

Discussion. The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the packing in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors β_{ik} for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

The asymmetric unit contains one molecule. The X-ray investigation of *rac*-(3) (m.p. 399 K) clearly demonstrates the *relative configuration* 4*R*,4*aS*,8*aR* for the cyclic acetal. The atoms C(4*a*) and C(4) in structure (3) during further transformation become C(6) and C(7) in β -turmerone (1) and thus allow unambiguous assignment for the latter.

The two six-membered rings both have the chair conformation. Planes through the following groups of atoms have been calculated: plane (A): O(1), C(2), C(8*a*); (B): C(2), C(3), C(4*a*), C(8*a*) [$\chi^2 = 39$, planar within 0.007 (2) Å]; (C): C(3), C(4), C(4*a*); (D): C(4*a*), C(5), C(6); (E): C(4*a*), C(6), C(7), C(8*a*) [$\chi^2 = 1738$, planar within 0.042 (2) Å]; (F): C(7), C(8), C(8*a*). The dihedral angles (A)/(B) 51.9 (5), (A)/(C) 2.5 (5), (B)/(C) 49.5 (5) and (E)/(F) 48.8 (5), (D)/(F) 3.2 (5), (D)/(E) 47.7 (5)° indicate similar chairs for these rings and the dihedral angles (A)/(F) 2.0 (5), (B)/(E) 5.4 (5), (C)/(D) 2.1 (5)° show that corresponding parts of the two chairs are coplanar. The group C(6), C(7), C(8), O(2) is planar within 0.002 (2) Å.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42777 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

A short intermolecular O...O distance [O(2)(1 - x , 1 - y , 1 - z)...O(3)(x , y , z) 2.800 (2) Å] indicates a hydrogen bond. If one places the H atom bound to O(3) on the line connecting O(2)(1 - x , 1 - y , 1 - z) and O(3)(x , y , z) at a distance of 0.95 Å from atom O(3) the following distance and angle result: H[O(3)]...O(2)(1 - x , 1 - y , 1 - z) 1.85 Å and C(8*a*)-O(3)-H[O(3)] 104°. Each molecule is connected *via* two symmetrically equivalent hydrogen bonds (dotted lines in Fig. 2) to one neighbouring molecule. That means pairs of two molecules form subunits in the crystal which is otherwise held together by van der Waals interactions.

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Structure of the (+)-*N*-Methylephedrine* Salt of (+)-(1*S*,1*aR*,6*bS*)-1,1*a*,2,6*b*-Tetrahydrocycloprop[*a*]indene-1-carboxylic Acid

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Abstract. C₁₁H₁₈NO⁺.C₁₁H₉O₂⁻, $M_r = 353.47$, orthorhombic, $P2_12_12_1$, $a = 11.994$ (1), $b = 27.578$ (1), $c = 5.814$ (1) Å, $V = 1923.1$ Å³, $Z = 4$, $D_x = 1.22$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 6.06$ cm⁻¹, $F(000) = 760$, $T = 295$ K, $R = 0.038$ for 1686 ob-

served reflections. The complex consists of discrete [C₁₁H₁₈NO]⁺ cations and [C₁₁H₉O₂]⁻ anions linked by two hydrogen bonds to form dimers. There are bands composed of hydrophilic and hydrophobic residues running parallel to the *ac* plane. The absolute configuration of the title anion was deduced from the known absolute configuration of (+)-*N*-methylephedrine.

* (+)-2-Dimethylamino-1-phenylpropanol.

Introduction. Reaction of an alkyl diazoacetate with an olefin catalyzed by a chiral Schiff base-copper complex gives an optically active alkyl cyclopropane-carboxylate (Nozaki, Takaya, Moriuti & Noyori, 1968; Aratani, Yoneyoshi & Nagase, 1982; Aratani, 1985). The title anion, (+)-1,1a,2,6b-tetrahydrocycloprop[*a*]indene-1-carboxylic acid, was prepared from ethyl diazoacetate and indene. In order to elucidate the steric course of the reaction, the absolute configuration of the title compound has been determined by X-ray analysis.

Experimental. Crystals suitable for X-ray work were obtained from ethyl acetate. Crystal used 0.4 × 0.3 × 0.2 mm. Enraf-Nonius CAD-4 diffractometer. Cell dimensions refined by least-squares fitting of θ values of 25 reflections with $20 < \theta < 27^\circ$. ω - 2θ scan, scan angle $0.7^\circ + 0.15^\circ \tan\theta$, three reference reflections monitored every 60 min showed no significant changes in intensity. Correction for Lorentz and polarization effects, empirical absorption correction (transmission factors 0.858–0.999, av. 0.959). 2132 unique reflections measured with $2 < \theta < 70^\circ$; 1686 with $I > 3\sigma(I)$ used in structure determination; h 0 to 14, k 0 to 33, l 0 to 7. Structure solved by direct methods with *MITHRIL* (Gilmore, 1984). H atoms located from difference Fourier map. Structure refined by full-matrix least squares; $\sum w(|F_o| - |F_c|)^2$ minimized. H positions and thermal parameters refined isotropically. Final $R = 0.038$, $wR = 0.050$ for 344 variables, $w = 4I/[\sigma^2(I) + (0.05I)^2]$, $S = 1.675$, $(\Delta/\sigma)_{\max} = 0.24$, extinction coefficient refined to $5.4(3) \times 10^{-6}$. Max. and min. peaks in final difference Fourier map 0.14 and $-0.13 \text{ e } \text{\AA}^{-3}$, respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations carried out using *MITHRIL* and *SDP* program system (Frenz, 1983).

Discussion. Final atomic coordinates are listed in Table 1.* Bond distances and angles are given in Table 2. An *ORTEP* drawing (Johnson, 1976) of the title compound with the numbering system is shown in Fig. 1. The structure consists of a (+)-*N*-methylephedrine cation and a (+)-1,1a,2,6b-tetrahydrocycloprop[*a*]indene-1-carboxylic acid anion held together by two hydrogen bonds.

The absolute structure of the anion moiety was determined to be 1*S*, 1*aR* and 6*bS* from the known configuration (1*S*, 2*R*) of (+)-*N*-methylephedrine. In the anion moiety, the aromatic six-membered ring is planar

within 0.011 (3) Å. The C(3) atom is almost in the plane, but C(10) and C(11) deviate by 0.037 (3) and $-0.030(3)$ Å, respectively, from the plane.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters for non-H atoms

$$B_{\text{eq}} = \frac{4}{3} \sum_{i=1}^3 \beta_i \mathbf{a}_i \cdot \mathbf{a}_i$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
O(1)	0.1388 (1)	0.18927 (6)	0.6161 (4)	4.04 (4)
O(2)	0.2975 (1)	0.16951 (6)	0.4398 (4)	4.46 (4)
O(3)	0.2251 (1)	0.26446 (6)	0.8321 (3)	3.80 (4)
N(1)	0.4334 (2)	0.21508 (6)	0.7102 (4)	3.09 (4)
C(1)	0.1949 (2)	0.16322 (8)	0.4835 (5)	3.16 (4)
C(2)	0.1382 (2)	0.12181 (7)	0.3697 (5)	2.97 (4)
C(3)	0.2019 (2)	0.07445 (8)	0.3473 (5)	3.39 (5)
C(4)	0.1317 (2)	0.03040 (8)	0.3209 (5)	3.12 (5)
C(5)	0.1205 (2)	-0.00845 (8)	0.4693 (5)	4.18 (6)
C(6)	0.0472 (3)	-0.04597 (8)	0.4104 (7)	5.02 (7)
C(7)	-0.0111 (2)	-0.04419 (9)	0.2056 (6)	4.66 (6)
C(8)	-0.0005 (2)	-0.00511 (9)	0.0619 (5)	4.16 (5)
C(9)	0.0713 (2)	0.03238 (8)	0.1176 (5)	3.39 (5)
C(10)	0.0998 (3)	0.07757 (9)	-0.0173 (5)	4.77 (6)
C(11)	0.1795 (2)	0.10477 (8)	0.1386 (5)	3.96 (5)
C(12)	0.4150 (3)	0.1888 (1)	0.9296 (6)	5.04 (6)
C(13)	0.5436 (2)	0.20121 (9)	0.6127 (6)	4.45 (6)
C(14)	0.4225 (2)	0.26998 (8)	0.7245 (4)	2.97 (4)
C(15)	0.4655 (2)	0.2900 (1)	0.9493 (6)	4.81 (6)
C(16)	0.3010 (2)	0.28397 (8)	0.6698 (4)	2.99 (4)
C(17)	0.2900 (2)	0.33908 (8)	0.6599 (5)	3.14 (4)
C(18)	0.2387 (3)	0.3646 (1)	0.8341 (6)	4.68 (6)
C(19)	0.2333 (3)	0.4152 (1)	0.8259 (6)	5.67 (7)
C(20)	0.2792 (2)	0.43969 (9)	0.6407 (7)	5.41 (7)
C(21)	0.3281 (3)	0.4143 (1)	0.4660 (6)	5.24 (7)
C(22)	0.3335 (2)	0.36404 (9)	0.4743 (5)	4.25 (6)

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

O(1)–C(1)	1.250 (2)	C(6)–C(7)	1.381 (5)
O(2)–C(1)	1.269 (2)	C(7)–C(8)	1.369 (4)
O(3)–C(16)	1.418 (3)	C(8)–C(9)	1.384 (3)
N(1)–C(12)	1.483 (3)	C(9)–C(10)	1.511 (3)
N(1)–C(13)	1.488 (3)	C(10)–C(11)	1.516 (4)
N(1)–C(14)	1.522 (2)	C(14)–C(15)	1.510 (4)
C(1)–C(2)	1.485 (3)	C(14)–C(16)	1.541 (3)
C(2)–C(3)	1.519 (3)	C(16)–C(17)	1.526 (3)
C(2)–C(11)	1.507 (3)	C(17)–C(18)	1.378 (3)
C(3)–C(4)	1.486 (3)	C(17)–C(22)	1.382 (3)
C(3)–C(11)	1.498 (4)	C(18)–C(19)	1.397 (4)
C(4)–C(5)	1.383 (3)	C(19)–C(20)	1.386 (5)
C(4)–C(9)	1.387 (3)	C(20)–C(21)	1.367 (4)
C(5)–C(6)	1.400 (4)	C(21)–C(22)	1.387 (4)
C(12)–N(1)–C(13)	109.5 (2)	C(4)–C(9)–C(10)	110.9 (2)
C(12)–N(1)–C(14)	115.2 (2)	C(8)–C(9)–C(10)	129.4 (2)
C(13)–N(1)–C(14)	110.6 (2)	C(9)–C(10)–C(11)	103.9 (2)
O(1)–C(1)–O(2)	124.5 (2)	C(2)–C(11)–C(3)	60.7 (2)
O(1)–C(1)–C(2)	118.1 (2)	C(2)–C(11)–C(10)	118.7 (2)
O(2)–C(1)–C(2)	117.4 (2)	C(3)–C(11)–C(10)	108.7 (2)
C(1)–C(2)–C(3)	118.0 (2)	N(1)–C(14)–C(15)	112.4 (2)
C(1)–C(2)–C(11)	119.1 (2)	N(1)–C(14)–C(16)	108.6 (2)
C(3)–C(2)–C(11)	59.3 (2)	C(15)–C(14)–C(16)	114.2 (2)
C(2)–C(3)–C(4)	115.3 (2)	O(3)–C(16)–C(14)	112.0 (2)
C(2)–C(3)–C(11)	60.0 (2)	O(3)–C(16)–C(17)	110.3 (2)
C(4)–C(3)–C(11)	105.7 (2)	C(14)–C(16)–C(17)	109.8 (2)
C(3)–C(4)–C(5)	128.6 (2)	C(16)–C(17)–C(18)	121.3 (2)
C(3)–C(4)–C(9)	110.6 (2)	C(16)–C(17)–C(22)	119.5 (2)
C(5)–C(4)–C(9)	120.8 (2)	C(18)–C(17)–C(22)	119.2 (2)
C(4)–C(5)–C(6)	118.7 (3)	C(17)–C(18)–C(19)	120.4 (3)
C(5)–C(6)–C(7)	120.1 (3)	C(18)–C(19)–C(20)	119.7 (3)
C(6)–C(7)–C(8)	120.5 (2)	C(19)–C(20)–C(21)	119.8 (2)
C(7)–C(8)–C(9)	120.2 (3)	C(20)–C(21)–C(22)	120.4 (3)
C(4)–C(9)–C(8)	119.6 (2)	C(17)–C(22)–C(21)	120.5 (3)

* Lists of structure factors, anisotropic thermal parameters for non-H atoms and positional and thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42772 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

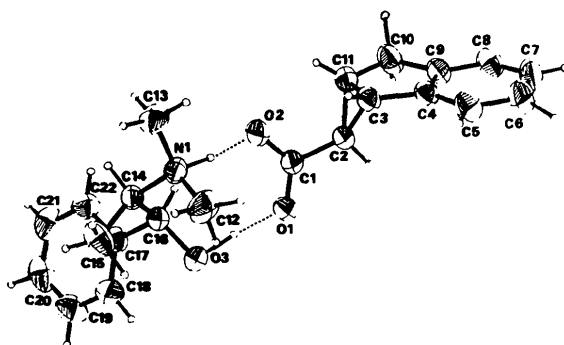


Fig. 1. An ORTEP drawing (Johnson, 1976) of the title complex showing the atom numbering. The ellipsoids are drawn at the 50% probability level.

The conformation of the (+)-*N*-methylephedrine moiety is in its most extended form, the torsion angle N(1)–C(14)–C(16)–C(17) being $-174.6(2)^\circ$ which is close to those values in other ephedrine derivatives such as (–)-ephedrine hydrochloride, 164.4° (Bergin, 1971), (–)-ephedrine dihydrogen phosphate, 162.8° (Hearn & Bugg, 1972) and *p*-hydroxyephedrine hydrochloride, 178.1° (Dattagupta, Pattanayek & Saha, 1981).

The crystal structure viewed along the *c* axis is shown in Fig. 2. The cation–anion pairs are formed through the two hydrogen bonds, O(3)–H(O3)···O(1) [$2.636(2)$ Å] and N(1)–H(N1)···O(2) [$2.590(2)$ Å]. There are bands composed of hydrophilic (near $y = \frac{1}{4}$)

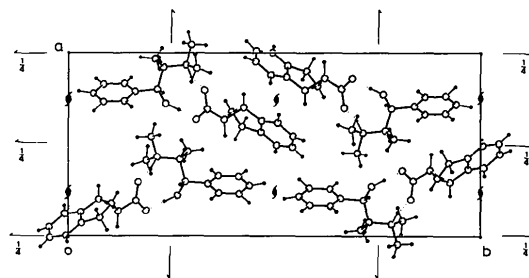


Fig. 2. The crystal structure viewed along the *c* axis.

and hydrophobic (near $y = \frac{1}{2}$) residues running parallel to the *ac* plane.

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Structure of 1',3,3'-Trimethyloxazolidine-5'-spiro-5'-hydantoin-2,4-dione*

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Abstract. $C_8H_9N_3O_5$, $M_r = 227.18$, hexagonal, $P6_1$, $a = 9.385(3)$, $c = 19.06(4)$ Å, $V = 1453.9(7)$ Å³, $Z = 6$, $D_x = 1.557$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.87$ cm⁻¹, $F(000) = 708$, $T = 138(2)$ K, $R = 0.031$ for 783 observed reflections. Room-temperature cell constants were also refined: $a = 9.449(4)$, $c = 19.20(4)$ Å, $V = 1484.6(9)$ Å³. No significant dif-

ferences were apparent between space groups $P6_1$ and $P6_5$. This unusual compound was formed as a slightly soluble product of electrochemical oxidation of 1,3,7,9-tetramethyluric acid in aqueous solution at pH 2. Both rings of the title compound are planar and are almost perpendicular to each other. The N–C(O)–N–C(O) bond lengths of the hydantoin ring show the characteristic short–long–short pattern. The C–N bond involving the spiro atom is extremely short

* Hydantoin is 2,4-imidazolidinedione.