

Zn atoms occupy the regions near $z = 0, 0.5, 1, \dots$, and the Ag atoms those near $z = 0.25, 0.75, \dots$ (Fig. 2).

Experimental

Crystal data

ZnAg ₂ (SCN) ₄	$\theta = 10\text{--}11.5^\circ$
$M_r = 513.4$	$V = 1170.0 (7) \text{ \AA}^3$
Monoclinic	$Z = 4$
$C2/c$	$D_x = 2.915 \text{ Mg m}^{-3}$
$a = 19.720 (9) \text{ \AA}$	Mo $K\alpha$
$b = 7.703 (3) \text{ \AA}$	$\lambda = 0.71069 \text{ \AA}$
$c = 7.753 (3) \text{ \AA}$	$\mu = 6.0 \text{ mm}^{-1}$
$\beta = 96.57 (4)^\circ$	$T = 178 \text{ K}$
Cell parameters from 49 reflections	Prism
	$0.9 \times 0.7 \times 0.4 \text{ mm}$
	Colourless

Data collection

Siemens R3 diffractometer	$R_{\text{int}} = 0.04$
ω scans	$\theta_{\text{max}} = 27.5^\circ$
Absorption correction: empirical ψ scans	$h = -25 \rightarrow 25$
$T_{\text{min}} = 0.23, T_{\text{max}} = 0.99$	$k = 0 \rightarrow 10$
5383 measured reflections	$l = 0 \rightarrow 10$
1351 independent reflections	3 standard reflections
1328 observed reflections	monitored every 147 reflections
$[F > 4\sigma(F)]$	intensity variation: $\pm 1.5\%$

Refinement

Refinement on F	$(\Delta/\sigma)_{\text{max}} = 0.002$
Final $R = 0.026$	$\Delta\rho_{\text{max}} = 1.0 \text{ e \AA}^{-3}$
$wR = 0.030$	$\Delta\rho_{\text{min}} = -0.9 \text{ e \AA}^{-3}$
$S = 1.6$	Atomic scattering factors
1328 reflections	from <i>International Tables</i>
70 parameters	for <i>X-ray Crystallography</i>
$w = 1/[\sigma^2(F) + 0.0001F^2]$	(1974, Vol. IV)
Extinction correction applied using $F_{\text{corr}} = F/[1 + 0.002x F^2/\sin 2\theta]^{1/4}$, with $x = 0.00201(9)$.	

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

	$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$			
	x	y	z	U_{eq}
Ag	0.20526 (1)	0.09578 (2)	0.26763 (2)	0.0251 (1)
Zn	0.50000	0.16483 (4)	0.25000	0.0167 (1)
S(1)	0.29939 (3)	-0.11251 (6)	0.38966 (6)	0.0175 (1)
S(2)	0.13289 (3)	0.10224 (6)	0.53246 (6)	0.0192 (2)
N(1)	0.42556 (10)	0.01780 (22)	0.31417 (24)	0.0237 (5)
N(2)	0.04535 (10)	-0.17250 (21)	0.42132 (23)	0.0208 (5)
C(1)	0.37350 (11)	-0.03712 (22)	0.34171 (24)	0.0168 (5)
C(2)	0.08141 (11)	-0.05824 (25)	0.46642 (24)	0.0168 (5)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Ag—S(1)	2.552 (2)	Ag—S(2)	2.632 (2)
Ag—S(1')	2.553 (2)	Ag—S(2')	2.663 (2)
Zn—N(1)	1.963 (2)	Zn—N(2')	1.965 (2)
S(1)—C(1)	1.654 (2)	S(2)—C(2)	1.644 (2)
N(1)—C(1)	1.153 (3)	N(2)—C(2)	1.160 (3)

S(1)—Ag—S(2)	99.2 (1)	S(1)—Ag—S(1')	135.7 (1)
S(2)—Ag—S(1')	111.3 (1)	S(1)—Ag—S(2')	101.8 (1)
S(2)—Ag—S(2')	104.4 (1)	S(1')—Ag—S(2)	100.9 (1)
N(1)—Zn—N(1 ⁱⁱⁱ)	109.5 (1)	N(1)—Zn—N(2 ⁱⁱ)	119.5 (1)
N(1)—Zn—N(2')	104.1 (1)	N(2 ⁱⁱ)—Zn—N(2)	100.8 (1)
Ag—S(1)—C(1)	108.7 (1)	Ag—S(1)—Ag ^{iv}	113.0 (1)
C(1)—S(1)—Ag ^{iv}	100.7 (1)	Ag—S(2)—C(2)	96.5 (1)
Ag—S(2)—Ag ^v	103.1 (1)	C(2)—S(2)—Ag ^v	96.6 (1)
Zn—N(1)—C(1)	165.1 (2)	C(2)—N(2)—Zn ^{vi}	154.6 (2)
S(1)—C(1)—N(1)	177.6 (2)	S(2)—C(2)—N(2)	179.3 (2)

Symmetry operators: (i) $0.5 - x, 0.5 + y, 0.5 - z$; (ii) $0.5 + x, 0.5 + y, z$; (iii) $1 - x, y, 0.5 - z$; (iv) $0.5 - x, -0.5 + y, 0.5 - z$; (v) $x, -y, 0.5 + z$; (vi) $-0.5 + x, -0.5 + y, z$.

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Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55067 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1006]

References

- Ermer, O. & Dunitz, J. D. (1970). *Acta Cryst.* **A26**, 163.
 Herbst-Irmer, R. (1990). PhD thesis, Göttingen Univ., Germany.
 Roesky, H. W., Schimkowiak, J., Meyer-Bäse, K. & Jones, P. G. (1986). *Angew. Chem.* **98**, 998–999.
 Shaofang, L., Meiyun, H. & Jinling, H. (1982). *J. Struct. Chem. (China)*, **1**, 71–75.

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2-(*tert*-Butyl)-*cis*-5-(*p*-chlorophenylthio)-*trans*-5-methyl-1,3-dioxolan-4-one

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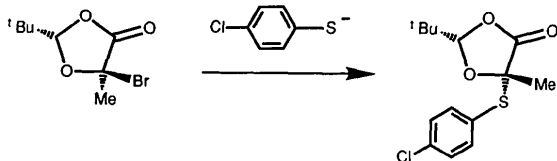
Abstract

The five-membered ring has a conformation intermediate between a twist form and an envelope with O1 deviating from the plane of the other four atoms. The *tert*-butyl and *p*-chlorophenylthio groups are both situated on the same side of the ring.

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Comment

The assignment of stereochemistry to small heterocyclic compounds is not trivial as NMR techniques, such as nuclear Overhauser enhancement methods, are not always reliable. The title compound was obtained from the corresponding *trans*-bromo derivative in a stereospecific reaction with the 4-chlorophenylthiolate ion, as part of a study of the diastereoselectivities of free-radical reactions (Beckwith & Chai, 1990; Chai, 1990).



This X-ray structure determination establishes that the *tert*-butyl and *p*-chlorophenylthio groups are *cis* in the product. Cremer & Pople puckering parameters (Cremer & Pople, 1975; Cremer, 1988) for the 1,3-dioxolan-4-one ring are $q_2 = 0.301 \text{ \AA}$ and $\varphi_2 = 188.4^\circ$, corresponding to a conformation intermediate between an envelope ($\varphi_2 = 180^\circ$) and a twist form ($\varphi_2 = 198^\circ$) (Cremer, 1980). The O3–C4 distance of $1.341(3) \text{ \AA}$ is significantly shorter than the other O–C distances within the ring, suggesting that it possesses some double-bond character arising from electron delocalization from the carbonyl centre towards the adjacent ether O. This is also consistent with the observation that C2–O3–C4–C5 has the smallest magnitude of the intra-annular torsion angles. Short O3–C4 bonds and small C2–O3–C4–C5 torsion angles are observed for all 1,3-dioxolan-4-one derivatives found in the July 1991 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983). *CNDO/2* calculations (Polonski, 1983) predicted an envelope conformation with O1 out-of-plane for *cis* 2,5-disubstituted species. Also of interest is the difference in bond lengths at the acetal centre, C2–O1 [$1.416(3) \text{ \AA}$] and C2–O3 [$1.457(3) \text{ \AA}$], which may indicate that the former is a stronger bond (Jones

& Kirby, 1979). We have previously reported structures of two 1,3-oxazolidin-5-one derivatives, corresponding to replacement of O1 by $\text{NCOCH}_2\text{C}_6\text{H}_5$ (Willis, Beckwith & Tozer, 1991) and $\text{NCOCH}_2\text{C}_6\text{H}_5$ (Beckwith, Chai & Willis, 1992). In the former $q_2 = 0.109 \text{ \AA}$ and $\varphi_2 = 26.6^\circ$, while in the latter $q_2 = 0.107 \text{ \AA}$ and $\varphi_2 = 75.5^\circ$. The q_2 values and the torsion angles indicate that those rings are less puckered than that of the present compound. Diagrams and most calculations were performed with the *Xtal3.0* package (Hall & Stewart, 1990), as was the generation of the Crystallographic Information File used for the submission of this paper.

Experimental

Crystal data

$\text{C}_{14}\text{H}_{17}\text{ClO}_3\text{S}$
 $M_r = 300.80$
 Monoclinic
C2/c
 $a = 28.795(3) \text{ \AA}$
 $b = 8.887(1) \text{ \AA}$
 $c = 12.139(1) \text{ \AA}$
 $\beta = 103.17(1)^\circ$
 $V = 3024.7(5) \text{ \AA}^3$
 $Z = 8$

Cell parameters from 25 reflections
 $\theta = 18\text{--}22^\circ$
 $D_x = 1.321 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{ \AA}$
 $\mu = 0.39 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 $0.29 \times 0.23 \times 0.20 \text{ mm}$
 Colourless

Data collection

Philips PW1100/20 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: analytical
 $T_{\min} = 0.9167$, $T_{\max} = 0.9379$
 2831 measured reflections
 2687 independent reflections

1780 observed reflections
 $[I_{\text{net}} > 3.00\sigma(I_{\text{net}})]$
 $R_{\text{int}} = 0.012$
 $\theta_{\max} = 25.05^\circ$
 $h = -34 \rightarrow 33$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 14$
 3 standard reflections
 frequency: 90 min
 intensity variation: 3%

Refinement

Refinement on F
 Final $R = 0.031$
 $wR = 0.039$
 $S = 1.311$
 1780 reflections
 224 parameters
 Only coordinates of H atoms refined
 Calculated weights
 $w = 1/[\sigma^2(F) + 0.0004F^2]$
 $(\Delta/\sigma)_{\max} = 0.013$

$\Delta\rho_{\max} = 0.155 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.211 \text{ e \AA}^{-3}$
 Extinction correction: Zachariasen
 Extinction coefficient: $1.4(1) \times 10^4$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Data collection: Philips PW1100/20 software 1976. Cell refinement: Philips PW1100/20 software 1976. Data reduction: *Xtal ADDREF*, *ABSORB*, *SORTRF* (Hall & Stewart, 1990). Program(s) used to solve structure: *Xtal SIMPEL* (Hall & Stewart, 1990). Program(s) used to refine structure: *Xtal CRYLSQ* (Hall & Stewart, 1990). Molecular graphics: *Xtal* (Hall & Stewart, 1990). Software used to prepare material for publication: *Xtal BONDLA*, *CIFIO* (Hall & Stewart, 1990).

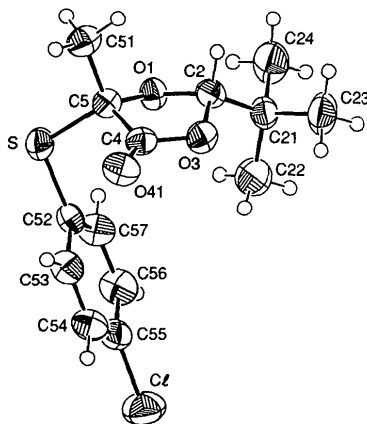


Fig. 1. View of $\text{C}_{14}\text{H}_{17}\text{ClO}_3\text{S}$ showing the labelling of the non-H atoms. The ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radius.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Cl	-0.02232 (3)	0.16553 (9)	0.58320 (7)	0.0787 (5)
S	0.12984 (2)	0.57086 (7)	0.43145 (5)	0.0464 (3)
O1	0.15402 (5)	0.3421 (2)	0.3127 (1)	0.0396 (8)
C2	0.17879 (8)	0.2036 (3)	0.3331 (2)	0.041 (1)
O3	0.18959 (5)	0.1880 (2)	0.4558 (1)	0.0455 (8)
C4	0.18986 (7)	0.3252 (3)	0.5021 (2)	0.040 (1)
C5	0.17392 (8)	0.4379 (2)	0.4064 (2)	0.038 (1)
C21	0.14997 (9)	0.0721 (3)	0.2749 (2)	0.047 (1)
C22	0.1023 (1)	0.0592 (4)	0.3078 (3)	0.072 (2)
C23	0.1796 (1)	-0.0704 (3)	0.3086 (3)	0.071 (2)
C24	0.1428 (1)	0.0967 (4)	0.1480 (3)	0.074 (2)
O41	0.20213 (6)	0.3479 (2)	0.6013 (1)	0.054 (1)
C51	0.2163 (1)	0.5277 (3)	0.3859 (3)	0.052 (1)
C52	0.08744 (7)	0.4531 (2)	0.4744 (2)	0.041 (1)
C53	0.09069 (9)	0.4234 (3)	0.5882 (2)	0.049 (1)
C54	0.05714 (9)	0.3352 (3)	0.6215 (2)	0.053 (1)
C55	0.02014 (8)	0.2766 (3)	0.5413 (2)	0.049 (1)
C56	0.01597 (9)	0.3066 (3)	0.4283 (2)	0.060 (2)
C57	0.04937 (9)	0.3951 (3)	0.3951 (2)	0.052 (1)

S—C5—O1	110.7 (1)	C53—C54—C55	119.5 (2)
S—C5—C4	113.7 (2)	Cl—C55—C54	119.6 (2)
S—C5—C51	107.6 (2)	Cl—C55—C56	119.7 (2)
O1—C5—C4	102.2 (2)	C54—C55—C56	120.7 (3)
O1—C5—C51	111.9 (2)	C55—C56—C57	119.8 (2)
C4—C5—C51	110.8 (2)	C52—C57—C56	120.4 (2)
C2—C21—C22	111.6 (2)		
C5—O1—C2—O3	33.2 (2)	C2—O3—C4—C5	5.5 (2)
C2—O1—C5—C4	-29.3 (2)	O3—C4—C5—O1	14.5 (2)
O1—C2—O3—C4	-23.8 (2)		

The θ -scan width was $(1.0 + 0.346 \tan \theta)^\circ$ with a θ -scan rate of $1.5^\circ \text{ min}^{-1}$ and background counts for 10 s on each end of every scan. Refinement was by full-matrix least-squares methods.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55084 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1010]

Table 2. Geometric parameters (\AA , $^\circ$)

Cl—C55	1.735 (3)	C5—C51	1.524 (4)
S—C5	1.810 (2)	C21—C22	1.519 (5)
S—C52	1.775 (2)	C21—C23	1.530 (4)
O1—C2	1.416 (3)	C21—C24	1.522 (4)
O1—C5	1.432 (2)	C52—C53	1.389 (3)
C2—O3	1.457 (3)	C52—C57	1.383 (3)
C2—C21	1.512 (3)	C53—C54	1.374 (4)
O3—C4	1.341 (3)	C54—C55	1.371 (3)
C4—C5	1.524 (3)	C55—C56	1.375 (4)
C4—O41	1.192 (3)	C56—C57	1.373 (4)
C5—S—C52	102.7 (1)	C2—C21—C23	107.6 (2)
C2—O1—C5	106.5 (1)	C2—C21—C24	107.2 (2)
O1—C2—O3	104.1 (2)	C22—C21—C23	110.4 (3)
O1—C2—C21	112.9 (2)	C22—C21—C24	110.7 (3)
O3—C2—C21	111.8 (2)	C23—C21—C24	109.2 (3)
C2—O3—C4	108.8 (2)	S—C52—C53	120.6 (2)
O3—C4—C5	107.8 (2)	S—C52—C57	120.3 (2)
O3—C4—O41	123.3 (2)	C53—C52—C57	119.0 (2)
C5—C4—O41	128.9 (2)	C52—C53—C54	120.5 (2)

References

- Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
- Beckwith, A. L. J. & Chai, C. L. L. (1990). *J. Chem. Soc. Chem. Commun.* pp. 1087–1088.
- Beckwith, A. L. J., Chai, C. L. L. & Willis, A. C. (1992). *Acta Cryst.* **C48**, 593–594.
- Chai, C. L. L. (1990). PhD thesis, Australian National Univ., Australia.
- Cremer, D. (1980). *Isr. J. Chem.* **20**, 12–19.
- Cremer, D. (1988). *RING88*. Univ. of Göteborg, Sweden.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual*. Univs. of Western Australia, Australia, and Maryland, USA.
- Jones, P. G. & Kirby, A. J. (1979). *J. Chem. Soc. Chem. Commun.* pp. 288–289.
- Polonski, T. (1983). *Tetrahedron*, **39**, 3131–3137.
- Willis, A. C., Beckwith, A. L. J. & Tozer, M. J. (1991). *Acta Cryst.* **C47**, 2276–2277.