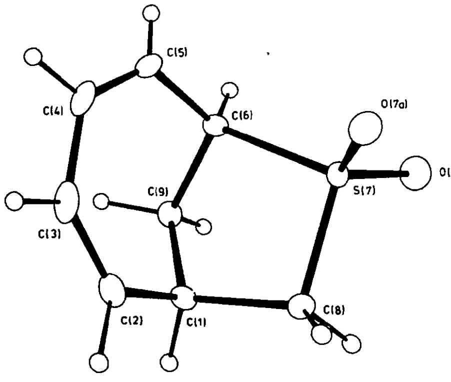


Fig. 1. A view of the molecular structure showing the atom labels.

scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Atomic coordinates and bond lengths are in Tables 1 and 2.* Fig. 1 shows the atom labels and Fig. 2 is a stereoview of the crystal packing.

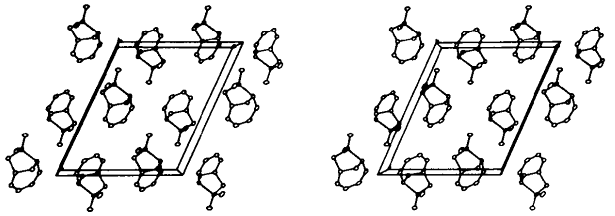
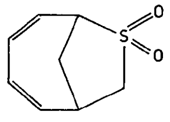


Fig. 2. A stereoview of the unit cell looking along the *b* axis. The *a* axis is across and the *c* axis down the page.

Related literature. The sulfone (I) was prepared in a novel reaction involving cyclopropane ring opening from 3,4-homotropilidene (bicyclo[5.1.0]octa-2,5-diene) as previously described (Dalling, Gall & MacNicol, 1979). (I) is also formed in high yield when the isomeric hydrocarbon bicyclo[5.1.0]octa-2,4-diene is heated with excess dry SO_2 in toluene-d_8. Interestingly, in this case ^1H NMR monitoring (unpublished results) shows the facile production of (I), even at room temperature.



(I)

References

Dalling, J., Gall, J. H. & MacNicol, D. D. (1979). *Tetrahedron Lett.* pp. 4789–4790.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

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* Lists of structure factors, H-atom coordinates and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51261 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (*R*)-3-[(*R*)-(6,7-Dichloro-2,3-dihydrobenzofuran-2-yl)carbonyl]-4-phenyl-2-oxazolidone

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Abstract. $C_{18}H_{13}Cl_2NO_4$, $M_r = 378{\cdot}21$, orthorhombic, $P2_12_12_1$, $a = 13{\cdot}479\,(2)$, $b = 24{\cdot}885\,(3)$, $c = 4{\cdot}977\,(1)$ Å, $V = 1669{\cdot}5\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}505$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 3{\cdot}71$ mm^{-1}, $F(000) = 776$, $T = 295$ K, $R = 0{\cdot}046$ for 1742 observed reflections [$F_o > 3\sigma(F_o)$]. The absolute configuration of C(2) was determined as *R* based on the configuration of the (*R*)-4-phenyl-2-oxazolidone portion which was already known. All bond lengths and angles are normal.

Experimental. Colorless prism crystals obtained from dioxane. Crystal of dimensions 0·4 × 0·4 × 0·5 mm. Rigaku AFC-5R diffractometer, graphite-monochro-

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å²) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
O(1)	0·9425 (1)	0·3477 (1)	−0·0885 (5)	3·96 (4)
C(2)	1·0324 (2)	0·3757 (1)	−0·1605 (6)	3·57 (6)
C(3)	1·1200 (2)	0·3464 (1)	−0·0230 (7)	4·07 (6)
C(4)	1·1010 (2)	0·2681 (1)	0·3320 (7)	4·12 (6)
C(5)	1·0347 (2)	0·2330 (1)	0·4485 (8)	4·81 (8)
C(6)	0·9350 (2)	0·2352 (1)	0·3791 (8)	4·40 (7)
C(7)	0·8983 (2)	0·2735 (1)	0·2014 (6)	3·66 (6)
C(8)	0·9666 (2)	0·3088 (1)	0·0919 (6)	3·14 (5)
C(9)	1·0669 (2)	0·3059 (1)	0·1473 (6)	3·31 (5)
C(10)	1·0233 (2)	0·4331 (1)	−0·0517 (5)	3·02 (5)
O(11)	0·9789 (1)	0·4438 (1)	0·1494 (4)	3·81 (4)
N(12)	1·0696 (1)	0·4735 (1)	−0·1988 (4)	2·76 (4)
C(13)	1·0572 (2)	0·5307 (1)	−0·1250 (4)	2·82 (4)
C(14)	1·1224 (2)	0·5570 (1)	−0·3407 (6)	3·66 (5)
O(15)	1·1757 (1)	0·5150 (1)	−0·4726 (4)	3·91 (4)
C(16)	1·1439 (2)	0·4667 (1)	−0·3883 (5)	3·39 (5)
O(17)	1·1784 (2)	0·4251 (1)	−0·4676 (5)	4·82 (5)
C(18)	0·9506 (2)	0·5494 (1)	−0·1283 (5)	2·92 (4)
C(19)	0·8822 (2)	0·5306 (1)	−0·3166 (5)	3·84 (6)
C(20)	0·7858 (2)	0·5500 (1)	−0·3161 (7)	4·65 (7)
C(21)	0·7556 (2)	0·5879 (1)	−0·1320 (8)	4·64 (7)
C(22)	0·8233 (3)	0·6071 (1)	0·0531 (7)	4·86 (8)
C(23)	0·9197 (2)	0·5877 (1)	0·0541 (6)	4·01 (6)
Cl(24)	0·77556 (4)	0·27798 (3)	0·1164 (2)	4·81 (2)
Cl(25)	0·85513 (7)	0·18738 (3)	0·5134 (3)	7·62 (4)

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

O(1)—C(2)	1·443 (4)	C(10)—N(12)	1·391 (4)
O(1)—C(8)	1·360 (4)	N(12)—C(13)	1·480 (4)
C(2)—C(3)	1·547 (5)	N(12)—C(16)	1·386 (4)
C(2)—C(10)	1·533 (4)	C(13)—C(14)	1·534 (4)
C(3)—C(9)	1·499 (5)	C(13)—C(18)	1·510 (4)
C(4)—C(5)	1·378 (5)	C(14)—O(15)	1·428 (4)
C(4)—C(9)	1·393 (5)	O(15)—C(16)	1·343 (4)
C(5)—C(6)	1·389 (6)	C(16)—O(17)	1·202 (4)
C(6)—C(7)	1·391 (5)	C(18)—C(19)	1·395 (4)
C(6)—Cl(25)	1·739 (4)	C(18)—C(23)	1·381 (4)
C(7)—C(8)	1·384 (4)	C(19)—C(20)	1·386 (4)
C(7)—Cl(24)	1·711 (3)	C(20)—C(21)	1·377 (5)
C(8)—C(9)	1·382 (4)	C(21)—C(22)	1·382 (6)
C(10)—O(11)	1·196 (4)	C(22)—C(23)	1·386 (5)

C(2)—O(1)—C(8)	107·9 (2)	O(11)—C(10)—N(12)	120·3 (3)
O(1)—C(2)—C(3)	107·7 (2)	C(10)—N(12)—C(13)	120·9 (2)
O(1)—C(2)—C(10)	107·2 (2)	C(10)—N(12)—C(16)	126·4 (2)
C(3)—C(2)—C(10)	110·1 (2)	C(13)—N(12)—C(16)	111·6 (2)
C(2)—C(3)—C(9)	101·7 (3)	N(12)—C(13)—C(14)	99·9 (2)
C(5)—C(4)—C(9)	119·5 (3)	N(12)—C(13)—C(18)	113·7 (2)
C(4)—C(5)—C(6)	119·9 (4)	C(14)—C(13)—C(18)	114·0 (2)
C(5)—C(6)—C(7)	122·0 (4)	C(13)—C(14)—O(15)	107·3 (2)
C(5)—C(6)—Cl(25)	118·5 (3)	C(14)—O(15)—C(16)	110·5 (2)
C(7)—C(6)—Cl(25)	119·5 (3)	N(12)—C(16)—O(15)	109·5 (2)
C(6)—C(7)—C(8)	116·7 (3)	N(12)—C(16)—O(17)	127·5 (3)
C(6)—C(7)—Cl(24)	123·1 (3)	O(15)—C(16)—O(17)	123·0 (3)
C(8)—C(7)—Cl(24)	120·3 (2)	C(13)—C(18)—C(19)	122·2 (2)
O(1)—C(8)—C(7)	123·6 (3)	C(13)—C(18)—C(23)	119·5 (2)
O(1)—C(8)—C(9)	113·7 (3)	C(19)—C(18)—C(23)	118·3 (3)
C(7)—C(8)—C(9)	122·6 (3)	C(18)—C(19)—C(20)	120·1 (3)
C(3)—C(9)—C(4)	132·0 (3)	C(19)—C(20)—C(21)	121·1 (3)
C(3)—C(9)—C(8)	108·6 (3)	C(20)—C(21)—C(22)	119·0 (4)
C(4)—C(9)—C(8)	119·3 (3)	C(21)—C(22)—C(23)	120·1 (4)
C(2)—C(10)—O(11)	122·9 (3)	C(18)—C(23)—C(22)	121·4 (3)
C(2)—C(10)—N(12)	116·8 (2)		

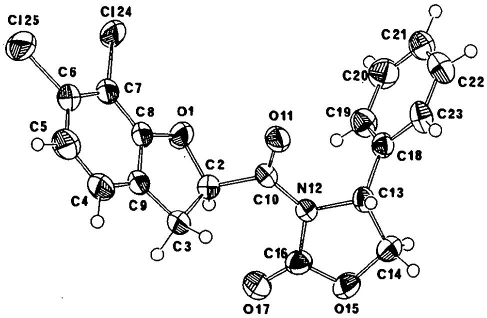

Fig. 1. *ORTEP* drawing (Davenport & Hall, 1987). C, N, O and Cl atoms are represented by thermal ellipsoids with 50% probability.

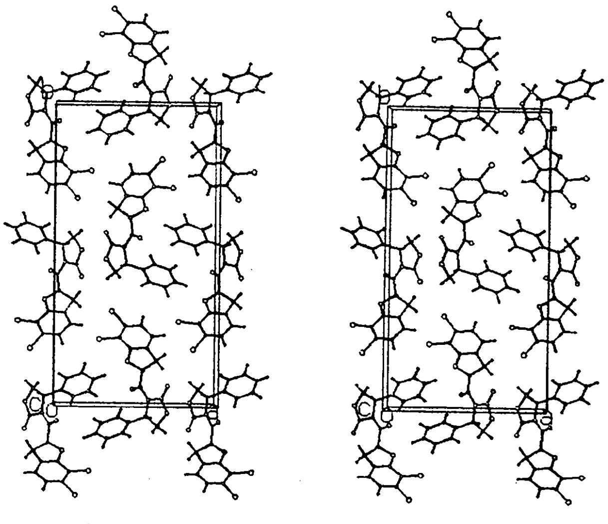

Fig. 2. A stereoscopic view of the unit-cell packing.

matized Cu *K*α. Cell dimensions determined from 2θ angles for 25 reflections in the range $41 < 2\theta < 54°$. Intensities measured up to $2\theta = 140°$ in *h* 0/16, *k* 0/29 and *l* 0/6, ω–2θ scans, ω-scan width (2·5 + 0·2tanθ)°, three standard reflections monitored every 100 measurements showed no significant change. 1790 unique reflections measured, 1742 intensities observed [$F_o > 3\sigma(F_o)$], no absorption corrections. Structure solved by *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to $B_{\rm eq}$ of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00349|F_o|^2]$, $w = 0$ for 63 reflections with $w^{1/2}|\Delta F| \geq 3$. Final $R = 0{\cdot}046$, $wR = 0{\cdot}058$, $S = 0{\cdot}99679$. The highest and lowest peaks in final difference map 0·3 and −0·3 e Å^{-3}. Max. Δ/σ in the final cycle 0·3. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ $(i = 1,\ldots,4)$ (*International Tables for X-ray Crystallography*, 1974). Calculations performed on a FACOM M340R computer at Shionogi Research Laboratories. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the molecule with the atom-numbering system and a stereoscopic view of the crystal packing drawn using the program *PLUTO* (Motherwell & Clegg, 1978) are presented in Figs. 1 and 2, respectively.

Related literature. The absolute configuration of the title compound reported here has been referred to by Yodo, Matsushita, Ohsugi & Harada (1988).

The author thanks Dr Ohsugi for the supply of the crystals.

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms and atomic coordinates of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51281 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Davenport, G. & Hall, S. R. (1987). *ORTEP. XTAL*2.2 *User's Manual,* edited by S. R. Hall & J. M. Stewart. Univs. of Western Australia, Australia, and Maryland, USA.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Main, P., Germain, G. & Woolfson, M. M. (1984). *MULTAN*84. *A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Yodo, M., Matsushita, Y., Ohsugi, E. & Harada, H. (1988). *Chem. Pharm. Bull.* **36**, 902–913.

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Structure of Bis[(*N*-methyl-2-imidazolyl)methyl]amine

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Abstract. $C_{10}H_{15}N_5$, $M_r = 205{\cdot}26$, triclinic, $P\bar{1}$, $a = 8{\cdot}364\,(1)$, $b = 9{\cdot}148\,(1)$, $c = 7{\cdot}350\,(2)$ Å, $\alpha = 99{\cdot}92\,(2)$, $\beta = 95{\cdot}28\,(2)$, $\gamma = 94{\cdot}16\,(1)°$, $V = 549{\cdot}4\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}24$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 220$, $T = 295$ K, final $R = 0{\cdot}042$ for 1516 observed reflections. The structure of the title compound has been examined by X-ray crystallography. The compound was prepared by a nine-step synthetic procedure starting with *N*-methylimidazole and has been used to prepare biologically relevant binucleating ligands. Both *N*-methylimidazolyl moieties are planar and twisted 71·3° relative to their individual molecular least-squares planes. There are no significant intermolecular interactions and the C—C and C—N bond lengths appear to be normal.

Experimental. Cut arrowhead-shaped colorless crystal, 0·30 × 0·40 × 0·50 mm, Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation, unit-cell parameters from least-squares refinement of 25 reflections with $12 < \theta < 19{\cdot}5°$, space group $P\bar{1}$ determined from intensity data and successful solution and refinement of the structure; 2200 data collected, 1915 unique reflections, 1516 observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics]; $\theta_{\max} = 25°$, scan range $(0{\cdot}8 + 0{\cdot}344\tan\theta)°$, $\omega/2\theta$ scans, variable scan speed 1–3° min^{-1}; three standard reflections measured every 3600 s of X-ray exposure time, variation −6·7%, linear decay correction applied; data collected $\pm h$, $\pm k$, $-l$ to max. indices of 9, 10, 8. Data corrected for background and Lp; empirical absorption correction based on a series of ψ scans, relative transmission coefficients ranging from 0·977 to 1·000 with average value of 0·987; secondary-extinction correction applied (Zachariasen, 1963), final coefficient refined in least squares was $8{\cdot}8\,(4) \times 10^{-6}$; intensities of equivalent reflections averaged, agreement factors for averaging of 275 observed and accepted reflections was 0·6% based on I. Structure solved by direct methods, all non-H located from E map, model refined by full-matrix least squares based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$; w defined as $[\sigma^2(F_o) + (0{\cdot}15F_o)^2 + 3{\cdot}0]^{-1}$, *SDP*/VAX package of programs

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