intermolecular hydrogen bond. The bond [2·884 (7) Å] is formed between N(3) of one molecule and O(4) of another related by the symmetry operation ($\frac{1}{2}-x$, $2-y$, $\frac{1}{2}+z$). There is no base-pairing of the uracil bases.

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Structure of Methyl 3,3-Dimethyl-7-phenylsulfinyl-1,5-dioxaspiro[5.5]undecane-9-carboxylate, $C_{19}H_{26}O_5S$*

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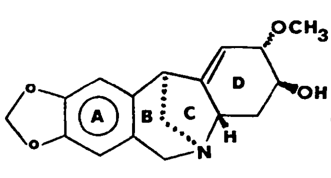
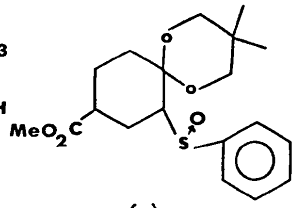
Abstract. $M_r = 366{\cdot}5$, monoclinic, $P2_1/a$, $a = 11{\cdot}094\ (1)$, $b = 11{\cdot}324\ (2)$, $c = 15{\cdot}298\ (2)$ Å, $\beta = 96{\cdot}22\ (1)°$, $V = 1911\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}27$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 1{\cdot}62$ mm^{-1}, $F(000) = 784$, $T = 293$ K. Final $R = 0{\cdot}075$ for 1496 observed reflections. Both the cyclohexane and 1,3-dioxane rings are in chair conformations. The angle between the mean planes of the methoxycarbonyl group and the 1,3-dioxane ring is 93·8 (5)° so that the molecule forms a spiral-like structure. The packing in the crystal is entirely due to van der Waals forces.

Introduction. As part of our studies (Sánchez, López, Flores & Larraza, 1983) regarding the total synthesis of natural products, we became interested in designing a new synthetic entry into the 5,11-methanomorfanthridine-like Amaryllidaceae alkaloids (Wildman, 1970), as shown by montanine (I). Careful analysis of the structural features characteristic of such alkaloids suggested the utilization of (II) (GG-O) as a versatile synthon for the oxygenated *D* ring (Garcia, Larraza & Sánchez, 1983). We now report the crystal and molecular structure of (II).



(I)



(II)

Experimental. Prismatic crystal 0·12 × 0·20 × 0·20 mm, Nicolet *R*3 four-circle diffractometer, graphite-monochromated Cu *Kα*, lattice parameters from 20 machine-centered reflections with $9{\cdot}7 < 2\theta < 24{\cdot}3°$; 2392 reflections with $3 < 2\theta < 115°$ for two octants, 1496 independent with $I > 2{\cdot}5\sigma(I)$, index range $h \pm 11$, k 0/12, l 0/15, ω-scan mode, variable scan speed, scan width 1·0° (θ), two standard reflections monitored every 50 measurements, Lp correction, absorption ignored; structure solved by direct methods using *SHELXTL* (Sheldrick, 1981); least-squares refinement of all non-H atoms treated anisotropically, H atoms riding on the bonded C with a fixed isotropic temperature factor $U = 0{\cdot}06$ Å^2, function minimized $\sum w(\Delta F)^2$, $w = [\sigma^2(F_o) + 0{\cdot}00009(F_o)^2]^{-1}$, $(\Delta/\sigma)_{\max} < 0{\cdot}04$; residual electron density within $\pm 0{\cdot}4$ e Å^{-3}, isotropic extinction parameter $X = 0{\cdot}00179$;

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final $R = 0{\cdot}075$, $wR = 0{\cdot}053$; scattering factors from *International Tables for X-ray Crystallography* (1974), anomalous-dispersion corrections for S, O and C.

Discussion. A perspective molecular drawing of (II) is shown in Fig. 1. Final atomic coordinates are in Table 1 and bond distances and angles in Table 2. Calculations of the least-squares planes passing through the cyclohexane, 1,3-dioxane and phenyl rings, and the methoxycarbonyl group, the deviations of individual atoms from the planes and interplanar angles have been deposited.*

The phenyl ring and the methoxycarbonyl group are planar within the limit of experimental error. The average phenyl C—C bond length of 1·372 (9) Å is in agreement with the accepted value of 1·39 Å.

The cyclohexane and phenyl rings are linked through a sulfoxide bridge. The cyclohexane ring exhibits a $^4C_1$ chair conformation. The average bond length and angle for the chair structure of 1·527 (8) Å and 111·5 (5)°, respectively, agree with values obtained for cyclohexane (Kahn, Fourme, André & Renaud, 1973). The average value of the ring-torsion angle is 54·5 (7)°. The substituents at C(2) and C(4) are in equatorial and axial positions respectively.

The two C—S bonds are unequal; C(13)—S is shorter by 0·04 Å than S—C(2); the contraction of the C(2)—S—C(13) angle is probably due to the tendency of the S atom to keep an unshared electron pair in the more stable *s* (relative to *p*) orbital, thereby reducing the amount of *s* character in the sulfur bond orbitals and

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² ×10³)

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}.$$

	x	y	z	U_{eq}
S	226 (2)	10175 (1)	1893 (1)	44 (1)
O(1)	−319 (4)	7658 (4)	1970 (2)	38 (2)
O(2)	−2068 (4)	8742 (4)	1482 (3)	42 (2)
O(3)	1511 (4)	9800 (4)	2146 (3)	63 (2)
O(4)	−1758 (4)	10309 (4)	4472 (3)	73 (2)
O(5)	−2383 (5)	8582 (5)	4951 (3)	79 (2)
C(1)	−1301 (6)	8354 (5)	2229 (4)	36 (2)
C(2)	−723 (5)	9475 (5)	2653 (4)	40 (2)
C(3)	9 (6)	9241 (6)	3534 (4)	50 (3)
C(4)	−706 (6)	8524 (5)	4159 (4)	49 (3)
C(5)	−1212 (6)	7403 (5)	3719 (4)	56 (3)
C(6)	−2014 (5)	7687 (5)	2871 (4)	49 (3)
C(7)	−713 (6)	6676 (5)	1420 (4)	45 (3)
C(8)	−1527 (6)	7071 (6)	602 (4)	43 (3)
C(9)	−2541 (6)	7795 (6)	925 (4)	49 (3)
C(10)	−2069 (6)	5941 (6)	149 (4)	75 (4)
C(11)	−828 (6)	7780 (6)	−18 (4)	64 (3)
C(12)	−1652 (7)	9260 (6)	4530 (4)	53 (3)
C(13)	131 (5)	11649 (5)	2310 (4)	40 (3)
C(14)	1112 (6)	12105 (6)	2823 (4)	57 (3)
C(15)	1105 (7)	13272 (7)	3073 (5)	71 (4)
C(16)	134 (7)	13972 (6)	2806 (5)	65 (3)
C(17)	−841 (6)	13527 (5)	2294 (4)	61 (3)
C(18)	−857 (5)	12351 (5)	2056 (4)	48 (3)
C(19)	−3320 (7)	9179 (7)	5332 (5)	108 (5)

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

E.s.d.'s are given in parentheses.

S—O(3)	1·497 (4)	S—C(2)	1·830 (6)
S—C(13)	1·794 (6)	O(1)—C(1)	1·434 (7)
O(1)—C(7)	1·433 (7)	O(2)—C(1)	1·418 (7)
O(2)—C(9)	1·434 (7)	O(4)—C(12)	1·196 (8)
O(5)—C(12)	1·332 (9)	O(5)—C(19)	1·417 (10)
C(1)—C(2)	1·534 (8)	C(1)—C(6)	1·526 (9)
C(2)—C(3)	1·520 (8)	C(3)—C(4)	1·539 (9)
C(4)—C(5)	1·516 (9)	C(4)—C(12)	1·499 (10)
C(5)—C(6)	1·526 (8)	C(7)—C(8)	1·529 (8)
C(8)—C(9)	1·518 (9)	C(8)—C(10)	1·546 (9)
C(8)—C(11)	1·517 (9)	C(13)—C(14)	1·372 (9)
C(13)—C(18)	1·375 (8)	C(14)—C(15)	1·377 (10)
C(15)—C(16)	1·363 (10)	C(16)—C(17)	1·362 (10)
C(17)—C(18)	1·380 (9)		
O(3)—S—C(2)	108·1 (3)	O(3)—S—C(13)	105·2 (3)
C(2)—S—C(13)	96·7 (3)	C(1)—O(1)—C(7)	113·3 (4)
C(1)—O(2)—C(9)	113·4 (4)	C(12)—O(5)—C(19)	115·8 (6)
O(1)—C(1)—O(2)	110·8 (5)	O(1)—C(1)—C(2)	106·1 (5)
O(2)—C(1)—C(2)	105·7 (5)	O(1)—C(1)—C(6)	111·5 (5)
O(2)—C(1)—C(6)	111·3 (5)	C(2)—C(1)—C(6)	111·1 (5)
S—C(2)—C(1)	109·4 (4)	S—C(2)—C(3)	110·5 (4)
C(1)—C(2)—C(3)	112·9 (5)	C(2)—C(3)—C(4)	112·5 (5)
C(3)—C(4)—C(5)	110·9 (5)	C(3)—C(4)—C(12)	111·8 (5)
C(5)—C(4)—C(12)	113·1 (6)	C(4)—C(5)—C(6)	110·8 (5)
C(1)—C(6)—C(5)	110·9 (5)	O(1)—C(7)—C(8)	111·6 (5)
C(7)—C(8)—C(9)	106·5 (5)	C(7)—C(8)—C(10)	106·9 (5)
C(9)- C(8) C(10)	109·1 (5)	C(7)—C(8)—C(11)	111·7 (5)
C(9)—C(8)—C(11)	111·4 (5)	C(10)—C(8)—C(11)	111·0 (5)
O(2)—C(9)—C(8)	111·1 (5)	O(4)—C(12)—O(5)	123·3 (7)
O(4)—C(12)—C(4)	126·3 (7)	O(5)—C(12)—C(4)	110·5 (6)
S—C(13)—C(14)	118·7 (5)	S—C(13)—C(18)	120·9 (4)
C(14)—C(13)—C(18)	120·2 (6)	C(13)—C(14)—C(15)	119·5 (6)
C(14)—C(15)—C(16)	120·2 (7)	C(15)—C(16)—C(17)	120·6 (7)
C(16)—C(17)—C(18)	119·7 (6)	C(13)—C(18)—C(17)	119·8 (6)

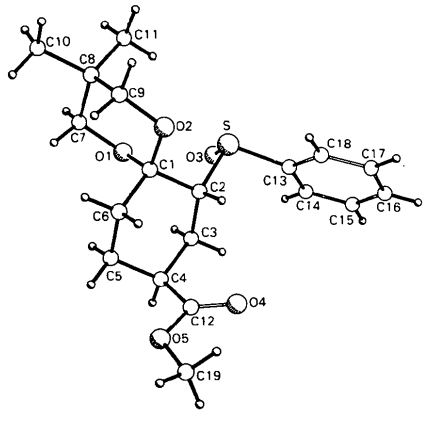

Fig. 1. View of the molecule showing its conformation and the atom numbering used.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and the results of mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39163 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

producing weaker (and longer) S–X bonds (where X = C or O) and smaller X–S–X' bond angles (Day, Kingsbury & Day, 1981; Hoyos-Guerrero, Martínez-Carrera & García-Blanco, 1983). O(3) is twisted out of the plane of the phenyl ring. The pertinent torsion angles are O(3)–S–C(13)–C(14) = −6·3 (6) and O(3)–S–C(13)–C(18) = 167·6 (5)°.

The 1,3-dioxane ring adopts a chair conformation. The best *anti* relation between vicinal atoms is found with torsion angles O(1)–C(1)–C(6)–C(5) and C(3)–C(2)–S–C(13), 62·5 (6) and −80·9 (4)°. However, the angle between the normals to the planes of the 1,3-dioxane and phenyl rings is only 27·4 (5)°.

There is no intramolecular interaction between the phenyl ring and the methoxycarbonyl group; the planes defined by C(13)–C(18) and C(4), C(12), O(4), O(5), C(19) are nearly perpendicular [114·2 (5)°] (Fig. 1).

The 1,3-dioxane ring and the methoxycarbonyl group are oriented to minimize transannular repulsions between their O atoms and the sulfoxide group and cyclohexane ring [O(1)···S, 2·918 (4); O(2)···S, 3·026 (4); O(3)···O(1), 3·16 (1); and O(4)···C(2), 3·26 (1) Å], forming a spiral-like structure.

There are no intermolecular contacts short enough to affect the conformation of the molecule. The shortest intermolecular distances between non-H atoms are C(6)···O(3) ($-0·5 + x$, $1·5 - y$, z) 3·38 (1) and C(11)···S ($-x$, $2 - y$, $-z$) 3·80 (1) Å. The packing in the crystal is entirely due to van der Waals forces.

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Structure of 2-(4,6-Dimethyl-2-pyridyliminomethyl)phenol, $C_{14}H_{14}N_2O$

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Abstract. $M_r = 226·3$, orthorhombic, $P2_12_12_1$, $a = 21·585$ (5), $b = 8·8285$ (8), $c = 6·2952$ (6) Å (JCPDS Diffraction File No. 33-1998), $V = 1199·6$ (3) Å^3, $Z = 4$, $D_m = 1·22$ (3) (flotation in dioxane and bromoform), $D_x = 1·253$ (1) Mg m^{-3}, λ(Mo $K\alpha_1$) = 0·7093 Å, $\mu = 0·09$ mm^{-1}, $F(000) = 480$, $T = 294$ K, final $R = 0·073$ for 1054 observed reflections. The molecule is not planar; the pyridine-ring plane is inclined at 5·4 (9)° to the salicylidene-ring plane. The orientation adopted by the pyridine ring is such that the exocyclic C atom is *cis* to the pyridine N. There is a strong intramolecular O–H···N hydrogen bond, O···N 2·60 (1) Å.

Introduction. The present work continues investigations on the structures of Schiff bases derived from salicylaldehyde and 2-aminopyridine.

Experimental. Dark yellow crystals (from methanol solution), 0·21 × 0·21 × 0·34 mm. Philips PW 1100 diffractometer, graphite-monochromatized Mo $K\alpha$, $\omega/2\theta$ scan mode. Two standard reflections every 55 measurements (variation ±2%). 2040 independent *hkl* with $2\theta < 60°$, 986 unobserved with $I < 2\sigma(I)$. Lp correction, absorption and anomalous dispersion ignored. 52 reflections with $2\theta < 47°$ used for refining cell parameters. Direct methods (*MULTAN*76, Main,

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