e.s.d.'s while Table 2 gives the bond lengths and angles for the non-hydrogen atoms.

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

C(1)–C(2)	1·584 (11)	C(5)–C(6)	1·537 (12)
C(1)–C(6)	1·549 (11)	C(7)–C(13)	1·540 (13)
C(1)–C(7)	1·569 (11)	C(7)–C(14)	1·539 (13)
C(1)–C(12)	1·539 (12)	C(8)–C(9)	1·504 (14)
C(2)–C(3)	1·561 (11)	C(8)–S(2)	1·814 (10)
C(2)–S(1)	1·879 (8)	C(9)–C(10)	1·562 (14)
C(2)–S(2)	1·822 (8)	C(10)–C(11)	1·521 (13)
C(3)–C(4)	1·514 (12)	C(10)–S(1)	1·813 (10)
C(4)–C(5)	1·560 (13)	O–S(1)	1·499 (6)
C(4)–C(7)	1·546 (12)		

C(2)–C(1)–C(6)	106·3 (6)	C(4)–C(5)–C(6)	103·7 (7)
C(2)–C(1)–C(7)	102·9 (6)	C(1)–C(6)–C(5)	103·2 (7)
C(2)–C(1)–C(12)	116·7 (6)	C(1)–C(7)–C(4)	93·2 (6)
C(6)–C(1)–C(7)	100·2 (6)	C(1)–C(7)–C(13)	115·8 (7)
C(6)–C(1)–C(12)	114·0 (7)	C(1)–C(7)–C(14)	114·0 (7)
C(7)–C(1)–C(12)	114·8 (7)	C(4)–C(7)–C(13)	114·0 (7)
C(1)–C(2)–C(3)	102·3 (6)	C(4)–C(7)–C(14)	112·6 (7)
C(1)–C(2)–S(1)	114·4 (5)	C(13)–C(7)–C(14)	107·1 (7)
C(1)–C(2)–S(2)	119·0 (5)	C(2)–S(2)–C(8)	105·6 (4)
C(3)–C(2)–S(1)	103·9 (5)	C(9)–C(8)–S(2)	114·6 (7)
C(3)–C(2)–S(2)	108·0 (5)	C(8)–C(9)–C(10)	112·8 (8)
S(1)–C(2)–S(2)	107·9 (4)	C(9)–C(10)–C(11)	111·5 (8)
C(2)–C(3)–C(4)	103·6 (7)	C(9)–C(10)–S(1)	110·7 (6)
C(3)–C(4)–C(5)	108·0 (7)	C(11)–C(10)–S(1)	106·4 (6)
C(3)–C(4)–C(7)	102·8 (7)	C(2)–S(1)–C(10)	101·7 (4)
C(5)–C(4)–C(7)	101·9 (7)	C(2)–S(1)–O	106·8 (4)
		C(10)–S(1)–O	106·0 (4)

Related literature. The S(1)→O bond distance is 1·499 (6) Å. Both the sulfoxide and the methyl groups are *cis*. The methyl group is equatorial while the sulfoxide is axial. The norbornane ring has a *synchro* twist (*S*++) (Acharya, Tavale & Guru Row, 1984). The molecules in the crystal are held together by van der Waals interactions.

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(2*S*,6*R*)-6-Carboxymethyl-2-ethyl-2-hydroxy-4,4-dimethylmorpholinium Bromide

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Abstract. $C_{10}H_{20}NO_4^+.Br^-$, $M_r = 298·2$, orthorhombic, $P2_12_12_1$, $a = 6·976$ (2), $b = 13·603$ (2), $c = 13·674$ (4) Å, $V = 1297·7$ (8) Å^3, $Z = 4$, $D_x = 1·526$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 43·7$ cm^{-1}, $F(000) = 616$, $T = 298$ K, $R = 0·032$ for 1463 observations (of 1554 unique data). The molecule is a morpholinium ring in a chair conformation, containing two chiral centers with the carboxymethyl and ethyl groups *cis*. Hydrogen bonding occurs between the carboxy H atom and the Br ion with an O···Br distance of 3·218 (3) Å, and H···Br distance of 2·45 (5) Å and an angle of 158 (5)° at the H atom. A hydrogen bond also exists between the hydroxy H atom and another Br ion, with an O···Br distance of 3·323 (3) Å, and H···Br distance of 2·35 (6) Å, and an angle of 161 (4)° at the H atom.

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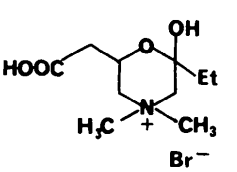
Experimental. Colorless needles, m.p. 431–432 K, of (2*S*,6*R*)-6-carboxymethyl-2-ethyl-2-hydroxy-4,4-dimethylmorpholinium bromide [hemipropanoylcarnitinium (1)] prepared by the reaction of norcarnitine



(1)

with 1-bromo-2-butanone followed by acid hydrolysis, were crystallized from ethanol by vapor diffusion with ethyl ether. Crystal size 0·36 × 0·48 × 0·60 mm, capillary-mounted for protection from humidity, space group from systematic absences *h*00 with *h* odd, 0*k*0 with *k* odd and 00*l* with *l* odd, cell dimensions from

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33})$.

	x	y	z	B_{eq}(Å^2)
Br	0·79518 (6)	0·00508 (3)	0·11999 (3)	4·003 (8)
O(1)	0·0860 (3)	0·2256 (2)	0·7444 (2)	2·14 (4)
O(2)	0·2406 (4)	0·2921 (2)	0·4807 (2)	4·32 (6)
O(3)	0·3832 (5)	0·3399 (2)	0·6182 (2)	4·09 (6)
O(4)	0·2157 (4)	0·2549 (2)	0·8990 (2)	3·03 (4)
N	0·1570 (5)	0·0423 (2)	0·8452 (2)	2·72 (5)
C(1)	0·2002 (5)	0·0577 (2)	0·7380 (2)	2·61 (6)
C(2)	0·2427 (4)	0·1652 (2)	0·7147 (2)	2·05 (5)
C(3)	0·0541 (5)	0·2229 (2)	0·8478 (2)	2·23 (5)
C(4)	0·0100 (5)	0·1171 (2)	0·8783 (2)	2·89 (6)
C(5)	0·0673 (8)	−0·0576 (3)	0·8575 (3)	4·27 (9)
C(6)	0·3375 (7)	0·0459 (3)	0·9052 (3)	4·03 (8)
C(7)	0·2616 (5)	0·1748 (3)	0·6050 (2)	2·60 (6)
C(8)	0·3034 (5)	0·2785 (2)	0·5715 (2)	2·60 (6)
C(9)	−0·1181 (6)	0·2895 (3)	0·8679 (3)	3·36 (7)
C(10)	−0·3016 (6)	0·2630 (3)	0·8155 (4)	5·2 (1)

Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

O(1)–C(2)	1·426 (4)	N–C(5)	1·505 (5)
O(1)–C(3)	1·431 (4)	N–C(6)	1·503 (6)
O(2)–C(8)	1·329 (4)	C(1)–C(2)	1·526 (4)
O(2)–H(20)	0·81 (5)	C(2)–C(7)	1·512 (4)
O(3)–C(8)	1·190 (4)	C(3)–C(4)	1·530 (5)
O(4)–C(3)	1·396 (4)	C(3)–C(9)	1·530 (5)
O(4)–H(40)	1·01 (6)	C(7)–C(8)	1·511 (5)
N–C(1)	1·512 (4)	C(9)–C(10)	1·511 (6)
N–C(4)	1·514 (5)		

C(2)–O(1)–C(3)	112·7 (2)	O(1)–C(3)–O(4)	111·2 (2)
C(8)–O(2)–H(20)	108 (4)	O(1)–C(3)–C(4)	108·9 (2)
C(3)–O(4)–H(40)	107 (3)	O(1)–C(3)–C(9)	106·5 (3)
C(1)–N–C(4)	109·3 (2)	O(4)–C(3)–C(4)	108·6 (3)
C(1)–N–C(5)	108·5 (2)	O(4)–C(3)–C(9)	111·1 (3)
C(1)–N–C(6)	111·0 (3)	C(4)–C(3)–C(9)	110·5 (3)
C(4)–N–C(5)	106·9 (3)	N–C(4)–C(3)	114·5 (3)
C(4)–N–C(6)	112·5 (3)	C(2)–C(7)–C(8)	113·5 (3)
C(5)–N–C(6)	108·5 (3)	O(2)–C(8)–O(3)	124·0 (3)
N–C(1)–C(2)	112·0 (2)	O(2)–C(8)–C(7)	110·4 (3)
O(1)–C(2)–C(1)	110·1 (2)	O(3)–C(8)–C(7)	125·6 (3)
O(1)–C(2)–C(7)	107·5 (2)	C(3)–C(9)–C(10)	116·0 (3)
C(1)–C(2)–C(7)	107·9 (2)		

C(3)–O(1)–C(2)–C(1)	63·0 (3)	N–C(1)–C(2)–O(1)	−56·8 (4)
C(3)–O(1)–C(2)–C(7)	−179·7 (2)	N–C(1)–C(2)–C(7)	−173·7 (3)
C(2)–O(1)–C(3)–O(4)	59·5 (3)	O(1)–C(2)–C(7)–C(8)	61·1 (3)
C(2)–O(1)–C(3)–C(9)	−179·3 (2)	O(4)–C(3)–C(4)–N	−68·2 (3)
H(40)–O(4)–C(3)–O(1)	83 (3)	C(9)–C(3)–C(4)–N	169·7 (3)
H(40)–O(4)–C(3)–C(4)	−157 (3)	O(1)–C(3)–C(9)–C(10)	58·1 (4)
H(40)–O(4)–C(3)–C(9)	−35 (3)	O(4)–C(3)–C(9)–C(10)	179·4 (3)
C(4)–N–C(1)–C(2)	49·0 (4)	C(2)–C(7)–C(8)–O(2)	−153·8 (3)
C(5)–N–C(1)–C(2)	165·2 (3)	C(2)–C(7)–C(8)–O(3)	27·2 (5)

setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0·46–3·30° min^{-1}. Data having $2 < \theta < 75°$, $0 \le h \le 8$, $0 \le k \le 17$, $0 \le l \le 17$ measured. Data corrected for background, Lorentz, polarization, decay and absorption effects. Absorption corrections were based on ψ scans, with a minimum relative transmission coefficient of 76·32%. Standard reflections 200, 060, 004 indicated a 5·3% intensity decay, and a linear correction was applied. 1554 unique data, 1463 observed with $I > 3\sigma(I)$, used in the refinement. Structure solved by heavy-atom methods and refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$, using *Enraf–Nonius SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically, H atoms located by ΔF; the OH and COOH H atoms were refined isotropically, while other H atoms were included as fixed contributions. Final $R = 0{\cdot}032$, $wR = 0{\cdot}050$, $S = 3{\cdot}555$ for 154 variables. Maximum shift $0{\cdot}22\sigma$ in the final cycle, max. residual density: 0·83 e Å^{-3} near the Br-ion position, min. −0·51 e Å^{-3}; extinction coefficient $g = 5{\cdot}2\,(2) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. The enantiomorphous structure was refined under identical conditions, yielding $R = 0{\cdot}041$, $wR = 0{\cdot}062$, $S = 4{\cdot}378$, thus the former model was taken as the correct absolute configuration, which is consistent with the known chirality of the starting materials. The coordinates are tabulated in Table 1,* bond distances, bond angles and selected torsion angles are given in Table 2, and the cation is illustrated in Fig. 1.

Related literature. Structure of carnitine: Gandour, Colucci & Fronczek (1985). 2-Carboxymethylmorpholinium derivatives as competitive inhibitors for carnitine acetyltransferase and carnitine palmitoyltransferase: Gandour, Colucci, Stelly, Brady & Brady (1986); Colucci, Gandour, Fronczek, Brady & Brady

* Tables of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51685 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

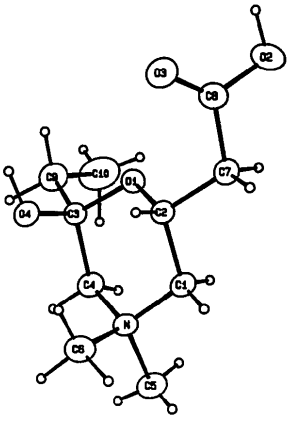


Fig. 1. *ORTEP* (Johnson, 1965) drawing of title compound.

(1987); as model enzymatic reaction intermediates: Colucci & Gandour (1988). Structure of *rel*-(2*S*,6*S*)-2-(6-hydroxy-4,4,6-trimethyl)morpholinomethanesulfonate: Colucci, Fronczek, Gandour & Watkins (1988).

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An Aminocyclohexanecarboxylic Acid

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Abstract. 8-Amino-1,4-dioxaspiro[4.5]decane-8-carboxylic acid, $C_9H_{15}NO_4$, $M_r = 201{\cdot}2$, orthorhombic, $Pna2_1$, $a = 27{\cdot}234\ (4)$, $b = 5{\cdot}5786\ (10)$, $c = 6{\cdot}4522\ (9)$ Å, $V = 980{\cdot}3\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}363$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}00$ cm^{-1}, $F(000) = 432$, $T = 297$ K, $R = 0{\cdot}036$ for 1469 data having $F_o^2 > 0$. The six-membered ring adopts a near ideal chair conformation with the carboxylate as an axial substituent. The ammonio group is nearly antiperiplanar to the carboxylate. All three NH bonds are involved in intermolecular hydrogen bonding with neighboring carboxylates.

Experimental. The title compound was prepared according to Britten & Lockwood (1974). Colorless, crystals, dec. 573 K, suitable for single-crystal X-ray diffraction were crystallized from methanol/water at room temperature. All standard spectroscopic measurements support the X-ray structure determination.

Intensity data were obtained from an irregular fragment of dimensions 0·28 × 0·40 × 0·48 mm mounted in a random orientation on an Enraf–Nonius CAD-4 diffractometer. Cell dimensions were determined at 297 K by a least-squares fit to setting angles of 25 reflections having $22 > 2\theta > 19°$. Two octants of data having $2 < 2\theta < 60°$, $-38 \le h \le 38$, $0 \le k \le 9$, $0 \le l \le 7$ were measured using graphite-monochromated Mo $K\alpha$ radiation. The ω–2θ scans were made at speeds ranging from 0·45 to 4·0° min^{-1} to measure all significant data with approximately equal precision. Three standard reflections (800, 020, 002) exhibited no decline in intensity during data collection. Data included corrections for background, Lorentz, and polarization. Absorption was negligible.

The space group was determined by systematic absences $0kl$ with $k+l$ odd, $h0l$ with h odd, and successful refinement of the noncentrosymmetric model. The structure was solved by direct methods and refined by full-matrix least squares based upon F, with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using the Enraf–Nonius *SDP* (Frenz, 1985), scattering factors of Cromer &

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{8}{3}\pi^2(U_{11} + U_{22} + U_{33})$.

	x	y	z	B_{eq}(Å^2)
O1	0·55512 (4)	−0·1689 (2)	1·0000	3·06 (2)
O2	0·53117 (4)	−0·2371 (2)	0·6789 (2)	3·27 (2)
O3	0·67193 (4)	0·4357 (2)	0·6062 (2)	3·35 (2)
O4	0·71427 (4)	0·0842 (2)	0·6064 (3)	3·66 (2)
N	0·51540 (4)	0·2913 (2)	0·7889 (2)	2·05 (2)
C1	0·54913 (4)	−0·1085 (2)	0·8170 (3)	2·11 (2)
C2	0·56257 (4)	0·1553 (2)	0·7592 (2)	1·86 (2)
C3	0·57845 (5)	0·1833 (2)	0·5333 (2)	2·21 (2)
C4	0·62917 (5)	0·0736 (3)	0·4974 (3)	2·65 (3)
C5	0·66668 (5)	0·1850 (3)	0·6428 (3)	2·62 (2)
C6	0·65149 (5)	0·1516 (3)	0·8668 (3)	2·83 (3)
C7	0·60097 (5)	0·2609 (3)	0·9060 (3)	2·39 (2)
C8	0·70990 (6)	0·4610 (4)	0·4552 (3)	3·97 (4)
C9	0·74349 (8)	0·2556 (4)	0·5033 (4)	5·32 (4)

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