0487 (2)	−0·1408 (5)	0·14179 (15)	0·0282 (8)
C(16)	−0·0850 (2)	0·0354 (5)	0·0884 (2)	0·0302 (8)
C(17)	−0·0309 (2)	0·2302 (5)	0·0731 (2)	0·0313 (8)
C(18)	0·0593 (2)	0·2502 (4)	0·11158 (15)	0·0277 (8)
H(C2*A*)	0·390 (2)	−0·334 (4)	−0·114 (2)	0·029 (7)
H(C2*B*)	0·282 (2)	−0·251 (4)	−0·1384 (15)	0·023 (6)
H(C3*A*)	0·308 (2)	−0·680 (4)	−0·0900 (13)	0·013 (6)
H(C3*B*)	0·223 (2)	−0·549 (5)	−0·062 (2)	0·048 (9)
H(C6*A*)	0·278 (2)	−0·348 (4)	0·1611 (15)	0·027 (7)
H(C6*B*)	0·209 (2)	−0·265 (4)	0·0849 (14)	0·025 (6)
H(C7)	0·272 (2)	0·120 (4)	0·0910 (15)	0·028 (6)
H(C9*A*)	0·456 (2)	0·088 (4)	0·0755 (14)	0·020 (6)
H(C9*B*)	0·518 (2)	−0·012 (4)	0·1533 (14)	0·028 (7)
H(C10*A*)	0·453 (2)	−0·390 (4)	0·135 (2)	0·032 (7)
H(C10*B*)	0·487 (2)	−0·311 (4)	0·048 (2)	0·030 (7)
H(C14)	0·065 (2)	−0·245 (4)	0·219 (2)	0·028 (7)
H(C15)	−0·085 (2)	−0·285 (5)	0·153 (2)	0·041 (8)
H(C16)	−0·144 (2)	0·022 (4)	0·0622 (14)	0·026 (7)
H(C17)	−0·055 (2)	0·356 (4)	0·0367 (15)	0·030 (7)
H(C18)	0·100 (2)	0·381 (4)	0·1011 (15)	0·031 (7)

* For anisotropic atoms, the U value is U_{eq}, calculated as $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$ where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

effects and absorption. $\sigma(I)$ estimated from counting statistics; $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times \text{(scan rate)}]$. Final $R = 0{\cdot}0450$ for 2005 reflections, $wR = 0{\cdot}0406$ ($R_{\rm all} = 0{\cdot}0675$, $wR_{\rm all} = 0{\cdot}0440$) and a goodness-of-fit = 1·410. Maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were −0·28 and 0·33 e Å^{-3}, respectively. Scattering factors for the non-H atoms were from Cromer & Mann (1968), with anomalous-dispersion corrections from Cromer & Liberman (1970), while scattering factors for the H atoms were from Stewart, Davidson & Simpson (1965); linear absorption coefficient from *International Tables for X-ray Crystallography* (1974).* Positional and isotropic thermal parameters are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. The least-squares-planes' program was supplied by Cordes (1983); other computer programs from reference 11 of Gadol & Davis (1982).

Table 2. *Bond lengths* (Å) *and angles* (°) *for non*-H *atoms of* $C_{14}H_{16}O_3S$

1	2	3	1–2	1–2–3
C(2)	O(1)	C(5)	1·434 (3)	107·2 (2)
C(5)	O(1)		1·431 (3)	
C(3)	C(2)	O(1)	1·501 (4)	102·7 (2)
O(4)	C(3)	C(2)	1·431 (3)	102·4 (2)
C(5)	O(4)	C(3)	1·426 (3)	107·8 (2)
C(6)	C(5)	C(10)	1·524 (4)	111·0 (2)
C(6)	C(5)	O(1)		108·5 (2)
C(6)	C(5)	O(4)		110·8 (2)
C(10)	C(5)	O(1)	1·513 (3)	111·3 (2)
C(10)	C(5)	O(4)		108·4 (2)
O(1)	C(5)	O(4)		106·8 (2)
C(7)	C(6)	C(5)	1·528 (3)	110·6 (2)
C(8)	C(7)	S(12)	1·518 (3)	107·4 (2)
C(8)	C(7)	C(6)		110·9 (2)
S(12)	C(7)	C(6)	1·824 (2)	115·1 (2)
C(9)	C(8)	O(11)	1·501 (4)	122·8 (2)
C(9)	C(8)	C(7)		115·0 (2)
O(11)	C(8)	C(7)	1·212 (3)	122·2 (2)
C(10)	C(9)	C(8)	1·531 (4)	112·5 (2)
C(5)	C(10)	C(9)		111·5 (2)
C(13)	S(12)	C(7)	1·781 (2)	100·91 (11)
C(14)	C(13)	C(18)	1·388 (3)	119·5 (2)
C(14)	C(13)	S(12)		120·0 (2)
C(18)	C(13)	S(12)	1·389 (3)	120·2 (2)
C(15)	C(14)	C(13)	1·388 (3)	119·8 (2)
C(16)	C(15)	C(14)	1·383 (4)	120·4 (2)
C(17)	C(16)	C(15)	1·380 (4)	119·8 (2)
C(18)	C(17)	C(16)	1·385 (4)	120·1 (2)
C(13)	C(18)	C(17)		120·3 (2)

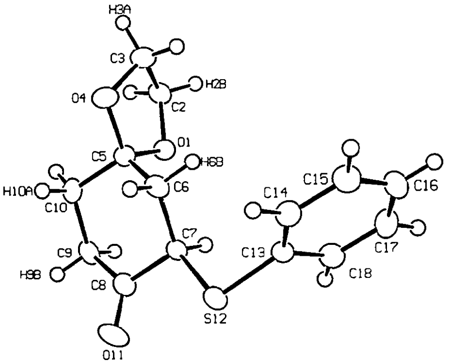

Fig. 1. View of title compound illustrating the atom-labeling scheme. Thermal ellipsoids are scaled to the 50% probability level.

Related literature. A discussion of the synthesis, the resulting stereochemistry and the chemical utility of sulfenylated esters and ketones is given by Trost *et al.* (1976) and references therein.

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*Tables of anisotropic thermal parameters, bond distances and angles involving the H atoms, torsion angles, least-squares planes, structure-factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43395 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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