

Table 2. Bond distances (Å) and angles (°)

C2—C1	1.534 (3)	C6—C1	1.520 (3)
C7—C1	1.511 (3)	C18—C1	1.745 (2)
N3—C2	1.468 (3)	C14—C2	1.513 (3)
C4—N3	1.336 (3)	C5—C4	1.525 (3)
O13—C4	1.230 (3)	C6—C5	1.532 (3)
C12—C5	1.522 (3)	C7—C6	1.521 (3)
C11—C6	1.750 (2)	C19—C7	1.757 (2)
C10—C7	1.743 (2)	C15—C14	1.392 (3)
C19—C14	1.383 (3)	C16—C15	1.379 (4)
C17—C16	1.374 (4)	C18—C17	1.365 (4)
C19—C18	1.389 (4)		
C6—C1—C2	112.3 (2)	C7—C1—C2	121.3 (2)
C7—C1—C6	60.3 (1)	C18—C1—C2	114.4 (2)
C18—C1—C6	119.4 (2)	C18—C1—C7	118.1 (2)
N3—C2—C1	106.0 (2)	C14—C2—C1	115.0 (2)
C14—C2—N3	113.7 (2)	C4—N3—C2	119.9 (2)
C5—C4—N3	115.0 (2)	O13—C4—N3	123.4 (2)
O13—C4—C5	121.6 (2)	C6—C5—C4	108.0 (2)
C12—C5—C4	112.4 (2)	C12—C5—C6	115.7 (2)
C5—C6—C1	113.7 (2)	C7—C6—C1	59.6 (1)
C7—C6—C5	121.8 (2)	C11—C6—C1	119.0 (2)
C11—C6—C5	114.5 (2)	C11—C6—C7	117.2 (2)
C19—C7—C1	119.8 (2)	C19—C7—C6	120.0 (2)
C19—C7—C10	110.2 (1)	C10—C7—C1	119.2 (2)
C6—C7—C1	60.2 (1)	C15—C14—C2	118.1 (2)
C10—C7—C6	119.6 (2)	C19—C14—C15	118.6 (2)
C19—C14—C2	123.2 (2)	C17—C16—C15	120.4 (2)
C16—C15—C14	120.3 (3)	C19—C18—C17	120.5 (3)
C18—C17—C16	119.8 (3)		
C18—C19—C14	120.3 (2)		

The atomic parameters are given in Table 1.* The bond lengths and angles are listed in Table 2. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978).

Related literature. Only one structure containing the 3-azabicyclo[4.1.0]heptan-4-one skeleton is classified

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54011 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Model for Spirochlorine (Antibiotic A30641)

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Abstract. 2*H*-5*a*,10*a*-Dihydro-10*a*,3-(iminomethano)-11-methoxy-2(4-methoxybenzyl)-1-benzofuran-

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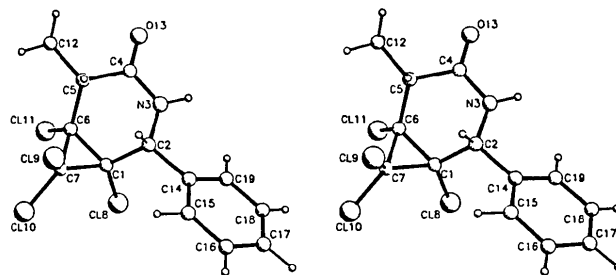


Fig. 1. Stereoscopic view of the molecule.

in the Cambridge Structural Database (Spek, 1978; Allen, Kennard & Taylor, 1983).

Substituent-induced bond-length asymmetry in cyclopropane derivatives has been extensively analysed (Allen, 1980; Schruppf & Jones, 1987). For chloro-substituted cyclopropanes, a lengthening of the distal bond is predicted. The geometry observed for the title derivative does not follow this expectation since we observe a symmetrical three-membered ring with C—C distances of 1.520 (3), 1.511 (3) and 1.521 (3) Å.

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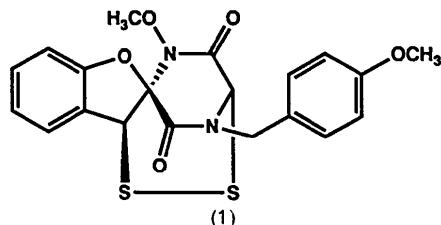
Table 1. Atomic coordinates and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) for (1)

	x	y	z	U
S(1)	0.0244 (2)	0.0800 (1)	-0.2686 (1)	27 (1)*
S(2)	0.0661 (2)	0.1775 (1)	-0.3097 (1)	25 (1)*
C(1)	0.1310 (7)	0.0801 (3)	-0.0435 (5)	26 (2)*
C(2)	-0.0286 (8)	0.0879 (3)	-0.1166 (5)	24 (2)*
C(3)	-0.0356 (7)	0.2066 (3)	-0.0862 (4)	20 (2)*
C(4)	0.1535 (7)	0.1995 (3)	-0.0786 (5)	20 (2)*
C(5)	0.2234 (7)	0.1987 (3)	-0.1974 (5)	22 (1)
C(6)	0.2827 (7)	0.2695 (3)	-0.2057 (5)	22 (2)*
C(7)	0.2762 (7)	0.2994 (3)	-0.0988 (5)	23 (2)*
C(8)	0.3194 (7)	0.3646 (3)	-0.0769 (5)	26 (2)*
C(9)	0.3784 (8)	0.3995 (3)	-0.1686 (6)	35 (2)*
C(10)	0.3890 (8)	0.3710 (3)	-0.2756 (6)	32 (2)*
C(11)	0.3437 (8)	0.3050 (3)	-0.2938 (5)	32 (2)*
C(12)	0.3805 (9)	0.1443 (3)	-0.1523 (5)	35 (2)*
C(13)	-0.2911 (7)	0.1446 (3)	-0.0843 (5)	28 (2)*
C(14)	-0.3842 (7)	0.1150 (3)	-0.1860 (5)	22 (2)*
C(15)	-0.4026 (7)	0.1482 (3)	-0.2894 (5)	24 (2)*
C(16)	-0.4974 (7)	0.1224 (3)	-0.3827 (5)	25 (2)*
C(17)	-0.5781 (7)	0.0615 (3)	-0.3702 (5)	25 (2)*
C(18)	-0.5590 (8)	0.0276 (3)	-0.2693 (6)	32 (2)*
C(19)	-0.4631 (7)	0.0537 (3)	-0.1762 (5)	24 (2)*
C(20)	-0.6978 (9)	0.0665 (4)	-0.5626 (6)	43 (3)*
N(1)	0.1968 (5)	0.1409 (2)	-0.0079 (4)	21 (2)*
N(2)	-0.1147 (6)	0.1481 (2)	-0.0926 (4)	21 (2)*
O(1)	0.1886 (5)	0.0263 (2)	-0.0177 (4)	34 (2)*
O(2)	-0.0968 (5)	0.2620 (2)	-0.0798 (3)	27 (1)*
O(3)	0.2177 (5)	0.2566 (2)	-0.0193 (3)	26 (1)*
O(4)	0.3618 (5)	0.1382 (2)	-0.0296 (3)	27 (1)*
O(5)	-0.6756 (5)	0.0323 (2)	-0.4553 (4)	35 (2)*

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

1.5418 \AA , $\mu = 27.6 \text{ cm}^{-1}$, $F(000) = 896$, $T = 115 \text{ K}$, $R = 0.077$ ($wR = 0.090$) for 1884 unique, observed reflections. A very close $\text{C}(2)\text{---H}(2)\cdots\text{O}(1)^i$ contact links the molecules into dimers.

Experimental. Crystals (colorless prisms) of $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_5\text{S}_2$ [hereafter (1)] obtained from Greg Miknis and Professor Robert M. Williams (Colorado State University); the synthesis of this compound will be reported at a later date. Crystal size $0.16 \times 0.26 \times 0.28 \text{ mm}$. Nicolet R3m diffractometer, unit-cell constants from least-squares fit of setting angles for 25 reflections ($2\theta_{\text{av}} = 51.83^\circ$). Data collected ($\theta/2\theta$ scans) to $(\sin \theta)/\lambda = 0.5313 \text{ \AA}^{-1}$, $-9 \leq h \leq 9$, $0 \leq k \leq 22$, $0 \leq l \leq 13$. Temperature was controlled using a Siemens LT1 low-temperature apparatus. Three standard reflections (400, 040, 004) every 97, correction for changes in intensity ($< 15\%$); Lorentz and polarization corrections; no absorption correction applied; 2369 unique reflections, 1884 reflections with $F_o > 2.5\sigma(F_o)$ observed.

Table 2. Bond lengths (\AA) and angles ($^\circ$) for (1)

S(1)—S(2)	2.040 (2)	S(1)—C(2)	1.878 (6)
S(2)—C(5)	1.822 (6)	C(1)—C(2)	1.522 (8)
C(1)—N(1)	1.380 (7)	C(1)—O(1)	1.203 (7)
C(2)—N(2)	1.438 (7)	C(3)—C(4)	1.570 (8)
C(3)—N(2)	1.338 (7)	C(3)—O(2)	1.220 (7)
C(4)—C(5)	1.553 (8)	C(4)—N(1)	1.460 (7)
C(4)—O(3)	1.414 (7)	C(5)—C(6)	1.502 (8)
C(6)—C(7)	1.392 (8)	C(6)—C(11)	1.383 (9)
C(7)—C(8)	1.368 (8)	C(7)—O(3)	1.382 (7)
C(8)—C(9)	1.403 (9)	C(9)—C(10)	1.386 (9)
C(10)—C(11)	1.380 (9)	C(13)—C(14)	1.487 (8)
C(13)—N(2)	1.476 (8)	C(14)—C(15)	1.378 (8)
C(14)—C(19)	1.395 (8)	C(15)—C(16)	1.391 (8)
C(16)—C(17)	1.400 (8)	C(17)—C(18)	1.360 (9)
C(17)—O(5)	1.361 (7)	C(18)—C(19)	1.396 (8)
N(1)—O(4)	1.403 (6)	O(4)—C(12)	1.438 (7)
O(5)—C(20)	1.428 (8)		
S(2)—S(1)—C(2)	101.7 (2)	S(1)—S(2)—C(5)	100.1 (2)
C(2)—C(1)—N(1)	112.5 (5)	C(2)—C(1)—O(1)	122.7 (5)
N(1)—C(1)—O(1)	124.8 (5)	S(1)—C(2)—C(1)	105.2 (4)
S(1)—C(2)—N(2)	114.5 (4)	C(1)—C(2)—N(2)	113.5 (5)
C(4)—C(3)—N(2)	114.0 (5)	C(4)—C(3)—O(2)	119.8 (5)
N(2)—C(3)—O(2)	126.1 (5)	C(3)—C(4)—C(5)	113.4 (4)
C(3)—C(4)—N(1)	107.5 (4)	C(5)—C(4)—N(1)	114.0 (5)
C(3)—C(4)—O(3)	106.7 (4)	C(5)—C(4)—O(3)	107.2 (4)
N(1)—C(4)—O(3)	107.6 (4)	S(2)—C(5)—C(4)	110.6 (4)
S(2)—C(5)—C(6)	112.9 (4)	C(4)—C(5)—C(6)	101.3 (5)
C(5)—C(6)—C(7)	107.9 (5)	C(5)—C(6)—C(11)	132.3 (5)
C(7)—C(6)—C(11)	119.8 (5)	C(6)—C(7)—C(8)	123.2 (6)
C(6)—C(7)—O(3)	112.6 (5)	C(8)—C(7)—O(3)	124.5 (6)
C(7)—C(8)—C(9)	115.5 (6)	C(8)—C(9)—C(10)	122.8 (6)
C(9)—C(10)—C(11)	119.6 (6)	C(6)—C(11)—C(10)	119.0 (6)
C(14)—C(13)—N(2)	114.6 (5)	C(13)—C(14)—C(15)	121.6 (5)
C(13)—C(14)—C(19)	119.8 (5)	C(15)—C(14)—C(19)	118.5 (5)
C(14)—C(15)—C(16)	121.7 (6)	C(15)—C(16)—C(17)	118.9 (5)
C(16)—C(17)—C(18)	120.1 (5)	C(16)—C(17)—O(5)	123.7 (5)
C(18)—C(17)—O(5)	116.2 (5)	C(17)—C(18)—C(19)	120.7 (6)
C(14)—C(19)—C(18)	120.2 (5)	C(1)—N(1)—C(4)	117.4 (4)
C(1)—N(1)—O(4)	114.3 (4)	C(4)—N(1)—O(4)	113.2 (4)
C(2)—N(2)—C(3)	119.4 (5)	C(2)—N(2)—C(13)	119.0 (5)
C(3)—N(2)—C(13)	121.4 (5)	C(4)—O(3)—C(7)	107.8 (4)
C(12)—O(4)—N(1)	109.2 (4)	C(17)—O(5)—C(20)	117.7 (5)

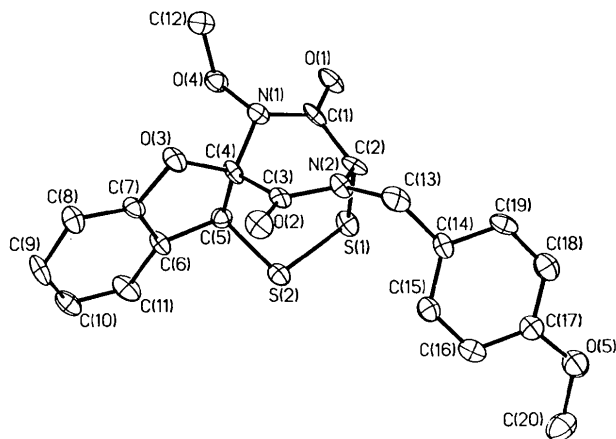


Fig. 1. The structure of (1) (50% probability thermal ellipsoids). H atoms have been omitted for clarity.

Structure solved by direct methods (*SOLV*) in $P2_1/n$ (variant of $P2_1/c$, No. 14); block-diagonal (max. 103 parameters/block, 263 parameters total, data/parameters = 7.2) weighted $\{w = [\sigma^2(F) + gF^2]^{-1}$, $g = 3.1 \times 10^{-3}$ least-squares refinement on

F. H atoms in idealized positions [$C-H = 0.96 \text{ \AA}$, $U(H) = 1.2 \times U_{iso}(C)$]. All non-H atoms [with the exception of C(5)] refined with anisotropic thermal parameters. Difficulty in refining C(5) anisotropically due to lack of absorption correction; necessary data unfortunately not collected before crystal removed from diffractometer. At convergence [$(\Delta/\sigma)_{max} = 0.009$, $(\Delta/\sigma)_{mean} = 0.002$ for the last three cycles] $R = 0.077$, $wR = 0.090$, $S = 1.20$, slope of normal probability plot = 1.07, $(\Delta\rho)_{max} = 0.70$, $(\Delta\rho)_{min} = -0.65 \text{ e \AA}^{-3}$. Neutral-atom scattering factors and anomalous-dispersion corrections used (*International Tables for X-ray Crystallography*, 1974, Vol. IV); all calculations performed on a Data General Eclipse S/140 computer using *SHELXTL* program library (Sheldrick, 1983). Table 1 gives atomic coordinates, and Table 2 gives bond lengths and angles.* Fig. 1

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54037 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

shows the structure of (1), as well as the numbering scheme used.

Related literature. The structure of *O,O'*-dimethylaspirochlorine has been previously studied (Sakata, Maruyama, Uzawa, Sakurai, Lu & Clardy, 1987). The structure of (1) and *O,O'*-dimethylaspirochlorine are similar with the exception of different substituents on the main three-ring system. In the structure of (1), a very close $C(2)-H(2)\cdots O(1)^i$ contact exists linking molecules of (1) into dimers [$C(2)-O(1)^i$, 3.126, $H(2)-O(1)^i$, 2.255 \AA].

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Mesogenic 4-Methoxyphenyl 4-Hexyloxybenzoate

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Abstract. $C_{20}H_{24}O_4$, $M_r = 328.4$, monoclinic, $P2_1/c$, $a = 16.211$ (4), $b = 5.457$ (2), $c = 21.688$ (5) \AA, $\beta = 109.41$ (1)°, $V = 1809.23 \text{ \AA}^3$, $Z = 4$, $D_x = 1.205 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 0.48 \text{ cm}^{-1}$, $F(000) = 704$, $T = 298 \text{ K}$, $R = 0.0480$ for 2094 independent reflections with $|F_o| > 2\sigma(F_o)$. The compound is fully extended with an all-*trans* conformation of the hexyloxy group. The phenyl ring of the benzoate group forms an interplanar angle of 66.1° with the phenyl ring of the methoxyphenyl group. The dihedral angle between the best planes through the hexyloxy group and the attached phenyl group is 3.1° . In the benzoate group the dihedral angle

between the best planes through the phenyl group and carboxylate group is 7.4° . In the crystalline state the molecules form layers parallel to (102).

Experimental. The title compound was prepared by Professor Finkelmann, Freiburg. Colorless prismatic crystals were obtained by evaporation of an acetone–water solution; dimensions $0.22 \times 0.27 \times 30 \text{ mm}$. Stoe–Siemens AED2 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan, cell parameters by least squares on 58 reflections ($13.1 \leq 2\theta \leq 43.8^\circ$). Analytical absorption correction, maximum and minimum transmission factors 0.766 and 0.862. Total of 4584 reflections ($3 < 2\theta < 45^\circ$) measured in the range $0 < h < 17$, $-5 < k < 5$, $-23 < l < 22$. Standard reflections 011, 006, $\bar{5}04$ measured every

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