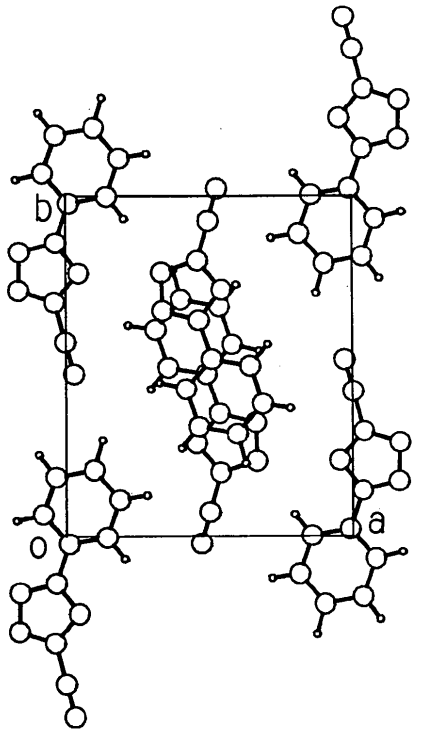

Fig. 2. Projection of the crystal structure along the *c* axis.

full-matrix least-squares method with anisotropic temperature factors for non-H atoms. Function minimized $\sum w(|(|F_o|)^2 - (|F_c|)^2|)^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}014(F_o)^2]$, $\sigma(F_o)$ determined from counting statistics. All H atoms located from the difference map and refined, the initial thermal parameters set at equivalent isotropic thermal parameter of each bonded atom. Final discrepancy indices $R = 0{\cdot}059$, $wR = 0{\cdot}050$, $S = 1{\cdot}301$ for 730 reflexions with $F > 3\sigma(F)$. Maximum $\Delta/\sigma = 0{\cdot}17$ in final least-squares cycle. Final difference Fourier map showed no residuals greater than 0·29 e $Å^{-3}$. All calculations performed using Panafacom computer with *RCRYSTAN* (Rigaku Corporation, 1985) X-ray analysis program system. 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 listed in Table 2. Fig. 1 shows the thermal-ellipsoid plot of the molecule with atomic labelling. Fig. 2 is the crystal structure.

Related literature. The title compound is one of the breakdown products from 6-phenyl-[1,2,3]triazolo-[4,5-*e*]-1,2,3,4-tetrazine (Kaihoh, Itoh, Yamaguchi & Ohsawa, 1988). See also Moderhack (1981) for the preparation of related compounds.

* Lists of structure amplitudes, anisotropic thermal parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52565 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KAIHOH, T., ITOH, T., YAMAGUCHI, K. & OHSAWA, (1988). *J. Chem. Soc. Chem. Commun.* pp. 1608–1609.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MODERHACK, D. (1981). *Chem. Ztg*, **105**, 194–195.

Rigaku Corporation (1985). *RCRYSTAN. X-ray Analysis Program System.* Rigaku Corporation, Tokyo, Japan.

YAO, J.-X., ZHENG, C.-D., QIAN, J.-Z., HAN, F.-S., GU, Y.-X. & FAN, H.-F. (1985). *SAPI85. A Computer Program for Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Institute of Physics, Academia Sinica, Beijing, People's Republic of China.

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Structure of a Cyclooctatetraene Derivative

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Abstract. 1-Carbamoyl-2-phenyl-9-oxabicyclo[6.1.0]-nona-2,4,6-triene, $C_{15}H_{13}NO_2$, $M_r = 239{\cdot}3$, triclinic, $P\bar{1}$, $a = 8{\cdot}833$ (3), $b = 10{\cdot}754$ (5), $c = 8{\cdot}204$ (1) Å, $\alpha = 93{\cdot}52$ (3), $\beta = 105{\cdot}12$ (3)°, $\gamma = 125{\cdot}20$ (1)°, $V = 592{\cdot}3$ (4) $Å^3$, $Z = 2$, $D_x = 1{\cdot}342$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·54050 Å, $\mu = 0{\cdot}733$ mm^{-1}, $F(000) = 252$, $T =$

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
C(1)	0·6357 (4)	0·3484 (3)	0·1981 (4)	3·42 (13)
C(2)	0·4834 (4)	0·1725 (3)	0·1422 (4)	3·11 (12)
C(3)	0·5214 (5)	0·0821 (4)	0·2192 (4)	3·66 (14)
C(4)	0·6837 (5)	0·1288 (4)	0·3786 (4)	4·32 (15)
C(5)	0·7337 (5)	0·2091 (4)	0·5362 (5)	4·56 (16)
C(6)	0·6479 (5)	0·2811 (4)	0·5871 (4)	4·92 (17)
C(7)	0·6257 (5)	0·3784 (4)	0·5114 (4)	4·90 (17)
C(8)	0·7034 (5)	0·4445 (4)	0·3753 (4)	4·08 (15)
O(9)	0·5712 (3)	0·4420 (2)	0·2242 (3)	4·54 (10)
C(10)	0·7828 (5)	0·4114 (3)	0·1050 (4)	3·90 (14)
O(11)	0·8487 (4)	0·3414 (2)	0·0784 (3)	6·99 (14)
N(12)	0·8333 (4)	0·5417 (3)	0·0551 (4)	4·89 (14)
C(13)	0·3039 (4)	0·1039 (3)	−0·0160 (4)	3·38 (13)
C(14)	0·1357 (5)	−0·0531 (3)	−0·0552 (4)	3·81 (14)
C(15)	−0·0340 (5)	−0·1155 (4)	−0·1984 (5)	4·41 (15)
C(16)	−0·0430 (6)	−0·0240 (5)	−0·3046 (5)	5·17 (18)
C(17)	0·1232 (5)	0·1312 (4)	−0·2695 (4)	5·06 (17)
C(18)	0·2942 (5)	0·1947 (4)	−0·1265 (4)	4·17 (15)

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

C(1)—C(2)	1·495 (4)	C(1)—C(10)	1·514 (6)
C(2)—C(3)	1·340 (6)	C(10)—O(11)	1·226 (7)
C(3)—C(4)	1·460 (5)	C(10)—N(12)	1·332 (5)
C(4)—C(5)	1·319 (6)	C(2)—C(13)	1·484 (4)
C(5)—C(6)	1·467 (9)	C(13)—C(14)	1·398 (4)
C(6)—C(7)	1·331 (8)	C(14)—C(15)	1·379 (5)
C(7)—C(8)	1·469 (6)	C(15)—C(16)	1·375 (7)
C(8)—C(1)	1·480 (5)	C(16)—C(17)	1·386 (5)
C(8)—O(9)	1·451 (5)	C(17)—C(18)	1·383 (5)
O(9)—C(1)	1·444 (6)	C(18)—C(13)	1·395 (6)

C(1)—C(2)—C(3)	120·4 (2)	C(2)—C(1)—C(10)	113·9 (3)
C(1)—C(2)—C(13)	116·9 (3)	C(8)—C(1)—C(10)	117·2 (2)
C(3)—C(2)—C(13)	122·0 (2)	O(9)—C(1)—C(10)	114·3 (3)
C(2)—C(3)—C(4)	129·1 (3)	C(1)—C(10)—O(11)	119·4 (1)
C(3)—C(4)—C(5)	127·5 (5)	C(1)—C(10)—N(12)	117·3 (4)
C(4)—C(5)—C(6)	127·5 (4)	O(11)—C(10)—N(12)	123·1 (4)
C(5)—C(6)—C(7)	125·9 (4)	C(2)—C(13)—C(14)	120·6 (3)
C(6)—C(7)—C(8)	124·9 (5)	C(2)—C(13)—C(18)	121·4 (2)
C(7)—C(8)—C(1)	123·8 (2)	C(13)—C(14)—C(15)	120·8 (3)
C(8)—C(1)—C(2)	122·6 (3)	C(14)—C(15)—C(16)	120·8 (3)
C(8)—O(9)—C(1)	61·5 (2)	C(15)—C(16)—C(17)	119·2 (3)
C(8)—C(1)—O(9)	59·4 (2)	C(17)—C(18)—C(13)	120·9 (3)
C(1)—C(8)—O(9)	59·0 (2)	C(18)—C(13)—C(14)	117·8 (1)

293 K, final $R = 0·055$ for 1539 unique observed reflexions. The eight-membered ring is in a tab-shaped conformation, and the epoxy oxygen takes an extended *trans* conformation. The puckering parameter, the angle between the base plane C(2)—C(3)—C(6)—C(7) and the C(1)—C(2)—C(7)—C(8) and C(3)—C(4)—C(5)—C(6) planes, is 49·9 (3) and 39·7 (3)° respectively. An intermolecular hydrogen bond between N(12) and O(11)′($-x+2$, $-y+1$, $-z$), with a distance of 2·894 (6) Å, is observed.

Experimental. A pale-yellow prism, 0·40 × 0·10 × 0·55 mm, by recrystallization from CH_3OH. Rigaku AFC-5 four-circle diffractometer used with θ–2θ-scan method, ω-scan width $(1·3 + 0·41\tan\theta)°$ and scan speed 16° min^{-1}. Lattice parameters obtained from least-squares analysis of 20 reflexions with 2θ values ranging from 59 to 61°. Out of 1951 reflexions scanned within index range h −9–9, k −12–12, l 0–9 up to $(\sin\theta)/\lambda \le 0·56$ Å^{-1} including 143 equivalent reflexions ($R_{int} = 0·019$), 1768 unique reflexions classified as observed. Three standard reflexions measured every 150 reflexions, no significant intensity variation. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using the direct-methods program package *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985), a version of

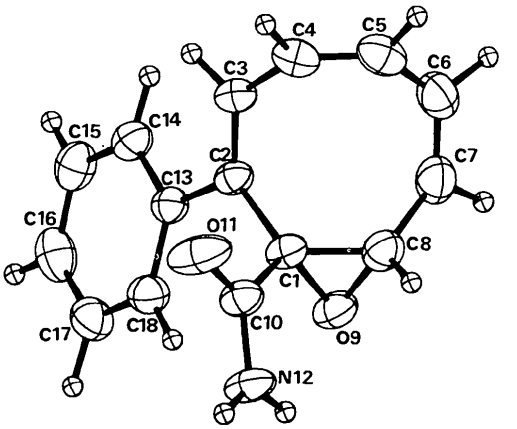

Fig. 1. Thermal-ellipsoid plot. Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

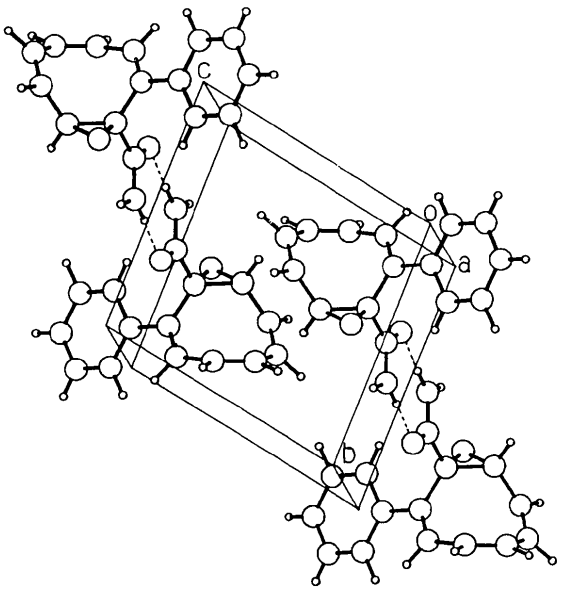

Fig. 2. The crystal structure.

*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.

References

ADAM, W. & KLUG, G. (1985). *Tetrahedron*, **41**, 2045–2056.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Rigaku Corporation (1985). *RCRYSTAN. X-ray Analysis Program System*. Rigaku Corporation, Tokyo, Japan.

YAO, J.-X., ZHENG, C.-D., QIAN, J.-Z., HAN, F.-S., GU, Y.-X. & FAN, H.-F. (1985). *SAPI*85. *A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Institute of Physics, Academia Sinica, Beijing, People's Republic of China.

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

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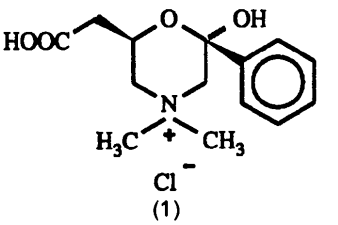
Abstract. $C_{14}H_{20}NO_4^+.Cl^-.\frac{1}{2}H_2O$, $M_r = 310{\cdot}8$, monoclinic, $P2_1$, $a = 15{\cdot}425\ (2)$, $b = 8{\cdot}725\ (2)$, $c = 11{\cdot}490\ (3)$ Å, $\beta = 90{\cdot}61\ (2)°$, $V = 1546{\cdot}2\ (10)$ Å^3, $Z = 4$, $D_x = 1{\cdot}335$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 23{\cdot}6$ 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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