

342 parameters. Refinement to final $R = 0.044$, $S = 1.0614$, unit weights. Largest peak on a final difference Fourier map 0.22 , lowest trough $-0.26 \text{ e } \text{Å}^{-3}$, $(\Delta/\sigma)_{\text{max}} = 0.29$. Scattering factors from *SHELX76*. The molecule and the numbering scheme are shown in Fig. 1, molecular packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* interatomic distances, angles and selected torsion angles in Table 2.

Related literature. The paper is a continuation of the study of the structure–biological activity relationship of oxazadibenzocycloalkenes. The title compound shows

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51743 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

neuroleptic activity (Glinka, 1986). Related studies: Stępień, Wajzman, Grabowski, Glinka & Perrin (1987), Olszak, Stępień, Wajzman, Grabowski, Glinka & Lecocq (1987).

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Structure of (+)-(1*R*,2*S*,3*S*)-3-Benzoyl-6,6-dimethyl-2-bicyclo[3.1.1]heptanecarboxylic Acid

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Abstract. $\text{C}_{17}\text{H}_{20}\text{O}_3$, $M_r = 272.35$, monoclinic, C_2 , $a = 20.324$ (3), $b = 6.779$ (1), $c = 13.890$ (2) Å, $\beta = 126.72$ (1)°, $V = 1534.1$ (5) Å³, $Z = 4$, $D_x = 1.18 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.0744 \text{ mm}^{-1}$, $F(000) = 584$, $T = 296 \text{ K}$, final $R_F = 0.042$ for 976 unique observed reflections. Pairs of molecules are held together across the twofold axis by the formation of two intermolecular hydrogen bonds between the carboxyl groups: the $\text{O}(1)\cdots\text{O}(2')$ distance is 2.628 (3) Å and the $\text{O}(1)\text{—H}\cdots\text{O}(2')$ angle 156 (6)°. The cyclohexane ring has an unusual conformation: five C atoms [C(1)–C(5)] are coplanar within the experimental uncertainty, with the sixth C atom [C(6)] 1.1 Å out of this plane. The carboxyl and benzoyl groups are in *trans* positions with regard to the cyclohexane ring, as suggested by ¹H NMR data.

Experimental. Colourless crystal obtained by evaporation of an ether solution, $0.60 \times 0.50 \times 0.35 \text{ mm}$, mounted on a glass fibre, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized $\text{Mo } K\alpha$, ω - 2θ method, lattice parameters from 25 reflections ($8 < \theta < 13^\circ$), three standard reflections measured every hour, no loss of intensity, 1600 measured reflections (h 0→24, k 0→8, l -16→16) with $\theta < 25^\circ$, 1463 independent, 976 with $I > 3\sigma(I)$, Lp correction, no absorption correction, direct methods, refinement by full-matrix least squares using $w = 4F_o^2/[(\sigma I)^2 + (pF_o)^2]$ and F ; all non-H atoms anisotropic. H atom for the carboxylic group [H(10), involved in hydrogen bonding] found on difference map and refined, all other H atoms included in the refinement at calculated positions ($\text{C—H} = 0.95 \text{ Å}$, $B = 5 \text{ Å}^2$) and not refined

Table 1. Atomic coordinates and equivalent isotropic temperature factors (Å²) with e.s.d.'s in parentheses

	x	y	z	B(Å ²)
O(1)	0.41473 (9)	0.149	0.5093 (1)	5.53 (6)*
O(2)	0.54934 (9)	0.1312 (4)	0.6436 (1)	5.25 (5)*
O(3)	0.5460 (2)	-0.0617 (5)	0.9101 (2)	8.9 (1)*
C(1)	0.3923 (1)	0.3072 (6)	0.6721 (2)	4.76 (8)*
C(2)	0.4675 (1)	0.1828 (5)	0.7129 (2)	3.93 (6)*
C(3)	0.5467 (1)	0.2530 (5)	0.8344 (2)	3.64 (6)*
C(4)	0.5342 (2)	0.4393 (5)	0.8884 (2)	4.36 (7)*
C(5)	0.4461 (2)	0.5109 (6)	0.8143 (2)	4.94 (8)*
C(6)	0.3902 (2)	0.3281 (7)	0.7803 (2)	5.94 (9)*
C(7)	0.4089 (2)	0.5306 (6)	0.6787 (2)	4.88 (8)*
C(8)	0.3296 (2)	0.6498 (9)	0.6103 (3)	8.1 (1)*
C(9)	0.4630 (2)	0.6063 (6)	0.6458 (2)	5.41 (8)*
C(10)	0.4821 (1)	0.1553 (5)	0.6203 (2)	3.89 (6)*
C(11)	0.5838 (2)	0.0873 (5)	0.9258 (2)	4.86 (8)*
C(12)	0.6692 (2)	0.1095 (6)	1.0400 (2)	4.99 (8)*
C(13)	0.7192 (2)	0.2646 (8)	1.0603 (2)	6.7 (1)*
C(14)	0.7957 (2)	0.282 (1)	1.1697 (3)	9.6 (2)*
C(15)	0.8220 (2)	0.146 (1)	1.2565 (3)	11.4 (2)*
C(16)	0.7733 (2)	-0.011 (1)	1.2369 (3)	9.7 (1)*
C(17)	0.6969 (2)	-0.0321 (7)	1.1293 (3)	7.2 (1)*
H(10)	0.430 (2)	0.18 (1)	0.458 (3)	8 (1)

$$* B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

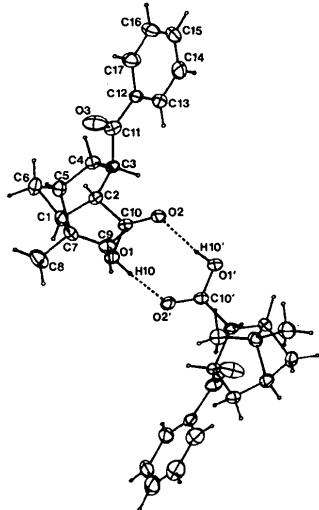


Fig. 1. ORTEP view (Johnson, 1976) of the dimeric unit. The atoms related by the twofold axis are indicated by primes. Intermolecular hydrogen bonds are shown as dashed lines.

but constrained to ride on their C atoms, max. shift/ σ = 0.04 in final cycle, $R = 0.042$, $wR = 0.046$ ($p = 0.04$), $S = 2.0$, final difference map with no features greater than $0.13 \text{ e } \text{Å}^{-3}$, scattering factors from *International Tables for X-ray Crystallography* (1974), VAX computer with programs SDP (Frenz, 1985), MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and ORTEP (Johnson, 1976).

Table 2. Selected bond lengths (Å) and angles (°)

The atoms related by the diad axis are indicated by primes.

C(1)—C(2)	1.526 (4)	C(3)—C(4)	1.565 (5)
C(1)—C(6)	1.537 (5)	C(4)—C(5)	1.515 (4)
C(1)—C(7)	1.543 (6)	C(5)—C(6)	1.553 (5)
C(2)—C(3)	1.555 (3)	C(5)—C(7)	1.560 (4)
C(2)—C(10)	1.494 (5)	C(7)—C(8)	1.524 (5)
C(3)—C(11)	1.516 (4)	C(7)—C(9)	1.512 (5)
C(11)—C(12)	1.503 (3)	C(15)—C(16)	1.367 (10)
C(12)—C(13)	1.369 (6)	C(16)—C(17)	1.374 (4)
C(13)—C(14)	1.384 (3)	C(17)—C(12)	1.394 (5)
C(14)—C(15)	1.351 (10)	C(11)—O(3)	1.207 (5)
C(10)—O(1)	1.315 (2)	C(10)—O(2)	1.213 (4)
O(1)—H(10)	0.96 (6)	H(10)—O(2')	1.72 (5)
C(2)—C(1)—C(6)	107.2 (2)	C(1)—C(7)—C(5)	85.3 (3)
C(1)—C(2)—C(3)	113.0 (3)	C(7)—C(5)—C(6)	87.9 (2)
C(2)—C(3)—C(4)	114.1 (2)	C(5)—C(6)—C(1)	85.8 (3)
C(3)—C(4)—C(5)	113.4 (2)	C(6)—C(1)—C(7)	89.1 (3)
C(4)—C(5)—C(6)	107.7 (3)	C(4)—C(5)—C(7)	111.8 (3)
C(2)—C(1)—C(7)	112.7 (2)	O(2)—C(10)—C(2)	124.0 (2)
O(2)—C(10)—O(1)	121.7 (3)	O(1)—C(10)—C(2)	114.3 (2)
C(10)—O(1)—H(10)	107 (2)	O(1)—H(10)—O(2')	156 (6)

Atomic coordinates are listed in Table 1 and selected molecular distances and angles in Table 2.* Fig. 1 is an ORTEP drawing which illustrates the dimeric structure.

Related literature. For the preparation of the title compound, see Pellet (1988). A similar *trans* stereochemistry has been proposed for a formylpinane (Himmele & Siegel, 1976). ¹H NMR data for the title compound can be found in Pellet & Wartski (1989).

* Lists of structure factors, anisotropic thermal parameters, bond distances, bond angles and torsion angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51817 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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