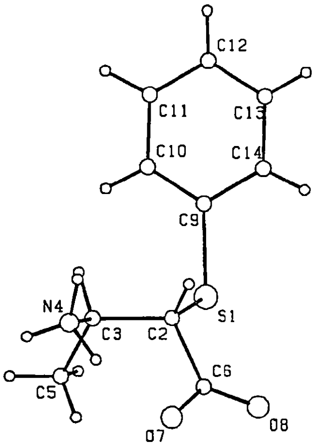

Fig. 1. Perspective view with the atom-numbering system.

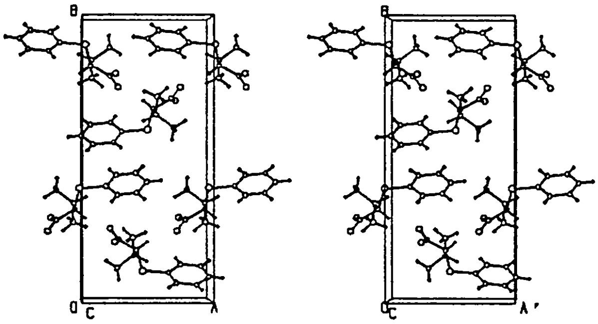

Fig. 2. A stereoview of the unit-cell packing.

least squares. Temperature factor of each H atom equal to B_{eq} of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00128|F_o|^2]$, $w = 0$ for nine reflections with $w^{1/2}|\Delta F| \geq 3$. Final $R = 0{\cdot}032$, $wR = 0{\cdot}042$, $S = 1{\cdot}0853$. Highest peak in final difference map using the F data of $\theta \leq 18°$ is 0·3 e Å^{-3}. Max. Δ/σ in the final cycle 0·1. 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 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 stereoview 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 discussed in Hata & Watanabe (1987).

The author thanks Dr Hata and Dr Watanabe for the supply of 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 51036 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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rel-(2*S*,6*S*)-2-(6-Hydroxy-4,4,6-trimethyl)morpholinomethanesulfonate

By William J. Colucci, Frank R. Fronczek, Richard D. Gandour and Steven F. Watkins

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, *USA*

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Abstract. $C_8H_{17}NO_5S$, $M_r = 239{\cdot}3$, orthorhombic, *Pbca*, $a = 11{\cdot}789\,(4)$, $b = 14{\cdot}679\,(2)$, $c = 12{\cdot}733\,(1)$ Å, $V = 2203{\cdot}5\,(8)$ Å^3, $Z = 8$, $D_x = 1{\cdot}443$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}8$ cm^{-1}, $F(000) = 1024$, $T = 295\,(1)$ K, 3005 unique reflections measured, final $R = 0{\cdot}058$ for 1360 reflections having $F_o > 5\sigma(F_o)$. Pairs of zwitterion molecules are bound centrosymmetrically by hydrogen bonds between the hydroxyl group and a sulfonate oxygen (O3): O2···O3 = 2·789 (4), O2—H = 1·05 (1), H···O3 = 1·80 (1) Å,

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Table 1. *Experimental details*

Crystal (mm)	0·36 × 0·32 × 0·06
Instrument	Enraf–Nonius CAD-4
Monochromator	Incident beam, graphite
Unit cell	25 reflections (23–28° 2θ)
Mode	ω–2θ
Scan rates (° min^{-1})	Variable (0·74–3·30)
Standards	400, 040, 004
Decay	None
Corrections	Background, Lorentz, polarization Empirical absorption (1·00–0·879 on I)
2θ (°)	4–60
hkl	h = 0 to 14, k = 0 to 18, l = 0 to 15
Reflections	3005 total 1360 observed [$F_o > 5\sigma(F_o)$]
Solution	Direct methods
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weights	$4F_o^2\,\mathrm{Lp}/[S^2(C+R^2B)+(0{\cdot}020F_o^2)^2]$ S = scan rate, C = integrated count, R = scan time/background time, B = background count
Parameters refined	136
R, wR, R(all)	0·058, 0·056, 0·184
Goodness of fit	2·33
Maximum Δ/σ	0·03
$\Delta\rho$ (e Å^{-3})	0·49 (8), –0·37 (8)

Table 2. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)
S1	0·43127 (9)	0·41788 (7)	0·23000 (8)	2·74 (2)
O1	0·6872 (2)	0·4315 (2)	0·0270 (2)	2·79 (6)
O2	0·6702 (3)	0·3569 (2)	–0·1335 (2)	3·46 (7)
O3	0·3870 (3)	0·5033 (2)	0·2720 (2)	4·29 (7)
O4	0·4680 (3)	0·3569 (2)	0·3086 (3)	5·78 (9)
O5	0·3552 (3)	0·3786 (2)	0·1553 (3)	4·80 (8)
N1	0·7130 (3)	0·2390 (2)	0·0589 (2)	2·66 (7)
C1	0·6019 (3)	0·3816 (3)	0·0827 (3)	2·48 (9)
C2	0·7448 (4)	0·3806 (3)	–0·0522 (3)	2·67 (9)
C3	0·7948 (4)	0·2947 (3)	–0·0052 (3)	2·38 (8)
C4	0·6546 (4)	0·3007 (3)	0·1372 (3)	2·70 (9)
C5	0·5540 (4)	0·4521 (3)	0·1584 (3)	3·2 (1)
C6	0·7801 (4)	0·1686 (3)	0·1169 (3)	4·2 (1)
C7	0·6286 (4)	0·1902 (3)	–0·0101 (4)	3·9 (1)
C8	0·8398 (4)	0·4418 (3)	–0·0900 (4)	3·8 (1)

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation

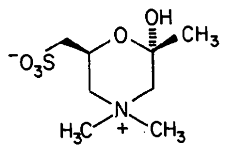
$$\tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha].$$

Table 3. *Bond distances* (Å) *and angles* (°)

S1–O3	1·459 (3)	N1–C4	1·512 (5)
S1–O4	1·411 (3)	N1–C6	1·496 (6)
S1–O5	1·429 (3)	N1–C7	1·508 (6)
S1–C5	1·782 (4)	C1–C4	1·509 (5)
O1–C1	1·432 (5)	C1–C5	1·523 (6)
O1–C2	1·427 (5)	C2–C3	1·515 (5)
O2–C2	1·402 (5)	C2–C8	1·515 (6)
N1–C3	1·505 (5)		
O3–S1–O4	113·3 (2)	O1–C1–C4	109·9 (3)
O3–S1–O5	111·5 (2)	O1–C1–C5	103·1 (3)
O3–S1–C5	103·6 (2)	C4–C1–C5	113·4 (3)
O4–S1–O5	114·1 (2)	O1–C2–O2	110·7 (3)
O4–S1–C5	107·0 (2)	O1–C2–C3	110·0 (3)
O5–S1–C5	106·4 (2)	O1–C2–C8	105·4 (3)
C1–O1–C2	114·6 (3)	O2–C2–C3	109·2 (3)
C3–N1–C4	108·9 (3)	O2–C2–C8	112·1 (3)
C3–N1–C6	107·7 (3)	C3–C2–C8	109·4 (4)
C3–N1–C7	111·4 (3)	N1–C3–C2	114·7 (3)
C4–N1–C6	109·2 (3)	N1–C4–C1	110·8 (3)
C4–N1–C7	111·6 (3)	S1–C5–C1	115·7 (3)
C6–N1–C7	108·0 (3)		

O2–H···O3 = 155 (1)°. As a result, the S–O3 bond [1·459 (3) Å] is significantly longer than the other two S–O bonds [average 1·420 (2) Å].

Experimental. Compound (1) was prepared as reported (Colucci, 1987). A colorless tabular crystal was mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



(2*S*,6*S*)-(1)

The structure was solved using direct methods. A total of 14 atoms was located from an *E* map. The remaining atoms, including H atoms, were located in successive difference Fourier syntheses. H atoms were included in the structure-factor calculation, but were constrained to ride on the atom to which they are bonded. Their *B* values were intially set at 1·3B_{eq} for the bonded atom, and shifts in B_{11} for the bonded atom were also applied to *B* for H. The structural model, refined by full-matrix least squares, included isotropic (H atoms) and anisotropic (non-H atoms) thermal parameters. Atomic scattering factors, including those for anomalous dispersion, were taken from the *International Tables for X-ray Crystallography* (1974). The final cycle of refinement included 136 variable parameters and converged to R = 0·058. Programs used were *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *SDP/VAX* (Frenz, 1978), *ORTEP* (Johnson, 1965), and *PLUTO*78 (Motherwell & Clegg, 1978).

Final positional and equivalent isotropic thermal parameters are given in Table 2,* and bond lengths and bond angles are in Table 3. Fig. 1 shows the molecule and the atomic numbering scheme.

Related literature. Structure of carnitine: Gandour, Colucci & Fronczek (1985); 2-carboxymethylmorpholinium derivatives as competitive inhibitors for carnitine acetyltransferase and carnitine palmitoyl-

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51041 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

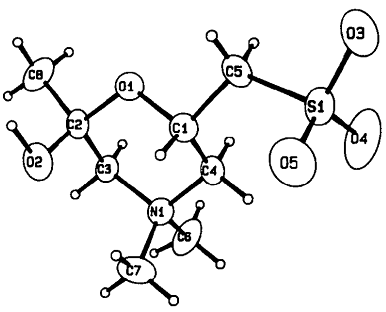

Fig. 1. View of the title compound, 50% thermal ellipsoids (Johnson, 1965).

transferase: Gandour, Colucci, Brady & Brady (1986), Colucci, Gandour, Fronczek, Brady & Brady (1987); as model enzyme-bound reaction intermediates: Colucci & Gandour (1988).

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(+)-7,9-*O,O*-Maleoylretronecine, $C_{12}H_{13}NO_4$: a Pyrrolizidine Alkaloid Analogue

By Michael Burton, Andrew A. Freer* and David J. Robins

Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, Scotland

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Abstract. (+)-7-Methyl-2,3,5,7a-tetrahydro-1*H*-pyrrolizine-1,8-diyl maleate, $M_r = 235{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}826\,(2)$, $b = 10{\cdot}510\,(1)$, $c = 13{\cdot}566\,(1)$ Å, $V = 1115{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}40$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}99$ cm^{-1}, $F(000) = 496$, $T = 291$ K, final $R = 0{\cdot}037$ for 1816 observed reflections. The title compound is isomorphous with the previously reported ten-membered macrocyclic diester of (+)-7,9-*O,O*-succinoylretronecine [Burton, Freer & Robins (1985). *Acta Cryst.* C**41**, 944–946]. The pyrrolizidine nucleus adopts the familiar *exo–endo* conformation with ring *A* exo-buckled with a puckering angle of 41·7 (4)° whilst the *endo* ring, *B*, has a puckering angle of 179·0 (5)°. The dihedral angle of 127·6 (3)° between the planes defined by atoms C(1), C(8), N(4), C(3) and C(5), N(4), C(8), C(7) is similar to values found for other pyrrolizidine nuclei. The ester-group carbonyls lie on either side of the macrocycle in a nearly antiparallel orientation.

Experimental. Colourless, plate-shaped crystals grown from hexane, crystal *ca* 0·5 × 0·4 × 0·1 mm used in data collection, CAD-4 diffractometer. Systematic absences from Weissenberg photographs indicated the crystals to be orthorhombic, $P2_12_12_1$. 3122 independent intensities, θ limit 28°, $\omega/2\theta$ scan. Two standard intensities ($0\bar{1}\bar{8}$ and $0\bar{5}\bar{5}$) were used to monitor variations in intensity data; <2% variation observed. Least-squares refinement on 25 reflections, $\theta > 12°$, used to determine lattice parameters. No absorption correction, h 0 to 10, k 0 to 13, l 0 to 17. Structure solution by direct methods with *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for all non-H atoms converged to R and wR of 0·037 and 0·046 with $w = 1/\sigma^2(F_o)$. H-atom coordinates located from difference Fourier synthesis were included, but not refined, in the final cycles of least squares. 1816 reflections with $I \geq 3{\cdot}0\sigma_I$ used. $(\Delta/\sigma)_{\max} = 0{\cdot}12$; max. and min. heights in final difference Fourier synthesis $= 0{\cdot}24$ and $-0{\cdot}18$ e Å^{-3}. Scattering factors from

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* To whom all correspondence should be addressed.

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