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Key indicators

Single-crystal X-ray study
 $T = 296\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.055
 wR factor = 0.162
Data-to-parameter ratio = 15.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Formation of non-centrosymmetric dimers
in (1*S**,4*aR**,8*aR**)-decahydronaphthalene-
1-carboxylic acid

The all-*cis* racemate of the title compound, $\text{C}_{11}\text{H}_{18}\text{O}_2$, crystallizes in the chair-chair conformation that places the carboxyl group in an equatorial position. The space group is centrosymmetric but the compound aggregates as dimers whose components are related by a C_2 axis [$\text{O}\cdots\text{O} = 2.665(3)\text{ \AA}$ and $\text{O}-\text{H}\cdots\text{O} = 177^\circ$]. In the crystal structure, one $\text{C}-\text{H}\cdots\text{O}=\text{C}$ close contact is found.

Received 28 February 2005

Accepted 1 March 2005

Online 11 March 2005

Comment

The title compound, (I), is a racemic mixture of the 1*S*,4*aR*,8*aR* diastereomer and its enantiomer. The molecular structure of (I) is illustrated in Fig. 1. The carboxyl group, attached in an equatorial position in the expected double-chair conformation, is found to be fully ordered and lies approximately parallel to the C1–C2 bond [$\text{O1}-\text{C9}-\text{C1}-\text{C2} = -13.1(3)^\circ$], so avoiding interaction with the C8 equatorial H atom.

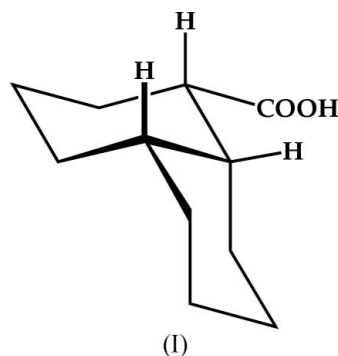


Fig. 2 shows the dimeric carboxyl unit, whose components are related by a twofold axis normal to the carboxyl plane. Such non-centrosymmetric dimers arise most frequently in chiral non-racemates, where no centrosymmetry is possible (Gavezzotti & Filippini, 1994; Allen *et al.*, 1999; Sørensen & Larsen, 2003). Our own study of the crystallography of keto-carboxylic acids has encountered several instances among achiral compounds and racemates of the formation of dimers lacking any element of symmetry (Lalancette *et al.*, 1991, 1996), and one case of a C_2 -dimer (Coté *et al.*, 1996). However, compound (I) is the only instance among our compounds of C_2 -dimer formation in a racemic acid. Within the 2.7 Å range we survey for non-bonded $\text{C}-\text{H}\cdots\text{O}$ packing interactions (Steiner, 1997), a pair of 2.70 Å close contacts is found, to the $\text{C}=\text{O}$ from atom H2A in molecules centrosymmetrically related across $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and the c cell edge.

Fig. 3 shows the crystal packing with extracellular molecules included, and shows the centrosymmetric packing of the non-

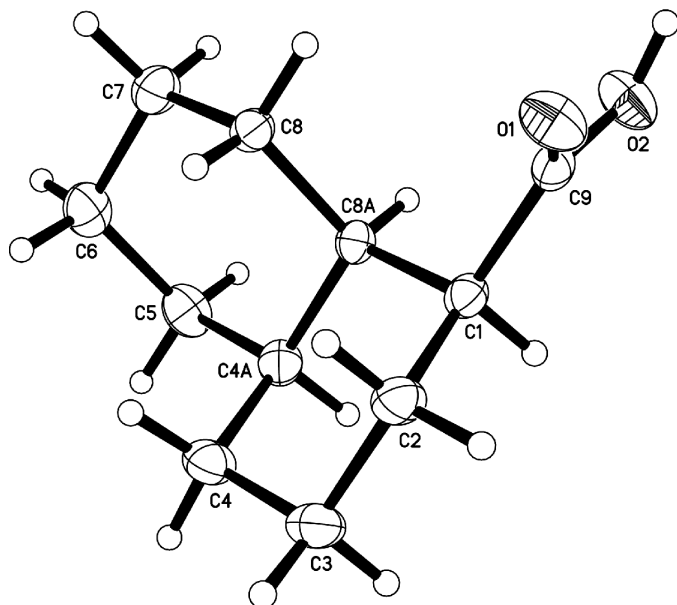


Figure 1
A view of the asymmetric unit of (I), with the numbering scheme. Displacement ellipsoids are drawn at the 20% probability level.

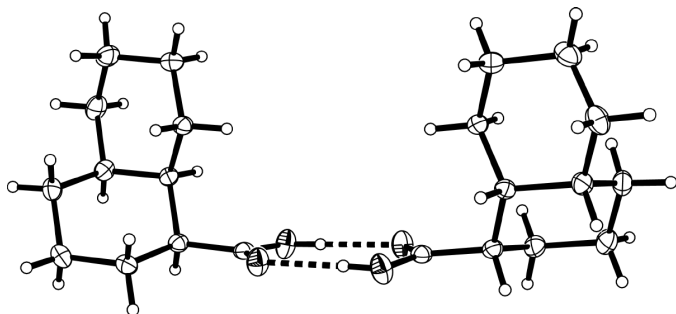


Figure 2
A view of the carboxyl dimer, pairing molecules of identical handedness related by a C_2 axis normal to the carboxyl plane. Displacement ellipsoids are drawn at the 20% probability level. The O—H...O hydrogen bonds are shown as dashed lines.

centrosymmetric dimer units. The C_2 axes of the dimers lie parallel to b .

Experimental

Exhaustive catalytic hydrogenation of 6-hydroxy-1-naphthoic acid dissolved in 95% ethanol over a 5% Rh/C catalyst yielded no isolable hydroxy acid, but gave compound (I) as the major product (Ungnade & Morriss, 1950). The crystal used was obtained from acetonitrile (m.p. 398 K).

Crystal data

$C_{11}H_{18}O_2$
 $M_r = 182.25$
 Monoclinic, $C2/c$
 $a = 21.893$ (9) Å
 $b = 7.756$ (3) Å
 $c = 13.187$ (7) Å
 $\beta = 112.23$ (3)°
 $V = 2072.7$ (16) Å³
 $Z = 8$

$D_x = 1.168$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 24 reflections
 $\theta = 3.5$ – 8.9°
 $\mu = 0.08$ mm⁻¹
 $T = 296$ (2) K
 Parallelepiped, colourless
 0.50 × 0.47 × 0.15 mm

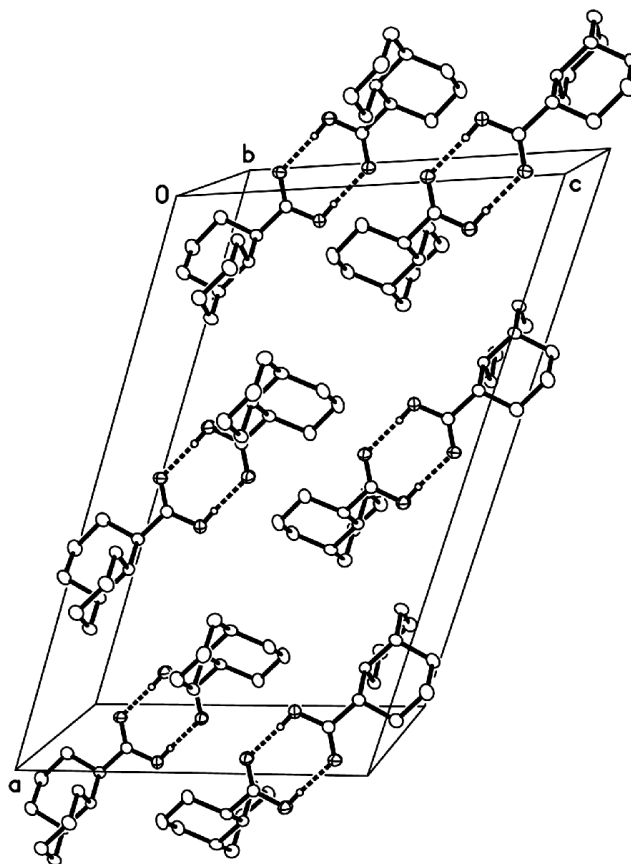


Figure 3
A partial packing diagram for (I), showing the non-centrosymmetric dimers arranged centrosymmetrically around $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and the c cell edge. Displacement ellipsoids are drawn at the 20% probability level. The O—H...O hydrogen bonds are shown as dashed lines. For clarity, all C-bound H atoms have been removed.

Data collection

Siemens P4 diffractometer
 ω - 2θ scans
 Absorption correction: analytical
 (SHELXTL; Sheldrick, 1997b)
 $T_{min} = 0.957$, $T_{max} = 0.987$
 2324 measured reflections
 1840 independent reflections
 1247 reflections with $I > 2\sigma(I)$

$R_{int} = 0.026$
 $\theta_{max} = 25.0^\circ$
 $h = -1 \rightarrow 26$
 $k = -9 \rightarrow 1$
 $l = -15 \rightarrow 14$
 3 standard reflections
 every 97 reflections
 intensity decay: variation <1.9%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.162$
 $S = 1.01$
 1840 reflections
 119 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2 + 1.5893P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.49$ e Å⁻³
 $\Delta\rho_{min} = -0.13$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0037 (10)

All H atoms for (I) were found in electron-density difference maps, but were placed in calculated positions and allowed to refine as riding atoms [C—H = 0.98 Å for methine groups and C—H = 0.97 Å for methylene groups, with $U_{iso}(H) = 1.2U_{eq}(C)$, and O—H = 0.82 Å, with $U_{iso}(H) = 1.5U_{eq}(O)$].

Data collection: XSCANS (Siemens, 1996); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

Support for MD from the Rutgers Undergraduate Research Fellows Program and helpful consultations with Professor G. L. Spoog are gratefully acknowledged.

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