**Discussion.** Le cycle benzénique *A* est plan. Les distances de ses atomes à son plan moyen *P(A)* ne dépassent pas 0,011 (3) Å. Dans l'hétérocycle *B*, les valeurs des angles de torsion endocycliques autour des liaisons voisines C(4)—C(4a) [−7,1 (4)°] et C(4a)—C(8a) [−4,4 (5)°] indiquent que O(1), C(3), C(4), C(4a) et C(8a) sont presque coplanaires. Ces atomes définissent un plan moyen *P(B)* dont C(2) est distant de 0,593 (4) Å. *B* possède donc approximativement la conformation sofa (1,2-diplanaire). Il en résulte que la symétrie dominante (Duax & Norton, 1975) est la symétrie par rapport à un plan passant par C(2) et par C(4a). L'écart à la symétrie idéale s'exprime par la relation $\Delta C_s^2 = 16,4°$.

Le cycle *C* est aussi du type sofa. Les valeurs des angles de torsion autour de C(4)—C(12) et de C(12)—C(13) sont respectivement de −8,5 (4) et de 1,0 (5)°. Les distances de C(4), C(12), C(13), C(14) et C(16) au plan moyen *P(C)*, calculées avec les mêmes atomes, appartiennent à l'intervalle 0,027 (3)– 0,066 (3) Å tandis que celle de C(15) à *P(C)* est de 0,659 (4) Å. D'autres conformations ont été observées avec des époxycyclohexanes substitués (Domiano & Macchia, 1980). La symétrie dominante est la symétrie par rapport à un plan passant par C(12) et par C(15). $\Delta C_s^{12} = 12,0°$. L'angle dièdre *P(B)P(C)* est égal à 90,7 (1)° et celui formé par *P(C)* avec le plan C(12)C(13)O(17) du cycle époxyde à 73,5 (2)°. La position du cycle époxyde par rapport à celle du cycle *C* est indiquée par la valeur de l'angle de torsion C(16)—C(4)—C(12)—O(17) qui, dans la molécule représentée par la Fig. 1, est de −77,0 (3)°.

Les liaisons O(1)—C(8a) [1,373 (4) Å] et C(8)— O(9) [1,372 (4) Å] sont plus courtes que leurs homo-logues O(1)—C(2) [1,433 (5) Å] et O(9)—C(10) [1,425 (4) Å] en raison de la conjugaison des élec-trons π du noyau benzénique avec les doublets non partagés de O(1) et de O(9). L'allongement de C(3)— C(4) [1,560 (5) Å] s'explique par l'encombrement stérique autour de C(4), tandis que le raccourcisse-ment de C(2)—C(3) [1,452 (5) Å] paraît dû à la rigi-dité de l'ensemble C(3)—C(4)—C(4a)—C(8a)—O(1), imposée par le noyau benzénique et par le groupe-ment spiranique, et à la présence de deux liaisons C—O nettement plus courtes que les autres liaisons du même cycle. Kissinger, Adman, Clark & Sten-kamp (1985) ont observé un léger raccourcissement de la même liaison [1,484 (8) Å] dans le sorbinil, lequel associe le benzopyrane et l'hydantoïne.

La Fig. 2 montre que la structure peut être con-sidérée comme formée de couches de molécules qui se développent le long des plans $x = \pm \frac{1}{8}$ et $x = \pm \frac{3}{8}$.

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# Structure of 1,1-Dichloro-2-(4-methoxyphenyl)-2,3-diphenylcyclopropane

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**Abstract.** $C_{22}H_{18}Cl_2O$, $M_r = 369.3$, monoclinic, $P2_1/a$, $a = 16.585$ (1), $b = 17.328$ (1), $c = 13.192$ (3) Å, $\beta = 107.443$ (8)°, $V = 3616.8$ Å$^3$, $Z = 8$, $D_x = 1.356$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71069 Å, $\mu = 3.2$ cm$^{-1}$, $F(000) = 1536$, $T = 138$ K, $R = 0.039$ for 5450 observed reflections. The structural features of the two independent molecules are quite similar except in the orientation of the methoxy group. The

cyclopropane ring shows the expected bond-length asymmetry with C(2)—C(3) as the longest bond. The two cis-arranged phenyl rings adopt similar conformations as observed in diarylcyclopropanes, with one ring in the bisecting position and the other near the perpendicular position. The conformation of the third aryl ring is also near the perpendicular position. The overall conformation of the three aryl rings is different from the helical propeller conformation consistently observed in tamoxifen and all other known tri(tetra)arylvinyl systems.

**Introduction.** The title compound (1) was synthesized (Day, Magarian, Jain, Mousissian & Meyer, 1992) as part of a continuing search for cyclopropyl antiestrogens with enhanced antiestrogenicity compared to the lead compound, 1,1-dichloro-2,3-cis-diphenylcyclopropane, analog II (2) (Magarian & Benjamin, 1975). Analog II is a weak antiestrogen but is shown to be devoid of estrogen agonist activity (Pento, Magarian, Wright, King & Benjamin, 1981) and is comparable to the well known triarylethylenic antiestrogen, tamoxifen (3), against the hormone-dependent 7,12-dimethylbenz[a]anthracene-induced rat mammary tumor model (Pento, Magarian & King, 1982).
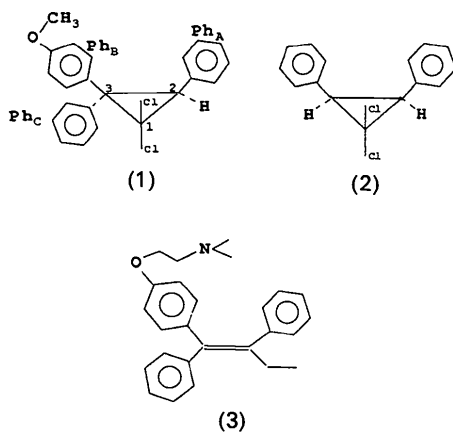


(1)            (2)



(3)

The present compound was prepared by introducing a third phenyl ring and a polar para substituent (a methoxy group) into (2) so that (1) resembles in part both the cyclopropyl antiestrogen (2) and the ethylenic antiestrogen, tamoxifen (3).

The crystal structure determination of the compound was undertaken to elucidate its structural features and compare them with those of tamoxifen and its derivatives.

**Experimental.** Clear colorless crystal, 0.20 × 0.24 × 0.90 mm; Enraf–Nonius CAD-4 diffractometer equipped with liquid nitrogen low-temperature device; Mo $K\alpha$ radiation; lattice parameters from

Table 1. *Positional and equivalent isotropic thermal parameters ($\text{Å}^2$) of non-H atoms with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| **Molecule A** | | | | |
| Cl(1) | 0.52063 (3) | 0.09034 (3) | 0.72531 (4) | 0.0268 (2) |
| Cl(2) | 0.39921 (4) | 0.10407 (3) | 0.84152 (4) | 0.0279 (2) |
| C(1) | 0.4353 (1) | 0.1440 (1) | 0.7401 (2) | 0.0217 (8) |
| C(2) | 0.3687 (1) | 0.1753 (1) | 0.6439 (2) | 0.0206 (8) |
| C(3) | 0.4300 (1) | 0.2302 (1) | 0.7227 (2) | 0.0214 (8) |
| C(21) | 0.3641 (1) | 0.1571 (1) | 0.5313 (2) | 0.0207 (8) |
| C(22) | 0.4327 (1) | 0.1554 (1) | 0.4910 (2) | 0.0238 (8) |
| C(23) | 0.4228 (2) | 0.1319 (1) | 0.3874 (2) | 0.0267 (9) |
| C(24) | 0.3439 (1) | 0.1101 (1) | 0.3217 (2) | 0.0282 (9) |
| C(25) | 0.2752 (1) | 0.1127 (1) | 0.3604 (2) | 0.0275 (8) |
| C(26) | 0.2852 (1) | 0.1360 (1) | 0.4637 (2) | 0.0223 (8) |
| C(31) | 0.4958 (1) | 0.2744 (1) | 0.6890 (2) | 0.0213 (8) |
| C(32) | 0.5807 (1) | 0.2766 (1) | 0.7512 (2) | 0.0222 (8) |
| C(33) | 0.6373 (1) | 0.3252 (1) | 0.7246 (2) | 0.0228 (8) |
| C(34) | 0.6107 (1) | 0.3722 (1) | 0.6357 (2) | 0.0212 (8) |
| C(35) | 0.5271 (1) | 0.3700 (1) | 0.5723 (2) | 0.0237 (8) |
| C(36) | 0.4706 (1) | 0.3215 (1) | 0.5998 (2) | 0.0236 (8) |
| O(1) | 0.6714 (1) | 0.4189 (1) | 0.6159 (1) | 0.0251 (6) |
| C(4) | 0.6421 (2) | 0.4793 (1) | 0.5402 (2) | 0.032 (1) |
| C(31') | 0.3948 (1) | 0.2779 (1) | 0.7957 (2) | 0.0227 (8) |
| C(32') | 0.3174 (1) | 0.3150 (1) | 0.7584 (2) | 0.0298 (9) |
| C(33') | 0.2874 (2) | 0.3609 (1) | 0.8258 (2) | 0.035 (1) |
| C(34') | 0.3344 (2) | 0.3700 (1) | 0.9302 (2) | 0.031 (1) |
| C(35') | 0.4115 (2) | 0.3339 (1) | 0.9681 (2) | 0.034 (1) |
| C(36') | 0.4422 (1) | 0.2884 (1) | 0.9014 (2) | 0.0293 (9) |
| **Molecule B** | | | | |
| Cl(1) | 0.62003 (4) | 0.10667 (4) | 1.00631 (4) | 0.0309 (2) |
| Cl(2) | 0.50450 (3) | 0.11503 (4) | 1.13205 (5) | 0.0327 (2) |
| C(1) | 0.6044 (1) | 0.1430 (1) | 1.1233 (2) | 0.0241 (8) |
| C(2) | 0.6341 (1) | 0.2230 (1) | 1.1600 (2) | 0.0232 (8) |
| C(3) | 0.6771 (1) | 0.1520 (1) | 1.2245 (2) | 0.0191 (7) |
| C(21) | 0.6728 (1) | 0.2800 (1) | 1.1033 (2) | 0.0223 (8) |
| C(22) | 0.7361 (1) | 0.2634 (1) | 1.0569 (2) | 0.0270 (9) |
| C(23) | 0.7710 (2) | 0.3211 (1) | 1.0113 (2) | 0.031 (1) |
| C(24) | 0.7430 (2) | 0.3961 (2) | 1.0098 (2) | 0.035 (1) |
| C(25) | 0.6789 (2) | 0.4134 (1) | 1.0530 (2) | 0.034 (1) |
| C(26) | 0.6444 (1) | 0.3557 (1) | 1.0993 (2) | 0.0267 (9) |
| C(31) | 0.7656 (1) | 0.1286 (1) | 1.2288 (2) | 0.0185 (7) |
| C(32) | 0.7831 (1) | 0.0558 (1) | 1.1970 (2) | 0.0211 (8) |
| C(33) | 0.8652 (1) | 0.0338 (1) | 1.2033 (2) | 0.0226 (8) |
| C(34) | 0.9310 (1) | 0.0844 (1) | 1.2447 (2) | 0.0240 (8) |
| C(35) | 0.9151 (1) | 0.1568 (1) | 1.2807 (2) | 0.0248 (8) |
| C(36) | 0.8331 (1) | 0.1780 (1) | 1.2727 (2) | 0.0232 (8) |
| O(1) | 1.0147 (1) | 0.0687 (1) | 1.2540 (1) | 0.0322 (6) |
| C(4) | 1.0321 (2) | −0.0043 (2) | 1.2150 (3) | 0.041 (1) |
| C(31') | 0.6598 (1) | 0.1393 (1) | 1.3292 (2) | 0.190 (7) |
| C(32') | 0.6601 (1) | 0.2011 (1) | 1.3960 (2) | 0.0245 (8) |
| C(33') | 0.6511 (1) | 0.1893 (1) | 1.4959 (2) | 0.0284 (9) |
| C(34') | 0.6410 (1) | 0.1161 (1) | 1.5295 (2) | 0.0277 (9) |
| C(35') | 0.6380 (1) | 0.0541 (1) | 1.4626 (2) | 0.0270 (9) |
| C(36') | 0.6482 (1) | 0.0656 (1) | 1.3631 (2) | 0.0233 (8) |

setting of 48 reflections with $18 < 2\theta < 24°$; 7479 unique reflections were measured using the $\omega$–$2\theta$ scan technique with $2\theta < 53°$, $-20 < h < 20$, $0 < k < 21$, $0 < l < 16$; scan width $(0.80 + 0.20\tan\theta)°$, extended 25% on each side for background measurement, horizontal aperture $(2.40 + 0.86\tan\theta)$ mm, vertical aperture 4 mm; three standard reflections (555, 11,1,3, 4,10,5) measured every 7200 s of X-ray exposure, maximum variation 2.5%; intensity data reductions by applying the peak profile analysis program *DREAM* (Blessing, 1987); Lorentz and polarization corrections, but no absorption correction; 5450 observed reflections with $I > 2\sigma(I)$. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1986), refined by full-matrix least squares

using *SHELX*76 (Sheldrick, 1976); $\sum w(F_o - F_c)^2$ minimized, where $w = 1.804/\sigma^2(F_o)$; all H atoms located from difference Fourier maps and refined with isotropic temperature factors; final $R = 0.039$, $wR = 0.033$ for 5450 observed reflections; $(\Delta/\sigma)_{max} = 0.03$, $S = 2.0$ for 596 variables; highest/lowest peaks in the final difference map $0.3$ e Å$^{-3}$; atomic scattering factors from *SHELX*76.

**Discussion.** The positional and thermal parameters of the non-H atoms are listed in Table 1. There are two independent molecules, molecule $A$ and molecule $B$, in the asymmetric unit. A stereoview of molecule $A$ is shown in Fig. 1 with atom labelling.

Selected bond distances, bond angles and torsion angles are given in Table 2.* The structures of molecules $A$ and $B$ are essentially the same with the exception that in molecule $B$ the methoxy moiety has a different conformation than that in molecule $A$: torsion angle C(33)—C(34)—O(1)—C(4) is $-166.4 (3)°$ in molecule $A$ and $1.8 (3)$ in molecule $B$. In addition, molecule $B$ shows a light twist about the C(2)—C(3) bond with torsion angle C(21)—C(2)—C(3)—C(31) $-5.4 (3)°$, while this angle is close to $0°$ in molecule $A$ and in all other known *cis*-diphenylcyclopropanes (Hossain, Wang, van der Helm, Magarian, Griffin & Day, 1991).

The bond distances and bond angles observed in the present structure are generally in good agreement with those observed in other 1,1-dichlorocyclopropane derivatives (Hossain *et al.*, 1991). One notable difference is the slightly longer bond lengths of the two bridging bonds, C(3)—C(31) = 1.507 (3) and C(3)—C(31′) = 1.511 (3) Å, compared to C(2)—C(21) = 1.496 (3) Å and the similar bridging bonds in the diphenylcyclopropane derivatives. Such a

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* Lists of anisotropic thermal parameters, H-atom parameters, complete bond distances and bond angles, and calculated and observed structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54672 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
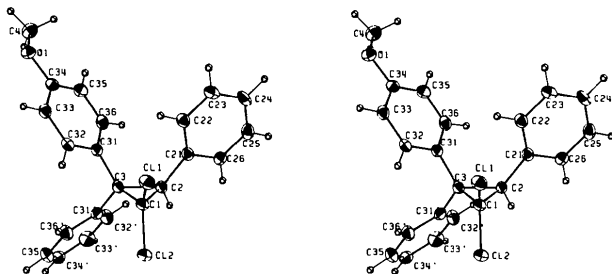


Fig. 1. Stereoview of an *ORTEP*II (Johnson, 1976) plot of molecule $A$. Atom labelling as in the text. Thermal ellipsoids 50% probability.

Table 2. *Selected bond distances (Å), angles (°) and torsion angles (°) with e.s.d.'s in parentheses*

| | Mol. *A* | Mol. *B* | Mean value |
|---|---|---|---|
| C(1)—Cl(1) | 1.752 (2) | 1.757 (2) | 1.755 (2) |
| C(1)—Cl(2) | 1.763 (2) | 1.763 (2) | 1.763 (2) |
| C(1)—C(2) | 1.511 (3) | 1.502 (3) | 1.507 (3) |
| C(1)—C(3) | 1.510 (3) | 1.515 (3) | 1.513 (3) |
| C(2)—C(3) | 1.544 (3) | 1.544 (3) | 1.544 (3) |
| C(2)—C(21) | 1.498 (2) | 1.494 (3) | 1.496 (3) |
| C(3)—C(31) | 1.507 (3) | 1.507 (2) | 1.507 (3) |
| C(3)—C(31′) | 1.513 (3) | 1.509 (2) | 1.511 (3) |
| C(34)—O(1) | 1.376 (2) | 1.383 (3) | 1.380 (3) |
| O(1)—C(4) | 1.428 (3) | 1.427 (3) | 1.428 (3) |
| | | | |
| Cl(1)—C(1)—Cl(2) | 110.2 (1) | 110.4 (1) | 110.3 (1) |
| Cl(1)—C(1)—C(2) | 120.6 (1) | 119.6 (1) | 120.1 (1) |
| Cl(1)—C(1)—C(3) | 121.4 (1) | 121.5 (1) | 121.5 (1) |
| Cl(2)—C(1)—C(2) | 116.7 (1) | 117.2 (1) | 117.0 (1) |
| Cl(2)—C(1)—C(3) | 119.0 (1) | 119.1 (1) | 119.0 (1) |
| C(2)—C(1)—C(3) | 61.5 (1) | 61.6 (1) | 61.6 (1) |
| C(1)—C(2)—C(3) | 59.2 (1) | 59.6 (1) | 59.4 (1) |
| C(1)—C(3)—C(2) | 59.3 (1) | 58.8 (1) | 59.0 (1) |
| C(1)—C(2)—C(21) | 124.3 (2) | 126.7 (1) | 125.5 (1) |
| C(3)—C(2)—C(21) | 128.7 (2) | 127.3 (2) | 128.0 (2) |
| C(1)—C(3)—C(31) | 122.2 (2) | 121.0 (1) | 121.6 (2) |
| C(2)—C(3)—C(31) | 120.2 (1) | 121.3 (2) | 120.8 (2) |
| C(1)—C(3)—C(31′) | 117.5 (2) | 118.2 (2) | 117.9 (2) |
| C(2)—C(3)—C(31′) | 117.7 (2) | 115.9 (2) | 116.8 (2) |
| C(31)—C(3)—C(31′) | 111.2 (2) | 112.1 (2) | 111.7 (2) |
| C(2)—C(21)—C(22) | 125.3 (2) | 125.1 (2) | 125.2 (2) |
| C(2)—C(21)—C(26) | 116.6 (2) | 116.9 (2) | 116.8 (2) |
| C(3)—C(31)—C(32) | 122.1 (2) | 121.5 (2) | 121.8 (2) |
| C(3)—C(31)—C(36) | 119.2 (2) | 120.3 (2) | 119.8 (2) |
| C(33)—C(34)—O(1) | 116.2 (2) | 124.3 (2) | – |
| C(35)—C(34)—O(1) | 123.8 (2) | 115.8 (2) | – |
| C(34)—O(1)—C(4) | 116.7 (2) | 116.6 (2) | 116.7 (2) |
| C(3)—C(31′)—C(32′) | 121.3 (2) | 120.3 (2) | 120.8 (2) |
| C(3)—C(31′)—C(36′) | 119.9 (2) | 120.8 (2) | 120.4 (2) |
| | | | |
| C(21)—C(2)—C(3)—C(31) | 0.5 (3) | -5.3 (3) | |
| C(21)—C(2)—C(3)—C(31′) | 141.5 (3) | 136.3 (3) | |
| C(3)—C(2)—C(21)—C(22) | 32.2 (3) | 31.3 (3) | |
| C(2)—C(3)—C(31)—C(36) | 55.8 (3) | 62.4 (2) | |
| C(2)—C(3)—C(31′)—C(32′) | -43.7 (3) | -42.3 (3) | |
| C(33)- C(34)—O(1)—C(4) | -166.4 (2) | 1.8 (3) | |
| *M*(13)*—C(2)—C(21)—C(22) | -6.4 (4) | -6.8 (4) | |
| *M*(12)*—C(3)—C(31)—C(32) | -97.2 (3) | -87.8 (3) | |
| *M*(12)*—C(3)—C(31′)—C(32′) | -77.2 (3) | -75.0 (3) | |

* *M*(13), *M*(12) are the midpoints of C(1)—C(3) and C(1)—C(2), respectively.

lengthening may be attributed to spatial overcrowding around the C(3) atom, as seen in 1,1,2,2-tetraphenylcyclopropanes (Cameron, Linden & Jochem, 1990).

The cyclopropane ring shows the commonly observed bond-length asymmetry with a much longer C(2)—C(3) bond of 1.544 (3) Å compared to a C(1)—C(2) distance of 1.507 (3) Å and a C(1)—C(3) distance of 1.512 (3) Å. This bond-length asymmetry approximately corresponds to the expectations for the substituent effects (Allen, 1980): (i) the two Cl substituents at C(1) cause lengthening of the distal bond C(2)—C(3) and shortening of the two vicinal bonds, C(1)—C(2) and C(1)—C(3); (ii) the phenyl substituent at C(2) causes the lengthening of the two vicinal bonds, C(1)—C(2) and C(2)—C(3), and shortening of C(1)—C(3) bond; (iii) the two phenyl substituents at C(3) cause lengthening of C(2)—C(3) and C(1)—C(3) and shortening of the distal C(1)—C(2) bond. Both Cl—C distances, Cl(1)—C(1) = 1.754 (2) and Cl(2)—C(1) = 1.763 (2) Å, are shorter

than the typical Cl—C single bond: 1.790–1.805 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). This indicates that there is some conjugation between Cl atoms and the cyclopropane ring. The Cl(1)—C(1) distance is slightly shorter than the Cl(2)—C(1) distance. Such asymmetry in the two Cl—C distances has been observed in other *gem*-dichlorocyclopropyl compounds (Hossain *et al.*, 1991).

The phenyl ring at C(2), $Ph_A$, adopts the bisecting conformation, while the two phenyl rings at C(3), $Ph_B$ and $Ph_C$, are both in the perpendicular conformation. The conformational parameter $\theta$ is defined as the acute angle between the normal of the plane of the phenyl ring and the C—C vector of the distal bond of the cyclopropane ring, and is 0° for bisecting and 90° for perpendicular conformations (Jason & Ibers, 1977). In the present structure $\theta$ for ring $Ph_A$ is 10.7° (molecule *A*) and 7.0° (molecule *B*); for ring $Ph_B$, 89.4° in molecule *A* and 84.0° in molecule *B*; for ring $Ph_C$, 77.0° in molecule *A* and 76.0° in molecule *B*. The conformation of the two *cis*-arranged phenyl rings, $Ph_A$ and $Ph_B$, is similar to that observed in the three *cis*-diphenylcyclopropanes (Hossain *et al.*, 1991) and is consistent with the propeller conformation prevalent in the stilbene derivative structures. However, the nearly perpendicular conformation of the third ring $Ph_C$ gives rise to a totally different overall conformation for the molecule from that of tamoxifen (Précigoux, Courseille, Geoffre & Hospital, 1979) and its derivatives. Fig. 2 shows a stereoview of the superimposed molecules of the present structure and tamoxifen.

From structure surveys of a large number of tri- and tetraarylvinyl systems, it has been shown (Kaftory, Biali & Rappaport, 1985; Duax & Griffin, 1987) that in all these structures, without exception, the three aryl rings are rotated in the same sense from the plane of the double bond, giving a helical propeller conformation for the molecule. Relevant torsion angles of some of these structures are listed in Table 3 for comparison with the present structure.

In all these structures, the three torsion angles, $\varphi_1$, $\varphi_2$ and $\varphi_3$, which define the aryl ring conformation

Table 3. *Torsion angles (°) defining phenyl-ring conformations in some triarylvinyl systems, and tetra- and triarylcyclopropanes*



Vinyl            Cyclopropyl

$\varphi_1 = a—b—d—f \quad \varphi_2 = b—a—c—e \quad \varphi_3 = b—a—g—h$
$\varphi_4 = c—a—b—d \quad \varphi_5 = d—b—a—g$

| Compound* | $\varphi_1$ | $\varphi_2$ | $\varphi_3$ | $\varphi_4$ | $\varphi_5$ |
|---|---|---|---|---|---|
| Vinyl | | | | | |
| (i) | 55.2 | 47.9 | 64.3 | 8.5 | 174.4 |
| (ii) | 57.7 | 49.5 | 66.0 | 7.5 | −176.2 |
| (iii) | 60.2 | 56.2 | 60.1 | 3.7 | −178.5 |
| (iv) | 59.6 | 43.1 | 67.9 | 9.9 | −170.9 |
| (v) | 55.3 | 47.5 | 56.2 | 10.2 | −168.0 |
| (vi) | 45.0 | 49.5 | 63.7 | 6.7 | 175.1 |
| (vii) | 50.8 | 48.9 | 63.4 | 9.8 | 169.6 |
| (viii) | 48.0 | 42.3 | 47.2 | 10.5 | −169.7 |
| (ix) | 56.6 | 54.2 | 55.0 | 8.0 | 174.5 |
| (x) | 48.6 | 42.9 | 46.1 | 11.0 | −172.2 |
| Cyclopropyl | | | | | |
| (xi) | −65.6 | −64.2 | −67.9 | 3.1 | 148.8 |
| (xii) | −50.8 | −60.1 | −63.5 | −4.0 | 144.5 |
| (xiii) | −59.9 | −61.1 | −63.8 | 1.3 | 148.1 |
| (xiv) | 32.2 | 55.7 | −43.7 | 0.5 | 141.5 |
| | 31.2 | 62.3 | −42.3 | −5.3 | 136.5 |

*(i) Tamoxifen (Précigoux *et al.*, 1979); (ii) 2-hydroxytamoxifen (Kuroda, Cutbush, Neidle & Leung, 1985); (iii) 3-hydroxytamoxifen (Kuroda *et al.*, 1985); (iv) 4-hydroxy-2-methyltamoxifen (Kuroda *et al.*, 1985); (v) (*E*)-1-iodotamoxifen (Hunter, Payne, Rahman, Richardson & Zea Ponce, 1983); (vi) (*Z*)-1-iodotamoxifen (Hunter *et al.*, 1983); (vii) 1-bromo-2-(*p*-ethylphenyl)-1,2-diphenylethylene (Fornies-Marquina, Courseille, Busetta & Hospital, 1972); (viii) 3,3-bis(*p*-methoxyphenyl)-2-phenylacrylonitrile (Barrans, Précigoux, Hospital, Sekera & Miquel, 1979); (ix) clomiphene hydrochloride (Ernst & Hite, 1976); (x) 1-[*p*-(2-bromoethoxy)phenyl]-2-chloro-1,2-diphenylethylene (Précigoux, Hospital, Leroy, Delbarre & Roques, 1982); (xi) tetraphenylcyclopropane derivative (I) (Cameron *et al.*, 1990); (xii) tetraphenylcyclopropane derivative (II) (Cameron *et al.*, 1990); (xiii) tetraphenylcyclopropane derviative (III) (Cameron *et al.*, 1990); (xiv) present structure.
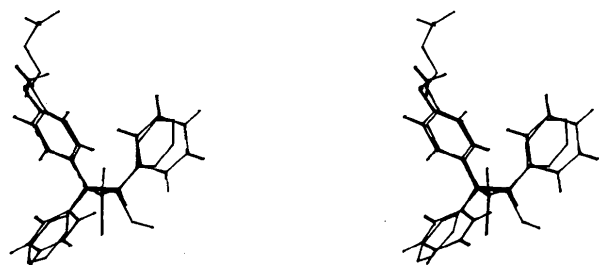


Fig. 2. Stereosuperposition of molecule *A* (thick bond) and tamoxifen (thin bond) (Précigoux *et al.*, 1979).

are remarkably consistent with small variations in individual angles. It has been shown (Kaftory *et al.*, 1985) that for all known triarylvinyl—*X* systems, the torsion angles lie within the region $\varphi_1 = 40$–65, $\varphi_2 = 41$–76 and $\varphi_3 = 40$–76°. Even in the three tetraphenylcyclopropane structures listed in Table 3, three of the aryl rings are arranged in a manner consistent with the helical propeller conformation. The present structure with torsion angles $\varphi_1 = 32.2$, $\varphi_2 = 55.7$ and $\varphi_3 = -43.7°$ (molecule *A*) clearly has a non-helical conformation and is the only exception.

It should be noted that a direct comparison of a triarylcyclopropyl system with a triarylvinyl system is only approximate because of the geometrical constraint imposed by the cyclopropane ring which forces the torsion angle $\varphi_5$ to be around 140°, while in all vinyl systems this angle lies within 10–12° of a perfect *trans* geometry ($\varphi_5 = 180°$). In addition, all the vinyl systems show a twist of about 5–10° about the double bond given by the angle $\varphi_4$. In the cyclopropyl system, $\varphi_4$ is close to 0° except in molecule (*B*) of the present structure ($\varphi_4 = -5.4°$).

Energy minimization calculations by using the molecular mechanics program *MM*2 showed that the crystal conformation is close to the global energy minimum in the conformational space (Hossain, Du, Symersky, Ji, van der Helm & Magarian, 1991). It therefore appears that this unique non-propeller conformation for the triaryl rings in the present structure is very likely a consequence of the constraint imposed by the cyclopropane ring with *gem*-dichloro substituent at C(1). However, more triarylcyclopropane (with and without dichloro substituents) structural results, including a systematic calculation of steric energies of such systems, are required to make any definitive conclusion. Work in both of these areas is in progress.

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# Structure of Asperketal B

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**Abstract.** 3,7,11-Trimethyl-13-oxabicyclo[8.3.0]trideca-2,6-diene-12-spiro-2′-(5′,5′-dimethyltetrahydrofuran), asperketal B, $C_{20}H_{30}O_2$, $M_r = 302.46$, orthorhombic, $P2_12_12_1$, $a = 10.064\,(1)$, $b = 22.214\,(2)$, $c = 8.330\,(1)$ Å, $V = 1862.3$ Å$^3$, $Z = 4$, $D_x = 1.08$ g cm$^{-3}$, Cu $K\bar{\alpha}$, $\lambda = 1.54178$ Å, $\mu = 4.55$ cm$^{-1}$, $F(000) = 664$, $T = 294\,(1)$ K, $R = 0.031$, $wR = 0.027$ for 953 data. The backbone of the molecule is composed of a cyclodecadiene ring which is *cis* fused to a bicyclic ketal system. The relative configurations at C(1), C(10), C(11) and C(12) are 1$S^*$, 10$R^*$, 11$R^*$ and 12$R^*$.

**Introduction.** Asperketal B (I) is one of the six new diterpenoids isolated from the Caribbean sea whip *Eunicea asperula*, for which the structures and the relative configurations have been assigned on the basis of chemical and spectral studies (Shin &