*MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement by full-matrix least squares with anisotropic temperature factors for non-H atoms. Function minimized $\sum w[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}007(F_o)^2]$, $\sigma(F_o)$ determined from counting statistics. All H atoms were located from difference map and refined isotropically. Final discrepancy indices $R = 0{\cdot}055$, $wR = 0{\cdot}053$, $S = 1{\cdot}132$ for 1539 reflexions with $F > 3\sigma(F)$. Maximum $\Delta/\sigma = 0{\cdot}08$ in final least-squares cycle. Final difference Fourier map showed no residuals greater than 0·44 e Å^{-3}. All calculations were performed using a Panafacom computer with *RCRYSTAN* (Rigaku Corporation, 1985). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Final atomic parameters are listed in Table 1.* The bond lengths and angles are given in Table 2. Fig. 1 shows the thermal-ellipsoid plot of the molecule with atomic labelling, Fig. 2 the crystal structure.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52569 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound is obtained from photoreaction of benzonitrile and phenylacetylene in CH_3OH. See also Adam & Klug (1985) for the preparation of related compounds.

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(2*S*,6*R*)-6-Carboxymethyl-2-hydroxy-4,4-dimethyl-2-phenylmorpholinium Chloride Hemihydrate

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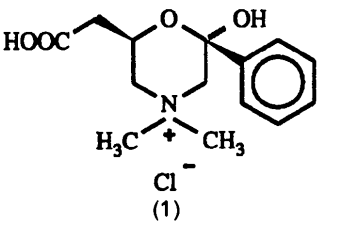
Abstract. $C_{14}H_{20}NO_4^+.Cl^-.\frac{1}{2}H_2O$, $M_r = 310{\cdot}8$, monoclinic, $P2_1$, $a = 15{\cdot}425\ (2)$, $b = 8{\cdot}725\ (2)$, $c = 11{\cdot}490\ (3)$ Å, $\beta = 90{\cdot}61\ (2)°$, $V = 1546{\cdot}2\ (10)$ Å^3, $Z = 4$, $D_x = 1{\cdot}335$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 23{\cdot}6$ cm^{-1}, $F(000) = 660$, $T = 299$ K, $R = 0{\cdot}037$ for 3027 observations (of 3394 unique data). The cation contains a morpholinium ring in a chair conformation. Attached at the two respective chiral centers are a carboxymethyl and a phenyl group, which are *cis* and diequatorial. There are two forms of the cation, which differ in the morpholinium O—C—C—COOH torsion angle by 122·4 (5)° [*A*, 72·7 (4) and *B*, 175·1 (3)°]. In cation *A*, the carboxyl H is in the *syn* or *Z* conformation, and in cation *B*, it is in the *anti* or *E* conformation. It both cases, the carboxy groups form nearly linear O—H···Cl$^-$ contacts, with O···Cl distances 2·982 (3) Å for molecule *A* and 2·956 (3) Å for molecule *B*. The hydroxy group of the *A* molecule donates a hydrogen bond of O···O length 2·668 (4) Å to the water molecule, and the hydroxy group of molecule *B* forms a bifurcated contact involving a chloride ion [O···Cl 3·324 (2) Å] and the carboxy carbonyl oxygen of molecule *A* [O···O 3·096 (3) Å].

Experimental. Colorless needles of (1), m.p. 468 K, were synthesized from sodium (*R*)-norcarnitine and chloroacetophenone in isopropyl alcohol followed by



(1)

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$B_{eq} = 4/3(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta)$.

	x	y	z	B_{eq}(Å^2)
Cl(1)	0·13365 (5)	0	0·64655 (6)	4·67 (2)
Cl(2)	0·57965 (7)	0·3490 (2)	0·81444 (8)	6·00 (2)
O1*A*	0·0341 (2)	0·1514 (4)	−0·1644 (2)	5·18 (6)
O2*A*	0·1046 (2)	−0·0134 (4)	−0·0522 (2)	5·75 (6)
O3*A*	0·0725 (1)	0·2050 (3)	0·1689 (2)	3·16 (4)
O4*A*	0·1606 (1)	0·0493 (3)	0·2842 (2)	4·03 (4)
N1*A*	−0·0260 (2)	0·0662 (3)	0·3537 (2)	3·33 (5)
C1*A*	0·0464 (2)	0·0754 (5)	−0·0691 (2)	3·92 (6)
C2*A*	−0·0225 (2)	0·1154 (5)	0·0224 (2)	3·85 (6)
C3*A*	0·0110 (2)	0·0865 (4)	0·1443 (2)	3·20 (5)
C4*A*	−0·0613 (2)	0·0905 (4)	0·2325 (2)	3·45 (6)
C5*A*	−0·0971 (2)	0·0972 (5)	0·4392 (3)	4·50 (7)
C6*A*	0·0042 (3)	−0·0961 (4)	0·3716 (3)	4·38 (7)
C7*A*	0·0454 (2)	0·1807 (4)	0·3751 (2)	3·11 (5)
C8*A*	0·1140 (2)	0·1859 (4)	0·2797 (2)	3·04 (5)
C9*A*	0·1696 (2)	0·3253 (4)	0·2998 (2)	3·26 (6)
C10*A*	0·2411 (2)	0·3158 (5)	0·3744 (3)	4·43 (7)
C11*A*	0·2918 (2)	0·4424 (6)	0·3962 (3)	5·72 (9)
C12*A*	0·2736 (2)	0·5812 (6)	0·3447 (3)	5·80 (9)
C13*A*	0·2016 (3)	0·5942 (5)	0·2726 (3)	5·78 (9)
C14*A*	0·1501 (2)	0·4653 (5)	0·2499 (3)	4·31 (7)
O1*B*	0·5591 (2)	0·5988 (4)	0·6425 (2)	6·21 (6)
O2*B*	0·5667 (2)	0·7030 (3)	0·4711 (2)	5·43 (6)
O3*B*	0·6542 (1)	0·2914 (3)	0·3255 (2)	3·06 (4)
O4*B*	0·7223 (1)	0·3768 (3)	0·1576 (2)	3·50 (4)
N1*B*	0·5284 (1)	0·4232 (3)	0·1665 (2)	3·07 (4)
C1*B*	0·5760 (2)	0·5903 (4)	0·5296 (3)	3·96 (6)
C2*B*	0·6091 (2)	0·4400 (4)	0·4851 (3)	3·63 (6)
C3*B*	0·6146 (2)	0·4366 (4)	0·3524 (2)	3·03 (5)
C4*B*	0·5253 (2)	0·4458 (4)	0·2963 (2)	3·16 (5)
C5*B*	0·4371 (2)	0·4021 (5)	0·1217 (3)	4·36 (7)
C6*B*	0·5664 (2)	0·5598 (4)	0·1068 (3)	3·90 (6)
C7*B*	0·5781 (2)	0·2788 (4)	0·1408 (2)	3·05 (5)
C8*B*	0·6654 (2)	0·2673 (4)	0·2051 (2)	2·85 (5)
C9*B*	0·6986 (2)	0·1054 (4)	0·1890 (2)	2·98 (5)
C10*B*	0·7299 (2)	0·0608 (4)	0·0803 (3)	3·61 (6)
C11*B*	0·7571 (2)	−0·0874 (4)	0·0614 (3)	4·16 (7)
C12*B*	0·7555 (2)	−0·1930 (4)	0·1510 (3)	4·49 (7)
C13*B*	0·7256 (2)	−0·1498 (4)	0·2587 (3)	4·64 (7)
C14*B*	0·6956 (2)	−0·0023 (4)	0·2771 (3)	3·90 (6)
O1*W*	0·7364 (2)	0·5373 (5)	0·9007 (2)	8·07 (9)

hydrolysis with 2*N* HCl and were crystallized from methanol by vapor diffusion with ethyl ether. Crystal size 0·15 × 0·32 × 0·50 mm, space group from systematic absences 0*k*0 with *k* odd and known chirality of the compound, cell dimensions from setting angles of 25 reflections having $20 < \theta < 25°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 90 s, scan rates varied 0·75–3·30° min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \le h \le 19$, $0 \le k \le 10$, $-14 \le l \le 14$ measured. Data corrected for background, Lorentz and polarization effects. Standard reflections 200, 020, 002 decreased by 6·8%, and linear decay correction was applied. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 86·71%. 3550 total data were collected, and redundant data merged, $R_{int} = 0·027$, to yield 3394 unique data, 3027 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0·02F_o^2)^2]^{-1}$ 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, and not refined. Those bonded to carbon were fixed with C—H 0·95 Å and $B_{iso} = 1·3B_{eq}$ for the C atom. Final $R = 0·037$ (0·044 for all data), $wR = 0·047$, $S = 2·463$ for 370 variables.

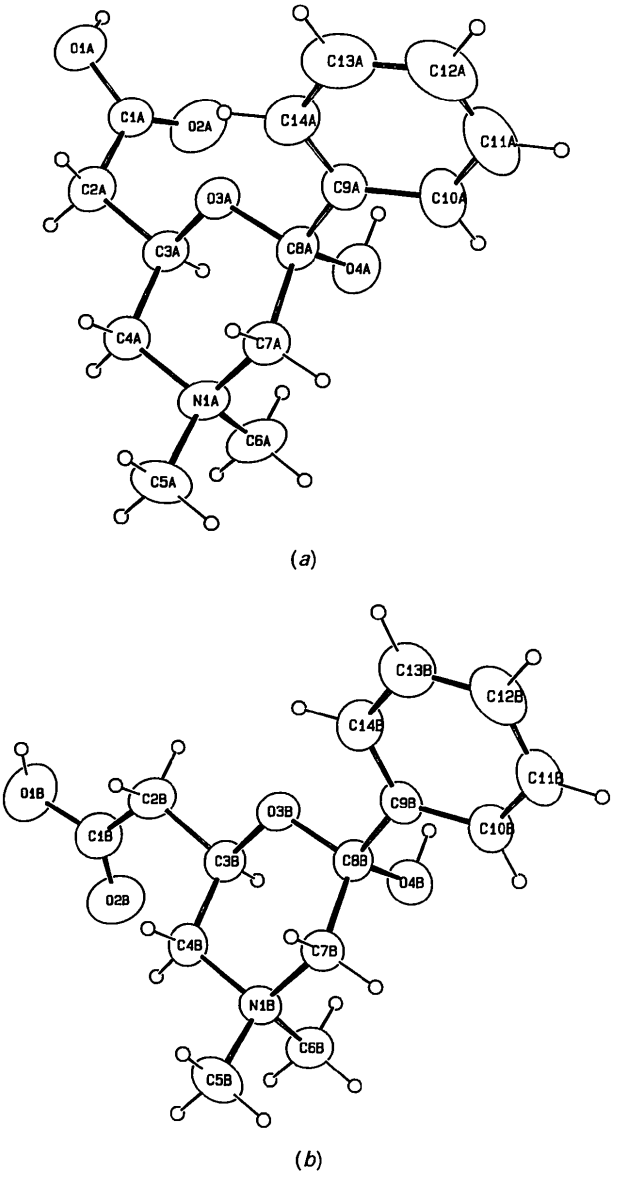

Fig. 1. *ORTEP* (Johnson, 1965) drawings of the two independent cations of the title compound, illustrating thermal ellipsoids at the 50% probability level and H atoms with arbitrary radius.

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

O1*A*—C1*A*	1·292 (4)	O1*B*—C1*B*	1·328 (4)
O2*A*—C1*A*	1·200 (5)	O2*B*—C1*B*	1·199 (4)
O3*A*—C3*A*	1·430 (4)	O3*B*—C3*B*	1·442 (4)
O3*A*—C8*A*	1·428 (3)	O3*B*—C8*B*	1·412 (3)
O4*A*—C8*A*	1·392 (4)	O4*B*—C8*B*	1·411 (4)
N1*A*—C4*A*	1·505 (3)	N1*B*—C4*B*	1·506 (3)
N1*A*—C5*A*	1·505 (4)	N1*B*—C5*B*	1·505 (4)
N1*A*—C6*A*	1·504 (5)	N1*B*—C6*B*	1·497 (4)
N1*A*—C7*A*	1·504 (4)	N1*B*—C7*B*	1·506 (4)
C1*A*—C2*A*	1·544 (4)	C1*B*—C2*B*	1·499 (5)
C2*A*—C3*A*	1·509 (4)	C2*B*—C3*B*	1·529 (4)
C3*A*—C4*A*	1·515 (4)	C3*B*—C4*B*	1·516 (4)
C7*A*—C8*A*	1·533 (4)	C7*B*—C8*B*	1·533 (4)
C8*A*—C9*A*	1·505 (5)	C8*B*—C9*B*	1·515 (4)
C9*A*—C10*A*	1·391 (4)	C9*B*—C10*B*	1·398 (4)
C9*A*—C14*A*	1·381 (5)	C9*B*—C14*B*	1·383 (4)
C10*A*—C11*A*	1·375 (6)	C10*B*—C11*B*	1·378 (5)
C11*A*—C12*A*	1·374 (7)	C11*B*—C12*B*	1·381 (5)
C12*A*—C13*A*	1·384 (6)	C12*B*—C13*B*	1·378 (5)
C13*A*—C14*A*	1·399 (6)	C13*B*—C14*B*	1·385 (5)
C3*A*—O3*A*—C8*A*	112·4 (2)	C9*A*—C10*A*—C11*A*	120·6 (4)
C4*A*—N1*A*—C5*A*	108·6 (2)	C10*A*—C11*A*—C12*A*	121·1 (3)
C4*A*—N1*A*—C6*A*	111·6 (2)	C11*A*—C12*A*—C13*A*	119·2 (4)
C4*A*—N1*A*—C7*A*	108·4 (2)	C12*A*—C13*A*—C14*A*	119·8 (4)
C5*A*—N1*A*—C6*A*	107·9 (3)	C9*A*—C14*A*—C13*A*	120·8 (3)
C5*A*—N1*A*—C7*A*	108·1 (2)	C3*B*—O3*B*—C8*B*	113·4 (2)
C6*A*—N1*A*—C7*A*	112·2 (2)	C4*B*—N1*B*—C5*B*	108·4 (2)
O1*A*—C1*A*—O2*A*	124·9 (3)	C4*B*—N1*B*—C6*B*	111·4 (2)
O1*A*—C1*A*—C2*A*	111·4 (3)	C4*B*—N1*B*—C7*B*	109·0 (2)
O2*A*—C1*A*—C2*A*	123·7 (3)	C5*B*—N1*B*—C6*B*	108·1 (2)
C1*A*—C2*A*—C3*A*	111·3 (3)	C5*B*—N1*B*—C7*B*	107·9 (2)
O3*A*—C3*A*—C2*A*	106·4 (2)	C6*B*—N1*B*—C7*B*	111·9 (2)
O3*A*—C3*A*—C4*A*	110·0 (2)	O1*B*—C1*B*—O2*B*	118·6 (3)
C2*A*—C3*A*—C4*A*	111·7 (2)	O1*B*—C1*B*—C2*B*	116·9 (3)
N1*A*—C4*A*—C3*A*	110·7 (2)	O2*B*—C1*B*—C2*B*	124·4 (3)
N1*A*—C7*A*—C8*A*	114·3 (2)	C1*B*—C2*B*—C3*B*	112·3 (3)
O3*A*—C8*A*—O4*A*	111·1 (2)	O3*B*—C3*B*—C2*B*	105·0 (2)
O3*A*—C8*A*—C7*A*	109·5 (2)	O3*B*—C3*B*—C4*B*	109·9 (2)
O3*A*—C8*A*—C9*A*	107·0 (2)	C2*B*—C3*B*—C4*B*	111·3 (2)
O4*A*—C8*A*—C7*A*	107·9 (2)	N1*B*—C4*B*—C3*B*	112·1 (2)
O4*A*—C8*A*—C9*A*	113·1 (2)	N1*B*—C7*B*—C8*B*	114·0 (2)
C7*A*—C8*A*—C9*A*	108·1 (2)	O3*B*—C8*B*—O4*B*	111·2 (2)
C8*A*—C9*A*—C10*A*	119·5 (3)	O3*B*—C8*B*—C7*B*	110·3 (2)
C8*A*—C9*A*—C14*A*	122·0 (3)	O3*B*—C8*B*—C9*B*	107·7 (2)
C10*A*—C9*A*—C14*A*	118·5 (3)	O4*B*—C8*B*—C7*B*	108·4 (2)
O4*B*—C8*B*—C9*B*	111·9 (2)	C9*B*—C10*B*—C11*B*	120·7 (3)
C7*B*—C8*B*—C9*B*	107·3 (2)	C10*B*—C11*B*—C12*B*	120·0 (3)
C8*B*—C9*B*—C10*B*	119·3 (3)	C11*B*—C12*B*—C13*B*	119·7 (3)
C8*B*—C9*B*—C14*B*	122·0 (3)	C12*B*—C13*B*—C14*B*	120·5 (3)
C10*B*—C9*B*—C14*B*	118·6 (3)	C9*B*—C14*B*—C13*B*	120·4 (3)

Maximum shift $0{\cdot}04\sigma$ in the final cycle, max. residual density 0·36 e Å^{-3}, min. −0·26 e Å^{-3} indicative of possible slight disorder of the carboxyl group of cation *A*, extinction coefficient $g = 1{\cdot}8\ (3) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Coordinates are given in Table 1;* bond distances and angles are given in Table 2. The two independent cations are illustrated in Fig. 1, and a stereoview of the unit cell is shown in Fig. 2. Refinement of the enantiomorphous structure under identical conditions yielded $R = 0{\cdot}041$, $wR = 0{\cdot}053$, and $S = 2{\cdot}753$. The absolute configuration thus determined agrees with the known configuration of the starting materials.

* Tables of H-atom coordinates, least-squares planes, torsion angles, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52596 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

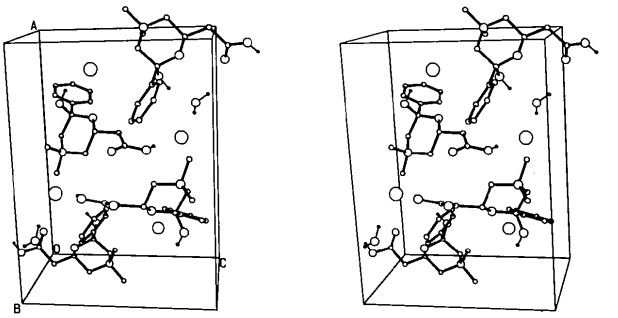

Fig. 2. Stereoscopic view of the unit cell.

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 (1987); as model enzymatic reaction intermediates: Colucci & Gandour (1988); structure of *rel*-(2*S*,6*S*)-2-(6-hydroxy-4,4,6-trimethyl)morpholinomenthanesulfonate: Colucci, Fronczek, Gandour & Watkins (1988); structure of (2*S*,6*R*)-6-carboxymethyl-2-hydroxy-2-ethyl-4,4-dimethylmorpholinium bromide: Blackwell, Fronczek & Gandour (1989).

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