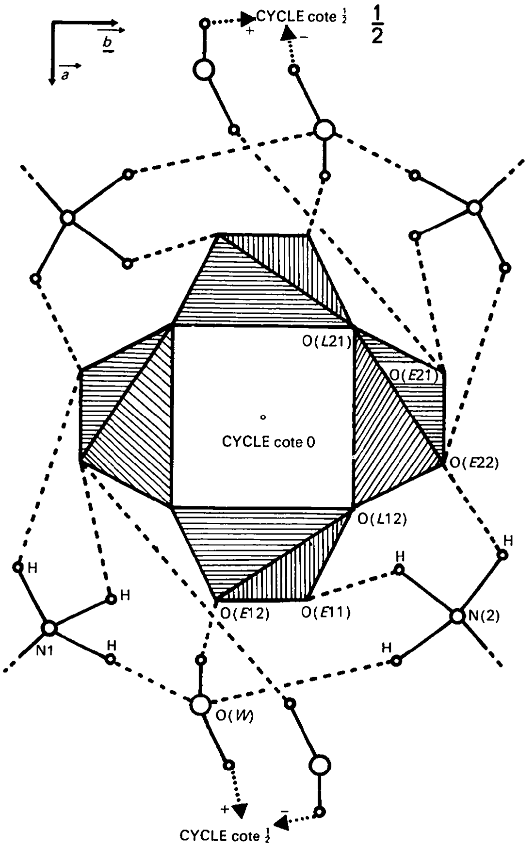

Fig. 2. Représentation schématique, dans une couche, des liens du cycle P_4O_{12} aux groupements organiques et aux molécules d'eau.

Parmi toutes les liaisons hydrogène du type N—H···O—P ou O*W*—H···O—P deux seulement N(2)—H···O(*E*11) et O*W*—H···O(*E*12) sont courtes (*cf.* Tableau 2) et considérées fortes selon le critère portant sur les distances N···O et O···O inférieures à 2,75 Å (Brown, 1976; Blessing, 1986). Le domaine de variation des distances N(O)···O est considérable: 2,706 (3)–2,913 (3) Å à cause de l'existence, dans ces liaisons hydrogène, de deux types de donneurs: N(O)—H forts et N(O)—H faibles. La structure contient huit donneurs (six NH et deux OH) et cinq acceptuers (quatre O*E* et un O*W*). Les atomes d'oxygène extérieur du tétraèdre $P(1)O_4$ jouent le rôle de simple accepteur, alors que ceux du tétraèdre $P(2)O_4$ et la molécule d'eau interviennent comme double accepteurs. Ce fait est en accord avec la force relative des liaisons hydrogène dans lesquelles sont engagés les atomes simplement ou doublement accepteurs.

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Structure of 10,10′,11,11′-Tetrahydro-5,5′-bis-5*H*-dibenzo[*a*,*d*]cylcoheptenylidene

By G. Shoham,* S. Cohen, R. M. Suissa and I. Agranat

Institute of Chemistry, The Hebrew University of Jerusalem, Jerusalem 91904, *Israel*

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Abstract. $C_{30}H_{24}$, $M_r = 384{\cdot}53$, monoclinic, $P2_1/n$, $a = 19{\cdot}679$ (4), $b = 10{\cdot}009$ (1), $c = 10{\cdot}896$ (1) Å, $\beta = 100{\cdot}41$ (1)°, $V = 2111$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}21$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, μ(Mo $K\alpha$) = 0·34 cm^{-1}, $F(000) = 816{\cdot}0$, room temperature, $R = 0{\cdot}065$ for 3343 independent reflections. The molecule adopts the *anti*-folded conformation with folding angles of 57·0 (3) and 58·6 (3)° for the two tricyclic halves. The expected overcrowding of the molecule is partially relieved by a combination of *anti*-folding, pyramidalization of the central bridging carbons, and twisting of the central double bond.

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* To whom correspondence should be addressed.

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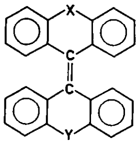
Except for the rather long central double bond [1·350 (2) Å] and relatively small internal sp^2 angles [115·4 (1), 115·7 (1)°], all structural parameters fall within the conventional ranges.

Introduction. The bistricyclic ethylenes (I) are attractive substrates for the study of the ground-state conformation and the dynamic behaviour of symmetrical [(I), $Y = X$] and asymmetrical [(I), $Y \neq X$] *overcrowded ethylenes* (Sandström, 1983). *Intramolecular overcrowding* is a steric effect shown by aromatic structures, in which the (intramolecular) distance of closest approach between non-bonded atoms, calculated on the basis of conventional bond lengths and bond angles, is smaller than the sum of the van der Waals radii of the involved atoms (Harnik, Herbstein & Schmidt, 1951). An idealized coplanar bistricyclic ethylene would maintain very short non-bonded carbon–carbon and hydrogen–hydrogen distances [*e.g.* C(1)—C(1)′ and H(1)—H(1)′ in Fig. 1], leading to a considerable overlap of the van der Waals radii in the region of the central carbon–carbon double bond [C(9)=C(9)′ or the 'pinch']. The associated repulsive interactions could, in principle, be relieved by deviations from coplanarity and by various other distortions. Several bistricyclic ethylenes have been shown to be thermochromic (Meyer, 1909), photochromic (Fischer, 1984) and piezochromic (Fanselow & Drickamer, 1974). The stereochemical basis of these chromic changes is not fully understood at the present time.



(I)

In order to understand better both the modes of deformation of overcrowded systems and the structural basis for the chromic transformations in those systems, we studied extensively a family of bistricyclic ethylenes. A series of these compounds [(I), with various X and Y] has been synthesized and analyzed in our laboratory in the past few years. We were interested mainly in the mode and magnitude of distortion of their structures from the expected 'classical' structure, and especially in the deformation of the central double bond and the presumably planar central ethylene unit. In the present paper we present the crystal and molecular structure of the first compound of this series, 10,10′,11,11′-tetrahydro-5,5′-bis-5*H*-dibenzo[*a*,*d*]cycloheptenylidene [(1), (I) where $X = Y = CH_2—CH_2$], and discuss the overcrowding effects mentioned above as manifested in this structure. Compound (1) is especially interesting because of the central seven-membered ring of the tricyclic unit which contains a saturated bridge. Of all bistricyclic ethylenes studied so far, only the system which contains a central unsaturated seven-membered ring [(2), a molecule with the general scheme of (I) where $X = Y = CH=CH$] has been found in both the *syn* and *anti* geometrical isomers (Bergmann, Rabinovitz & Agranat, 1968; Schönberg, Sodtke & Praefcke, 1969; Dichmann, Nyburg, Pickard & Potworowski, 1974), while all those with various central six-membered rings have been found only in the *anti*-folded isomer. It was expected, therefore, that compound (1), the saturated analog of (2), would be similar to (2), and allow the existence of both isomers in non-extreme conditions. All our attempts to synthesize directly the *syn* isomer of (1) have been unsuccessful so far. Nevertheless, both compounds (1) and (2) are expected to undergo a dynamic *syn*$\rightleftarrows$*anti* interconversion at high temperatures. The dynamic stereochemistry of bistricyclic ethylenes with a seven-membered central ring, and of related moderately overcrowded systems, has not been investigated as yet.

Experimental. Compound (1) was synthesized by SeO_2 oxidation of 10,10′,11,11′-tetrahydro-bis-5*H*-dibenzo[*a*,*d*]cycloheptenyl according to reported procedures (Schönberg *et al.*, 1969). The latter tetrahydro derivative was prepared by a reductive coupling of 5*H*-dibenzo[*a*,*d*]cyclohepten-5-one, using low-valent titanium (Agranat *et al.*, 1987). Compound (1) was purified by repeated column chromatography on silica gel (hexane/dichloromethane, 4:1). Slow evaporation from chloroform yielded colorless

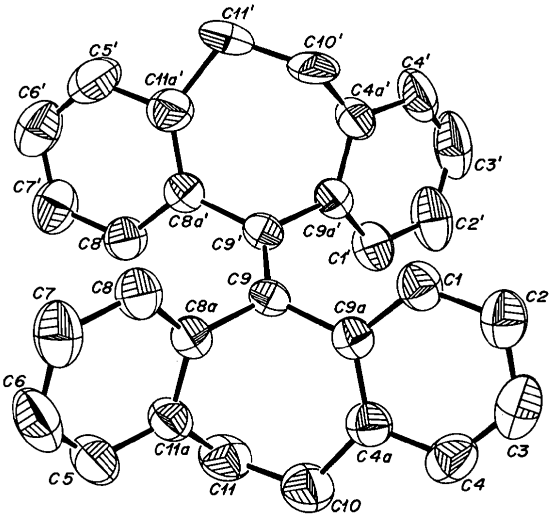


Fig. 1. Final structure and numbering scheme of (1) (*ORTEP*II; Johnson, 1976).

well formed single crystals, with average dimensions of 0·3 × 0·3 × 0·4 mm and m.p. of 588 K. Diffraction data were measured on an Enraf–Nonius CAD-4 automatic four-circle diffractometer with molybdenum radiation and a graphite monochromator. Lattice parameters were determined by least-squares fit of 25 reflections in the range of $18 < 2\theta < 34°$, monoclinic, space group $P2_1/n$. Intensity data were collected in the $\omega/2\theta$ scan mode, $4 \leq 2\theta \leq 56°$, $00\bar{l} \rightarrow hkl$, on one crystal, in three shells, three orientation check reflections ($84\bar{4}$, $64\bar{2}$, $60\bar{6}$) measured every 100 reflections, three intensity check reflections (12,0,0, $44\bar{4}$, 447) measured every 60 min. The intensities of the check reflections varied on average by 2% and decayed throughout the data collection by < 5%. Intensity data were reduced, corrected for Lorentz and polarization effects and for radiation damage. Owing to the small absorption coefficient no absorption correction was applied. 5794 reflections were measured, of which 4479 were unique. For the final stages of refinement only those reflections for which $F_o \geq 4{\cdot}0\sigma(F_o)$ were included (3343 reflections, 75% of the total, range of h $0 \rightarrow 26$, k $0 \rightarrow 13$, l $-14 \rightarrow 14$).

The structure was solved by direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The best phase set, with a combined figure of merit (CFOM) of 3·0, yielded a preliminary model with all carbon atoms of the structure clearly recognized in the electron density map. All hydrogen atoms were located by successive cycles of least-squares refinements and difference electron density maps. Conventional full-matrix least-squares techniques were used for refinement of atomic parameters (Cruickshank, 1970; Sheldrick, 1976). All non-hydrogen atoms were refined anisotropically (nine parameters per atom), while 16 of the 24 hydrogen atoms were refined isotropically (four parameters per atom). During refinement it was apparent that the two saturated regions [C(10), C(11) on one half and C(10)′ and C(11)′ on the other half of the molecule] are slightly disordered. While the disorder at C(10) and C(11) could be accounted for by allowing for large temperature factors, it was necessary to assign two alternative conformations for the C(10)′—C(11)′ region. The statistical occupancies of these two conformations, designated as C(10)′—C(11)′ and C(10)′*—C(11)′*, were found to be 0·6 and 0·4, respectively. The carbon atoms in these two regions were refined with no constraint (see above), while the methylenic hydrogens were positioned at a fixed idealized distance from their corresponding carbon atoms. A weighting scheme based on $w = a/[\sigma^2(F) + bF^2]$ was introduced and optimized in the final stages of refinement. The overall scale factor was refined as well. Altogether, 365 parameters (P) (in one block) were refined using 3343 observations (O), $O/P = 9{\cdot}2$. Final $R = 0{\cdot}065$, $wR = 0{\cdot}077$. Final $S = 3{\cdot}19$, where $S = [\sum w|F_o - F_c|^2/(O - P)]^{1/2}$. In the final cycle of refinement the maximum ratio of shift/e.s.d. was 0·02, where the average ratio was 0·005. In the final difference map maximum and minimum electron densities were 0·48 and $-0{\cdot}19$ e Å^{-3}, respectively. It should be noted that these highest values of difference electron density are located, as expected, around the disordered segments of the molecule. The difference peaks in the rest of the unit cell are randomly distributed and are of much smaller values ($\pm 0{\cdot}10$ e Å^{-3}). Atomic scattering factors and f', f'' values have been taken from *SHELX*76 (Sheldrick, 1976).

Discussion. The essential structural results are presented in Tables 1 and 2† and in Figs. 1, 2 and 3. Fig. 1 is a top view (*ORTEP*II; Johnson, 1976) of the final crystal structure of (1), including the numbering scheme we used. Figs. 2 and 3 are two different stereoviews (*ORTEP*II) of (1), demonstrating its overall conformation and stereochemistry around the central double bond. In all three figures the alternative conformation of the disordered part [C(10)′*—C(11)′*, with statistical occupancy of 0·4] has been omitted for clarity.

In general, the molecule adopts the *anti*-folded conformation (Sandström, 1983; Shoham, Cohen, Suissa & Agranat, 1988). However, while in the classical *anti*-folded structure of bistricyclic ethylenes the folding axes of the two tricyclic parts are collinear (coinciding with the central C═C double bond), here the two tricyclic parts are displaced away from each other such that their folding axes do not coincide with each other or with the central double bond (Figs. 2 and 3). The two halves of the molecule, although rather similar in shape, are not related by either crystallographic or non-crystallographic symmetry. The peripheral benzene rings are very nearly planar. The least-squares planes of these four rings are labeled (Shoham *et al.*, 1988) as *A* [C(1), C(2), C(3), C(4), C(4*a*), C(9*a*)], *B* [C(5), C(6), C(7), C(8), C(8*a*), C(11*a*)], *C* [C(1)′, C(2)′, C(3)′, C(4)′, C(4*a*)′, C(9*a*)′] and *D* [C(5)′, C(6)′, C(7)′, C(8)′, C(8*a*)′, C(11*a*)′]. The mean deviations from these planes are 0·003 (1), 0·004 (1), 0·003 (1) and 0·003 (1) Å, respectively. The dihedral angles among these planes are *A*—*B* 58·6 (3), *C*—*D* 57·0 (3), *A*—*C* 60·0 (4), *A*—*D* 5·4 (2), *B*—*C* 6·5 (2) and *B*—*D* 54·9 (3)°. The *A*—*B* and

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† Lists of structure factors, anisotropic thermal parameters and H-atom parameters, and complete lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52622 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and temperature factors for non-hydrogen atoms in* (1)

U_{eq} is $\frac{1}{3}$(trace of the orthogonalized U_{ij} tensor).

	x	y	z	U_{eq}(Å^2)
C(1)	0·8659 (1)	0·5286 (2)	0·5720 (2)	0·0530 (10)
C(2)	0·9270 (1)	0·5809 (2)	0·6381 (2)	0·0662 (14)
C(3)	0·9415 (1)	0·5670 (2)	0·7652 (2)	0·0697 (15)
C(4)	0·8964 (1)	0·5012 (2)	0·8261 (2)	0·0631 (13)
C(4*a*)	0·8340 (1)	0·4470 (2)	0·7616 (1)	0·0509 (10)
C(5)	0·6127 (1)	0·4543 (2)	0·7403 (2)	0·0728 (14)
C(6)	0·5684 (1)	0·5409 (3)	0·6663 (2)	0·0830 (17)
C(7)	0·5834 (1)	0·5864 (2)	0·5549 (2)	0·0720 (15)
C(8)	0·6441 (1)	0·5449 (2)	0·5173 (2)	0·0549 (11)
C(8*a*)	0·6886 (1)	0·4588 (1)	0·5917 (1)	0·0426 (8)
C(9)	0·7537 (1)	0·4115 (1)	0·5535 (1)	0·0395 (8)
C(9*a*)	0·8190 (1)	0·4605 (1)	0·6322 (1)	0·0415 (8)
C(10)	0·7888 (1)	0·3835 (2)	0·8428 (2)	0·0700 (14)
C(11)	0·7230 (1)	0·3172 (2)	0·7821 (2)	0·0680 (13)
C(11*a*)	0·6737 (1)	0·4127 (2)	0·7053 (1)	0·0525 (10)
C(1)′	0·8600 (1)	0·2005 (2)	0·4978 (2)	0·0589 (12)
C(2)′	0·9191 (1)	0·1452 (2)	0·4668 (3)	0·0834 (17)
C(3)′	0·9353 (1)	0·1702 (3)	0·3525 (4)	0·0995 (23)
C(4)′	0·8933 (1)	0·2505 (4)	0·2700 (3)	0·0993 (22)
C(4*a*)′	0·8332 (1)	0·3084 (2)	0·2988 (1)	0·0666 (13)
C(5)′	0·6088 (1)	0·2761 (3)	0·1743 (2)	0·0758 (16)
C(6)′	0·5618 (1)	0·2082 (2)	0·2275 (2)	0·0741 (16)
C(7)′	0·5767 (1)	0·1800 (2)	0·3518 (2)	0·0706 (15)
C(8)′	0·6383 (1)	0·2213 (2)	0·4224 (2)	0·0558 (11)
C(8*a*)′	0·6868 (1)	0·2927 (1)	0·3699 (1)	0·0436 (8)
C(9)′	0·7526 (1)	0·3359 (1)	0·4505 (1)	0·0398 (8)
C(9*a*)′	0·8172 (1)	0·2819 (1)	0·4154 (1)	0·0441 (8)
C(10)′	0·7886 (2)	0·4173 (4)	0·2249 (4)	0·0882 (36)
C(10)′*	0·7902 (3)	0·3643 (9)	0·1809 (3)	0·0822 (51)
C(11)′	0·7189 (1)	0·3700 (5)	0·1559 (3)	0·0747 (30)
C(11)′*	0·7236 (2)	0·4242 (4)	0·2078 (5)	0·0637 (39)
C(11*a*)′	0·6726 (1)	0·3210 (2)	0·2425 (1)	0·0611 (12)

* Disordered positions (lower occupancy).

Table 2. *Selected bond lengths* (Å), *bond angles* (°) *and torsion angles* (*av. e.s.d. of* 0·4°), *in* (1)

C(10)—C(4*a*)	1·505 (3)	C(9*a*)—C(4*a*)	1·394 (2)
C(9)—C(8*a*)	1·493 (3)	C(11*a*)—C(8*a*)	1·401 (3)
C(9*a*)—C(9)	1·494 (2)	C(9)′—C(9)	1·350 (2)
C(11)—C(10)	1·498 (3)	C(11*a*)—C(11)	1·505 (3)
C(9*a*)′—C(4*a*)′	1·389 (3)	C(9)′—C(8*a*)′	1·492 (2)
C(10)′—C(4*a*)	1·533 (5)	C(10)′*—C(4*a*)′	1·513 (6)
C(11*a*)′—C(8*a*)′	1·395 (3)	C(9*a*)′—C(9)′	1·493 (3)
C(11)′—C(10)′	1·517 (6)	C(11)′*—C(10)′*	1·516 (8)
C(11*a*)′—C(11)′	1·508 (5)	C(11*a*)′—C(11*a*)′*	1·535 (6)

C(9*a*)—C(4*a*)—C(4)	118·3 (2)	C(10)—C(4*a*)—C(4)	115·1 (2)
C(10)—C(4*a*)—C(9*a*)	126·6 (2)	C(11*a*)—C(5)—C(6)	121·0 (3)
C(8*a*)—C(8)—C(7)	120·0 (2)	C(9)—C(8*a*)—C(8)	121·3 (2)
C(11*a*)—C(8*a*)—C(8)	120·7 (2)	C(11*a*)—C(8*a*)—C(9)	118·0 (2)
C(9*a*)—C(9)—C(8*a*)	115·4 (1)	C(9)′—C(9)—C(8*a*)	121·7 (1)
C(9)′—C(9)—C(9*a*)	122·9 (2)	C(4*a*)—C(9*a*)—C(1)	118·9 (2)
C(9)—C(9*a*)—C(1)	117·5 (2)	C(9)—C(9*a*)—C(4*a*)	123·5 (2)
C(11)—C(10)—C(4*a*)	118·9 (2)	C(11*a*)—C(11)—C(10)	112·6 (2)
C(8*a*)—C(11*a*)—C(5)	118·3 (2)	C(11)—C(11*a*)—C(5)	122·8 (2)
C(11)—C(11*a*)—C(8*a*)	118·9 (2)	C(9*a*)′—C(1)′—C(2)′	121·2 (3)
C(8*a*)′—C(8)′—C(7)′	121·6 (2)	C(9)′—C(8*a*)′—C(8)′	119·5 (2)
C(11*a*)′—C(8*a*)′—C(8)′	118·9 (2)	C(11*a*)′—C(8*a*)′—C(9)′	121·6 (2)
C(8*a*)′—C(9)—C(9)	122·1 (2)	C(9*a*)′—C(9)′—C(9)	122·0 (1)
C(9*a*)′—C(9)′—C(8*a*)′	115·7 (1)	C(4*a*)′—C(9*a*)′—C(1)′	119·6 (2)
C(9)′—C(9*a*)′—C(1)′	119·6 (2)	C(9)′—C(9*a*)′—C(4*a*)′	120·7 (2)

C(8*a*)—C(9)—C(9)′—C(8*a*)′	−0·8	C(8*a*)—C(9)—C(9)′—C(9*a*)′	−176·6
C(9*a*)—C(9)—C(9)′—C(8*a*)′	−177·4	C(9*a*)—C(9)—C(9)′—C(9*a*)′	6·7
C(1)···C(9)—C(9)′···C(1)′	56·1	C(1)···C(9)—C(9)′···C(8)′	−178·0
C(8)···C(9)—C(9)′···C(1)′	−177·5	C(8)···C(9)—C(9)′···C(8)′	−51·7
C(8*a*)—C(9)—C(9)′—C(9*a*)	176·6	C(9*a*)···C(9)—C(9)′—C(8*a*)′	175·8

C—*D* dihedral angles show the degree of folding of each half of the molecule, which is rather large [average 57·7 (3)°] in comparison to reported structures of related molecules (Harnik & Schmidt, 1954; Mills & Nyburg, 1963; Dichmann *et al.*, 1974; Bailey & Hull, 1978; Lee & Nyburg, 1985; Shoham *et al.*, 1988). The 'molecular torsion angles' C(1)—C(9)—C(9)′—C(1)′ ($\omega_{99'}^{11'}$) and C(8)—C(9)—C(9)′—C(8)′ ($\omega_{99'}^{88'}$) of 56·1 (4) and −51·7 (4)°, respectively, provide an alternative way to evaluate the degree of folding of the tricyclic halves and to estimate the deviation from planarity of the overall structure.

Most of the bond lengths, bond angles and torsion angles in (1) are within the accepted 'classical' ranges; however, distances and angles around the central double bond deviate considerably, as expected, from the corresponding structural values in non-strained systems. Three parameters indicate significant strain distortions (Shoham *et al.*, 1988) around C(9) and C(9)′: (*a*) The C(9)—C(9)′ bond [1·350 (2) Å] is slightly longer than the usual sp^2—sp^2 double bond. (*b*) The internal bond angles C(8*a*)—C(9)—C(9*a*) and C(8*a*)′—C(9)′—C(9*a*)′ [115·4 (1) and 115·7 (1)°, respectively] are smaller than in normal sp^2 carbon. (*c*) C(9) and C(9)′ are slightly out of the plane of the three carbons bonded to them [0·023 (5) and 0·029 (5) Å, respectively], reflecting a certain degree of pyramidalization of both C(9) and C(9)′. Another parameter, the torsion angle within the central ethylenic unit (Ermer, 1976), which is 6·8 (4)° for C(9*a*)—C(9)—C(9)′—C(9*a*)′ ($\omega_{99'}^{9a9a'}$) and −0·8 (4)° for C(8)—C(9)—C(9)′—C(8*a*)′ ($\omega_{99'}^{8a8a'}$), indicates a slight twisting of the otherwise planar double bond.

The unusual structural parameters of (1) discussed above demonstrate the modes used to relieve overcrowding in such a strained molecule. A combination of a considerable *folding* of each tricyclic unit in the *anti*-folded conformation with a slight *pyramidalization* of the central carbons and a slight *twisting* of

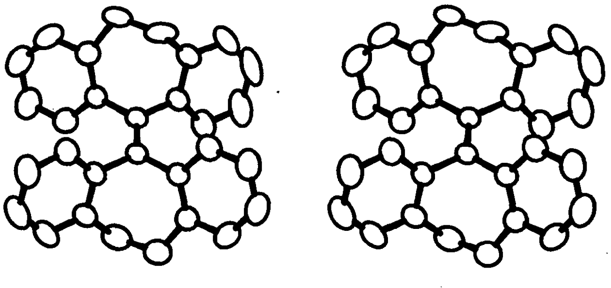

Fig. 2. Top stereoview of (1) (*ORTEP*II).

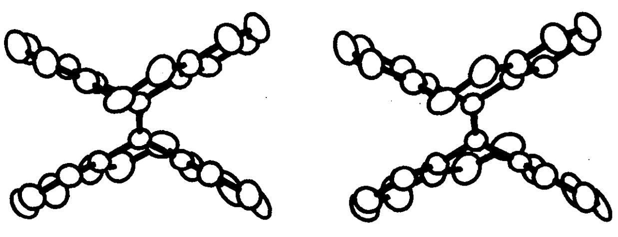

Fig. 3. Side stereoview of (1) (*ORTEP*II).

the central double bond, do indeed enable the relief of the overcrowding. The resulting C(1)—C(1)′ and C(8)—C(8)′ distances, which are 3·38 (1) and 3·39 (1) Å, respectively, are below the usual van der Waals distance between carbon atoms in a linear C—H···H—C arrangement (4·0 Å; Bondi, 1964; Baur, 1972); however, they are significantly longer than the calculated distances in a hypothetically coplanar bistricyclic ethylene. It should be noted that even with these compensating modes of distortion, molecule (1) does not cease to be overcrowded, although the degree of overcrowding has been considerably lowered.

We could not find a simple correlation between the degree of overcrowding, folding, pyramidalization and twisting in (1). Although various such modes should be considered concomitantly, in many cases it may be expected that no more than one of these modes will be predominant (Greenberg & Liebman, 1978). We expect, however, that as more structures of different bistricyclic ethylenes are analyzed, we will be able to understand and correlate distortion modes and overcrowding in strained molecules in general, and in bistricyclic ethylenes in particular.

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Structure of *cis*-4-Hydroxy-*N*-triphenylmethyl-L-proline Methyl Ester

BY G. STAVROPOULOS,* D. PAPAIOANNOU,* K. KARAGIANNIS,* M. VLASSI† AND V. NASTOPOULOS‡

Department of Chemistry, University of Patras, GR-261 10 Patras, Greece

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Abstract. $C_{25}H_{25}NO_3$, $M_r = 387·5$, monoclinic, $P2_1$, $a = 14·7875$ (28), $b = 8·7717$ (13), $c = 17·5559$ (19) Å, $\beta = 114·833$ (8)°, $V = 2066·6$ (6) Å^3, $Z = 4$, $D_x = 1·24$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6·58$ cm^{-1}, $F(000) = 824$, $T = 293$ K. Final $R = 0·049$ for 2763 observed reflections. The asymmetric unit contains two crystallographically independent molecules which show no significant differences in corresponding bond lengths or angles. These molecules are linked by an O—H···O hydrogen bond. The *N*-trityl group is oriented *trans* to the hydroxyl and methoxycarbonyl substituents.

Introduction. In a recent communication we reported the synthesis of *N*-tritylated derivatives of *cis*-4-

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* Division of Organic Chemistry, Biochemistry and Natural Products.

† Division of Analytical, Environmental and Applied Chemistry.

‡ Author to whom correspondence should be addressed.

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