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Acta Cryst. (1984). **C40**, 1914–1915

The Structure of (1*RS*,7*SR*,8*aRS*)-4,7-Dimethyl-1,2,3,5,6,7,8,8*a*-octahydro-1-naphthoic Acid, C₁₃H₂₀O₂*

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(Received 29 March 1984; accepted 2 July 1984)

Abstract. $M_r = 208.3$, monoclinic, $P2_1/n$, $a = 9.597$ (2), $b = 9.461$ (1), $c = 13.065$ (2) Å, $\beta = 93.75$ (1)°, $V = 1183.7$ Å³, D_m (flotation) = 1.152, $D_x = 1.169$ Mg m⁻³, $Z = 4$, $\mu(\text{Mo } K\alpha) = 1.03$ mm⁻¹, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $F(000) = 456.0$, $T = 293$ K. $R = 0.043$ for 575 observed reflections. Ring *A* has the half-chair form while ring *B* has the chair form. The dihedral angle between ring planes is 46.8 (7)°. Bond lengths and angles resemble those in capsidiol.

Introduction. The title compound (I) was synthesized to correlate with a number of naturally occurring compounds having the carbon skeleton of cadalene. It was obtained by reacting a conjugated diene with ethyl acrylate followed by saponification of the Diels–Alder adduct (Shankaran & Rao, 1983).

Experimental. Crystal $\sim 0.30 \times 0.30 \times 0.22$ mm. Nonius CAD-4F-11 M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min⁻¹, $\theta < 24^\circ$, h 0 to 10, k 0 to 10, l -14 to 14. 1982 reflections collected, 575 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 25 reflections ($15^\circ < 2\theta < 35^\circ$), three standard reflections (30 $\bar{3}$, $\bar{1}12$ and $\bar{1}\bar{2}5$) every 2000 s, 4% variation in intensity. No corrections for absorption. Structure solved by direct methods using Sayre-equation program (Long, 1965). Full-matrix least-squares refinement (on F) of scale factors, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, initial H positions calculated by stereochemistry) converged to $R = 0.043$ and $R_w = 0.040$, $S = 2.09$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (3.5 + 1.0|F_o| +$

$0.025|F_o|^2)^{-1}$. $(\Delta/\sigma)_{\max} = 0.1$. Final $\Delta\rho$ excursions < 10.3 e Å⁻³. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs LALS (Gantzel, Sparks & Trueblood, 1961) for refinement, NRC-12 and NRC-22 (Ahmed, Hall, Pippy & Huber, 1973) for bond lengths, bond angles, mean planes and torsion angles.†

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1. Bond lengths and bond angles involving the non-hydrogen atoms are given in Table 2. Fig. 1 shows the chemical formula together with the numbering of the atoms. Fig. 2 shows the packing of the molecules in the unit cell.

The purpose of this work was to determine the stereochemistry; the nomenclature of the compound being established subsequently. Ring *A* is in a half-chair conformation owing to C(4a) being sp^2 -hybridized, while ring *B* is in the chair conformation (Hilderbrandt, Wieser & Montgomery, 1973). The dihedral angle between the mean planes of the two rings is 46.8 (7)°. The molecules form dimers around the centre of symmetry by hydrogen bonding through carboxylic groups, the O–H...O distance being 2.639 (6) Å [O–H = 1.08 (8), H...O = 1.57 (8) Å, \angle O–H...O = 171 (7)°]. All other contacts are of the normal van der Waals type.

† Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39613 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* NCL Communication No. 3506.

Table 1. Fractional atomic coordinates ($\times 10^4$) with their standard deviations in parentheses and equivalent values of the isotropic temperature-factor coefficients

$$B_{eq} = \frac{1}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}accos\beta).$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
C(1)	-322 (6)	1840 (6)	2225 (4)	3.67
C(2)	-1570 (6)	1357 (7)	2801 (5)	4.90
C(3)	-1735 (7)	2394 (8)	3673 (5)	5.10
C(4)	-421 (6)	2665 (6)	4317 (4)	3.84
C(5)	2195 (7)	2486 (7)	4596 (4)	4.54
C(6)	2838 (7)	1032 (8)	4743 (5)	5.15
C(7)	3040 (6)	312 (6)	3718 (5)	4.16
C(8)	1692 (6)	305 (6)	3035 (5)	4.10
C(8a)	1039 (5)	1781 (6)	2922 (3)	3.24
C(4a)	826 (5)	2401 (5)	3974 (4)	3.67
C(11)	-649 (7)	3261 (7)	5358 (4)	5.84
C(12)	-172 (5)	996 (7)	1254 (4)	3.58
C(13)	4290 (6)	953 (8)	3163 (5)	5.88
O(1)	672 (4)	1570 (4)	636 (3)	4.70
O(2)	-779 (4)	-103 (4)	1075 (3)	4.99

 Table 2. Intramolecular bond lengths (\AA) and angles ($^\circ$) with their standard deviations in parentheses

C(1)–C(8a)	1.544 (7)	C(1)–C(12)	1.514 (8)
C(2)–C(1)	1.526 (8)	C(3)–C(2)	1.520 (10)
C(4)–C(3)	1.492 (9)	C(4)–C(4a)	1.329 (8)
C(4)–C(11)	1.502 (8)	C(5)–C(4a)	1.501 (8)
C(6)–C(5)	1.515 (10)	C(7)–C(6)	1.526 (9)
C(7)–C(13)	1.564 (9)	C(8)–C(7)	1.523 (9)
C(8)–C(8a)	1.534 (8)	C(12)–O(1)	1.299 (7)
C(12)–O(2)	1.207 (7)	C(8a)–C(4a)	1.520 (7)
C(3)–C(4)–C(4a)	121.6 (5)	C(3)–C(4)–C(11)	114.1 (5)
C(4a)–C(4)–C(11)	124.3 (5)	C(4)–C(3)–C(2)	114.1 (5)
C(3)–C(2)–C(1)	107.5 (5)	C(2)–C(1)–C(8a)	111.1 (5)
C(2)–C(1)–C(12)	112.1 (5)	C(8a)–C(1)–C(12)	110.6 (4)
C(7)–C(8)–C(8a)	112.2 (5)	C(8)–C(7)–C(6)	111.4 (5)
C(8)–C(7)–C(13)	112.2 (5)	C(6)–C(7)–C(13)	112.3 (5)
C(7)–C(6)–C(5)	111.6 (5)	C(6)–C(5)–C(4a)	110.8 (5)
C(1)–C(8a)–C(8)	114.5 (4)	C(1)–C(8a)–C(4a)	111.1 (4)
C(8)–C(8a)–C(4a)	110.0 (4)	C(4)–C(4a)–C(5)	125.7 (5)
C(4)–C(4a)–C(8a)	123.7 (5)	C(5)–C(4a)–C(8a)	110.2 (4)
C(1)–C(12)–O(1)	113.4 (5)	C(1)–C(12)–O(2)	123.1 (5)
O(1)–C(12)–O(2)	123.5 (5)		

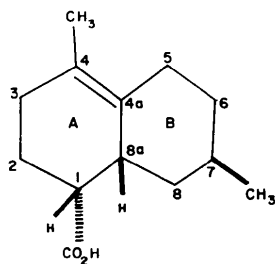
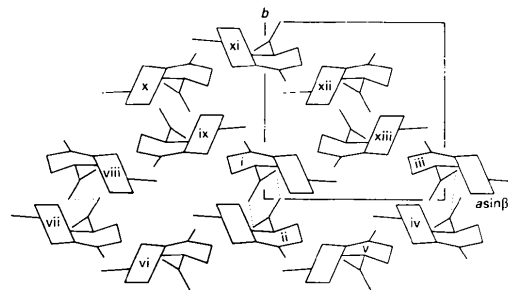


Fig. 1. Atom numbering.


 Fig. 2. [001] projection. Symmetry code: (i) x, y, z ; (ii) $\bar{x}, \bar{y}, \bar{z}$; (iii) $1+x, y, z$; (iv) $1-x, \bar{y}, \bar{z}$; (v) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vi) $-\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vii) $-1-x, \bar{y}, \bar{z}$; (viii) $x-1, y, z$; (ix) $x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z$; (x) $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (xi) $\bar{x}, 1-y, \bar{z}$; (xii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (xiii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$. The dotted lines represent interaction between: molecules i, ii 2.639 (6), molecules iii, iv 3.474 (7), molecules vii, viii 3.273 (6) \AA .

The main skeleton, consisting of the two rings, is similar to that of capsidiol (Birnbaum, Stoessl, Grover & Stothers, 1974) with similar bond lengths and angles.

As in capsidiol all the bonds involving C(8a) appear longer than other bonds due to the full substitution of that atom. The dimensions of the carboxylic group reveal a significant distinction between the two C–O bonds, both in bond lengths and valency angles. This feature is generally observed in carboxylic acids.

We thank Dr A. S. Rao for suggesting this problem, for supplying the crystals and for useful discussions. We also thank Drs A. P. B. Sinha and L. M. Pant for their constant encouragement and keen interest.

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