

were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).<sup>\*</sup> Figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). The positional and equivalent isotropic thermal parameters for non-H atoms are listed in Tables 2 and 3 for (1) and (2), respectively. The bond lengths and angles for the non-H atoms are listed in Tables 4 and 5. The atom-labeling scheme for (1) is shown in Fig. 1 and that for (2) in Fig. 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

**Related literature.** The above compounds were prepared in the course of a study directed towards the synthesis of breynolide (Sakai, Ohkuma, Koshiyama, Naito & Kawaguchi, 1976; Nishiyama, Ikeda, Yoshida & Yamamura, 1989). Breynolide is the aglycon of Breynin A which has been shown to reduce cholesterol in the blood of mammals (Koshiyama, Hatori, Ohkuma, Sakai, Imanishi, Ohbayashi & Kawaguchi, 1976). In an attempt to control the

<sup>\*</sup> Tables of anisotropic thermal parameters, H-atom positional and thermal parameters, bond distances and angles involving the H atoms, torsion angles, structure-factor amplitudes, a unit-cell packing diagram and a view of each molecule highlighting the different orientations of the siloxy groups have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52372 (81 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure at 173 K of 1,2-Bis(1-naphthyl)cyclopentene

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**Abstract.** C<sub>25</sub>H<sub>20</sub>, *M*<sub>r</sub> = 320.4, monoclinic, *P*2<sub>1</sub>, *a* = 10.566 (6), *b* = 7.618 (3), *c* = 10.795 (7) Å, β = 108.00 (4)°, *V* = 826.3 (8) Å<sup>3</sup>, *Z* = 2, *D*<sub>x</sub> = 1.288 g cm<sup>-3</sup>, μ = 0.68 cm<sup>-1</sup>, λ(Mo Kα) = 0.71073 Å, *F*(000) = 340, *T* = 173 K, *R* = 0.0373 for 1134 reflections [*F*<sub>o</sub> ≥ 6σ(*F*<sub>o</sub>)]. No single-crystal photochemical reaction analogous to those reported for 1,2-diphenylcyclopentene in solution and for 1,2-bis(1-naphthyl)ethylene in the crystal was observed. This is consistent with the considerable twist of the naphthyl groups [dihedral angles: naphthyl–cyclopentene 64.9 (2), naphthyl–naphthyl 76.8 (2)°].

facial selectivity of a Diels–Alder intermolecular reaction, several different siloxy-protected adducts were prepared and structures investigated to determine any facial bias.

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**Experimental.** The title compound (1) was synthesized, using a procedure published for the 2-naphthyl isomer (Rio & Bricout, 1971), by addition of two equivalents of the Grignard reagent from α-bromonaphthalene to cyclobutanecarboxylic acid methyl

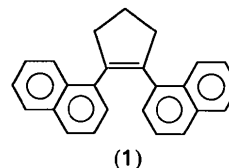


Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$U_{eq}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	x	y	z	$U_{eq}(\text{\AA}^2)$
C(1)	0	0.8941	0.5000	0.029 (1)
C(2)	-0.0080 (2)	0.7738 (4)	0.3834 (2)	0.028 (1)
C(3)	-0.0046 (2)	0.5894 (4)	0.4369 (2)	0.020 (1)
C(4)	-0.0099 (2)	0.4377 (4)	0.3493 (2)	0.020 (1)
C(5)	-0.1201 (2)	0.4162 (4)	0.2413 (2)	0.026 (1)
C(6)	-0.1284 (2)	0.2787 (5)	0.1514 (2)	0.029 (1)
C(7)	-0.0237 (2)	0.1666 (5)	0.1680 (2)	0.027 (1)
C(8)	0.0938 (2)	0.1857 (4)	0.2750 (2)	0.022 (1)
C(9)	0.2050 (2)	0.0736 (5)	0.2909 (2)	0.027 (1)
C(10)	0.3175 (2)	0.0924 (4)	0.3941 (2)	0.029 (1)
C(11)	0.3245 (2)	0.2252 (4)	0.4870 (2)	0.026 (1)
C(12)	0.2197 (2)	0.3366 (5)	0.4748 (2)	0.022 (1)
C(13)	0.1003 (2)	0.3208 (4)	0.3682 (2)	0.020 (1)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) for the non-hydrogen atoms

C(1)—C(2)	1.538 (3)	C(1)—C(2A)	1.538 (3)
C(2)—C(3)	1.515 (5)	C(3)—C(4)	1.482 (4)
C(3)—C(3A)	1.336 (4)	C(4)—C(5)	1.380 (2)
C(4)—C(13)	1.430 (4)	C(5)—C(6)	1.412 (4)
C(6)—C(7)	1.364 (4)	C(7)—C(8)	1.418 (3)
C(8)—C(9)	1.419 (4)	C(8)—C(13)	1.426 (4)
C(9)—C(10)	1.362 (3)	C(10)—C(11)	1.410 (4)
C(11)—C(12)	1.368 (4)	C(12)—C(13)	1.426 (2) †
C(2)—C(1)—C(2A)	106.8 (2)	C(1)—C(2)—C(3)	104.6 (2)
C(2)—C(3)—C(4)	119.2 (2)	C(2)—C(3)—C(3A)	112.0 (1)
C(4)—C(3)—C(3A)	128.8 (1)	C(3)—C(4)—C(5)	119.1 (2)
C(3)—C(4)—C(13)	121.8 (2)	C(5)—C(4)—C(13)	118.9 (2)
C(4)—C(5)—C(6)	121.7 (2)	C(5)—C(6)—C(7)	119.9 (2)
C(6)—C(7)—C(8)	120.9 (3)	C(7)—C(8)—C(9)	121.4 (3)
C(7)—C(8)—C(13)	119.2 (2)	C(9)—C(8)—C(13)	119.4 (2)
C(8)—C(9)—C(10)	121.1 (3)	C(9)—C(10)—C(11)	119.8 (2)
C(10)—C(11)—C(12)	120.9 (2)	C(11)—C(12)—C(13)	120.9 (3)
C(4)—C(13)—C(8)	119.3 (2)	C(4)—C(13)—C(12)	122.8 (2)
C(8)—C(13)—C(12)	117.9 (2)		

ester. The resulting tertiary alcohol was dehydrated with rearrangement by stirring with methanesulfonic acid at room temperature. The crude product was recrystallized first from benzene and hexane, then from chloroform to form clear plates. The data crystal had dimensions  $0.15 \times 0.21 \times 0.25$  mm. A Nicolet R3m/V diffractometer equipped with a graphite monochromator and a Nicolet LT-2 low-temperature delivery system (173 K) was used to collect the data. The lattice parameters were obtained from the least-squares refinement of 50 reflections with  $21.91 < 2\theta < 34.83^\circ$ . The data were collected using the  $\omega$  scan technique with a  $2\theta$  range from  $3.0$  to  $60.0^\circ$  and a  $1.2^\circ$   $\omega$  scan at  $2.4 \text{ min}^{-1}$  (two symmetry-related quadrants:  $h = -14 \rightarrow 14$ ,  $k = 0 \rightarrow 10$ ,  $l = 0 \rightarrow 15$ ;  $h = -14 \rightarrow 14$ ,  $k = -10 \rightarrow 0$ ,  $l = 0 \rightarrow 15$ ). Of the 2539 reflections measured, 1298 were unique, with  $R_{int} = 0.019$  from averaging symmetry-equivalent reflections. Four reflections

( $21\bar{3}$ ,  $020$ ,  $200$ ,  $0\bar{2}0$ ) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on  $I$  was  $< 1.01\%$ ). The data were also corrected for Lp effects and for absorption (numerical corrections based on measurement of indexed crystal faces,  $\mu = 0.68 \text{ cm}^{-1}$ ). Reflections having  $F_o < 6\sigma(F_o)$  were considered unobserved (164 reflections). Minimum and maximum transmission were 0.9984 and 0.9992, respectively. Data reduction and decay correction were performed using the Nicolet XRD SHELXTL-PLUS software package (Sheldrick, 1987). The structure was solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976). The molecule is located on a twofold rotation axis. In all, 153 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. The H-atom positions were obtained from a  $\Delta F$  map. The H atoms were refined with isotropic thermal parameters. The function  $\sum w(|F_o| - |F_c|)^2$  was minimized, where  $w = 1/[\sigma(F_o)]^2$  and  $\sigma(F_o) = (0.5kI^{-1/2} \times \{[\sigma(I)]^2 + (0.02I)^2\}^{1/2})$ . The intensity,  $I$ , is given by  $(I_{peak} - I_{background}) \times (\text{scan rate})$ ; the factor 0.02 serves to downweight intense reflections and to account for instrument instability and  $k$  is the correction due to Lp effects and decay.  $\sigma(I)$  was estimated from counting statistics as  $\sigma(I) = [(I_{peak} - I_{background})^{1/2} \times (\text{scan rate})]$ . The final  $R = 0.0373$  for 1134 reflections, with  $wR = 0.0457$  ( $R_{all} = 0.0429$ ,  $wR_{all} = 0.0473$ ) and goodness of fit = 1.61. The minimum and maximum peaks in the final  $\Delta F$  map were  $-0.18$  and  $0.21 \text{ e \AA}^{-3}$ , respectively, and the maximum  $|\Delta/\sigma|$  was 0.001. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering

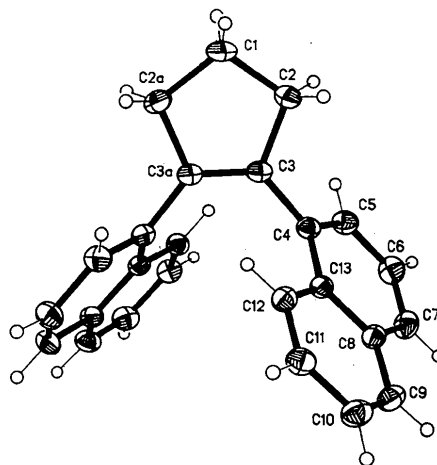


Fig. 1. Molecular structure of (1), with 50% probability ellipsoids, showing the atom-numbering scheme.

factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).<sup>\*</sup> Figures were generated using *SHELXTL-PLUS* (Sheldrick, 1987). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1.

**Related literature.** The title compound, 1,2-bis(1-naphthyl)cyclopentene, was prepared for studies on single-crystal-to-single-crystal photochemical transformations that would provide extended, non-aromatic  $\pi$  systems possibly suitable for non-linear optical applications (Chemla & Zyss, 1987). Photocyclization of 1,2-diphenylcyclopentene (Griffith, 1978) and 1,2-bis(2-naphthyl)cyclopentene (Wismontski-Knittel, Kaganowitch, Seger & Fischer, 1979) in solution and 1,2-bis(1-naphthyl)ethylene (Aldoshin, Alfimov, Atovnyan, Kaminsky, Razumov

<sup>\*</sup> Tables of crystallographic data and intensity collection/structure refinement information, anisotropic thermal parameters, H-atom positional parameters, bond lengths and angles involving the H atoms, structure-factor amplitudes, and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52303 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of 3,4,5,6,7,8-Hexa-*O*-acetyl-1,2-dideoxy-2-*C*-nitro-*D*-*threo*-*L*-*talo*-octitol

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**Abstract.**  $C_{20}H_{29}NO_{14}$ ,  $M_r = 507.4$ , orthorhombic,  $P2_12_12_1$ ,  $a = 12.950$  (3),  $b = 19.713$  (6),  $c = 9.942$  (10) Å,  $V = 2538$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.328$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.17$  mm<sup>-1</sup>,  $F(000) = 1072$ , room temperature, final  $wR = 0.037$  for 1311 observed reflexions. Bond distances and angles are all within the expected ranges. The crystal structure is stabilized by C—H...O hydrogen bonds along [001] and [100].

**Experimental.** Single crystals in the form of colourless needles elongated along [001]. Dimensions

& Rachinsky, 1984) in the crystal have been reported.

Acknowledgement is made to the Donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research (Grant ACS-PRF AC-20714 to JKW) and to the Robert A. Welch Foundation (Grant F-626 to JKW and F-233 to RED).

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of the crystal used  $0.2 \times 0.2 \times 0.4$  mm. Cell parameters were refined by least-squares fit from 25 reflections with  $6 < \theta < 20^\circ$  on an Enraf–Nonius CAD-4 diffractometer. Data collection by  $\omega$ - $2\theta$  scan, up to  $(\sin \theta)/\lambda = 0.60$  Å<sup>-1</sup>, using graphite-monochromated Mo  $K\alpha$  radiation, 2602 unique reflections measured ( $0 \leq h \leq 15$ ,  $0 \leq k \leq 23$ ,  $0 \leq l \leq 11$ ), 1311 with  $I > 2\sigma(I)$  considered observed; three intensity standards (142;  $\bar{1}42$ ; 421) monitored at intervals of 60 reflections showed no significant variations ( $< 2\sigma$ ) during data collection. Intensity data corrected for Lorentz-polarization factors; no absorption or extinction cor-