

The poor quality of the crystals of (2) and their weak diffraction power are probably responsible for the relatively high value for *R*. In this case, *DIFABS* (Walker & Stuart, 1983) was used for an absorption correction as the appropriate number of strong reflections with useful setting angles was not found.

For both compounds, data collection: *P2*<sub>1</sub> software; cell refinement: *P2*<sub>1</sub> software; data reduction: *PRADIR* (Jaskólski, 1990); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL93*.

This study was supported jointly by projects 2 0759 91 01 KBN and S/II.

---

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms for compound (1), bond distances and angles involving H atoms for compound (2), and torsion angles have been deposited with the IUCr (Reference: AB1206). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

---

## References

- Allen, F. H. (1981). *Acta Cryst.* B37, 900–906.  
 Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.  
 Antkowiak, R., Antkowiak, W. Z. & Czerwiński, G. (1990). *Tetrahedron*, **46**, 2445–2452.  
 Antkowiak, W. Z. & Gessner, W. P. (1979). *Tetrahedron Lett.* **20**, 1931–1934.  
 Antkowiak, W. Z. & Gessner, W. P. (1984). *Tetrahedron Lett.* **25**, 4045–4048.  
 Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.  
 Bou-Abdallah, E., Jossang, A., Tadic, D. & Cave, A. (1989). *J. Nat. Prod.* **52**, 273–278.  
 Jaskólski, M. (1990). *PRADIR. Program for Data Reduction*. Adam Mickiewicz Univ., Poznań, Poland.  
 Kennard, C. H. L., Smith, G. & Katekar, G. F. (1981). *Aust. J. Chem.* **34**, 1143–1146.  
 Koyama, J., Okatani, T., Tagahara, K. & Irie, H. (1989). *Heterocycles*, **29**, 1649–1654.  
 Kubicki, M., Borowiak, T. & Antkowiak, W. Z. (1995). *Acta Cryst.* C51, 458–461.  
 Kubicki, M., Borowiak, T., Antkowiak, R. & Antkowiak, W. Z. (1990). *J. Crystallogr. Spectrosc. Res.* **20**, 381–387.  
 Kubicki, M., Borowiak, T., Antkowiak, R., Antkowiak, W. Z. & Chruścicki, H. (1994). *Recl Trav. Chim. Pays-Bas.* **113**, 383–389.  
 Kubicki, M., Borowiak, T., Chruścicki, H. & Antkowiak, W. Z. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 113–118.  
 Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.  
 Siemens (1989). *Stereochemical Workstation*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sørensen, G. O., Mahler, L. & Rastrup-Andersen, N. (1974). *J. Mol. Struct.* **20**, 119–124.  
 Tulip, T. H., Corfield, P. W. R. & Ibers, J. A. (1978). *Acta Cryst.* B34, 1549–1555.  
 Walker, N. & Stuart, D. (1983). *Acta Cryst.* A39, 158–166.  
 Zenger, R., Rhine, W. & Stucky, G. D. (1974). *J. Am. Chem. Soc.* **96**, 5441–5448.

*Acta Cryst.* (1995). C51, 465–468

## A Baccatin III Derivative

ANGÈLE CHIARONI, DANIEL GUÉNARD,  
 FRANÇOISE GUÉRITTE-VOEGELEIN,  
 FRANÇOISE KHUONG-HUU, SANDRINE PY,  
 CLAUDE RICHE AND ANNE WAHL

*Institut de Chimie des Substances Naturelles, CNRS,  
 91198 Gif sur Yvette CEDEX, France*

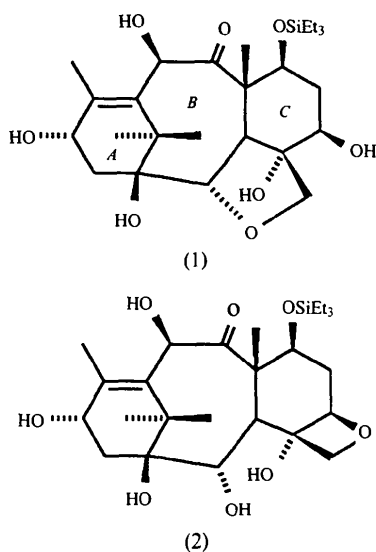
(Received 12 May 1994; accepted 26 June 1994)

## Abstract

The title compound, 3,4,6,8,11-pentahydroxy-4-demethyl-1-triethylsiloxy-5,4-epoxymethanotaxen-12-one (C<sub>26</sub>H<sub>44</sub>O<sub>8</sub>Si), has been prepared in the search for new semi-synthetic analogues of taxol. It has been obtained from 2-debenzoyl-4,10-bisdeacetyl-7-triethylsilyl-baccatin III when treated in an alkaline or acidic medium. The structure was established as part of a contribution to the knowledge of structure–activity relationships. An intramolecular hydrogen bond is established between the hydroxyl groups HO13 and HO4. In addition, a hydrogen-bond network engaging all the hydroxyl groups of the molecule links the different molecules in the crystal.

## Comment

Taxol paclitaxel, a diterpene isolated in only low yield from the bark of several species of the *Taxus* genus (Wani, Taylor, Wall, Coggon & McPhail, 1971) and taxotere, a semi-synthetic analogue, are two of the most promising new drugs studied in the field of cancer chemotherapy (Guénard, Guéritte-Voegelein & Potier, 1993; Rose, 1992; Horwitz, 1992; Therre, 1993). They act by a unique mechanism: both promoting the assembly of tubulin into microtubules and stabilizing the microtubule assembly against depolymerization. The knowledge of the three-dimensional structure of either active or inactive compounds in this series is important in order to establish structure–activity relationships. The title compound, (1), has been obtained by rearrangement of 2-debenzoyl-4,10-bisdeacetyl-7-triethylsilyl-baccatin III (2) in an alkaline or acidic medium (Wahl, Guéritte-Voegelein, Guénard, Le Goff & Potier, 1992; Farina & Huang, 1992), 10-deacetyl-baccatin III being a natural precursor of taxol isolated from yew leaves (Chauvière, Guénard, Picot, Senilh & Potier, 1981). After introduction of the taxotere-O13-side-chain in compound (1), the product thus obtained shows no inhibition of the rate of disassembly (IC<sub>50</sub> = 0) of tubulin.



The structure of (1) with atomic labelling is shown in Fig. 1. The absolute configuration is that of the taxane tricyclic (A, B, C) ring system (Wani, Taylor, Wall, Coggon & McPhail, 1971).

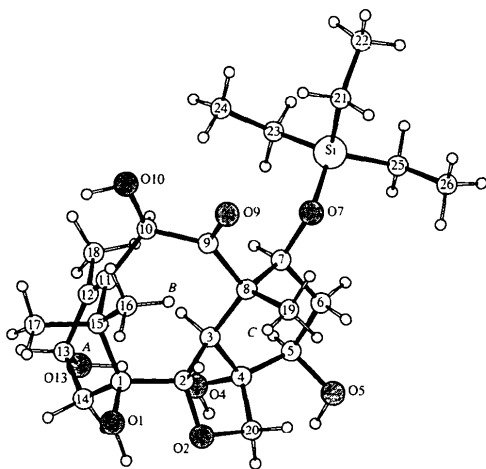


Fig. 1. Perspective view of the molecule.

The cyclooctane ring (B) adopts the most stable boat-chair conformation, as shown by the torsion angles with atoms C1 and C9 as the ends. This ring is *trans*-fused along the C3—C8 bond to the six-membered ring C, which exhibits a chair conformation flattened in C4 [atoms C7 and C4 deviated by 0.76(2) and  $-0.34(1)$  Å, respectively, from the mean plane of the other four atoms]. The adjacent tetrahydrofuran ring, fused at C2, C3 and C4, is envelope-shaped with C20 as the flap at  $-0.52(1)$  Å from the mean plane formed by atoms C2, C3, C4 and O2. Ring A, 'double-bridged' to the central ring, exhibits the 1,3-diplanar boat conformation with C11 only 0.36(1)

and C15 0.98(1) Å fully above the mean plane of the atoms C13, C12, C14 and C1 [maximum deviation: 0.01(1) Å]. These conformations are more or less identical for all the baccatin derivatives resolved by X-ray analysis: baccatin V (Castellano & Hodder, 1973), *O*-cinnamoyltaxicin-I triacetate (Begley, Frecknall & Pattenden, 1984), 14 $\beta$ -hydroxy-10-deacetyl baccatin III (Appendino *et al.*, 1992), *N*-*tert*-butoxycarbonyl-10-deacetyl-*N*-debenzoyltaxol (Guéritte-Voegelein *et al.*, 1990), 9-dihydro-13-acetyl-baccatin III (Gunawardana *et al.*, 1992).

It can be noted that all the hydroxyl groups are engaged in hydrogen bonds. A strong intramolecular hydrogen bond (2.78 Å) appears between HO13 and the O atom of HO4 with the following characteristics: O13—H $\cdots$ O4 = 2.78(2), H(O13) $\cdots$ O4 = 1.82 Å; O13—H $\cdots$ O4 = 160.2°.

In the crystal (Fig. 2), the molecules are linked together in the direction of the *a* axis through two hydrogen bonds of 2.82 and 2.94 Å. These hydrogen bonds are established between the hydroxyl groups HO4 and HO5 of one molecule and the carbonyl O9 atom of the next molecule according to the following scheme: O4—H $\cdots$ O9( $x - 1, y, z$ ) = 2.82(2), HO4 $\cdots$ O9 = 1.84 Å, O4—H $\cdots$ O9 = 164.4°; O5—H $\cdots$ O9( $x - 1, y, z$ ) = 2.94(2), HO5 $\cdots$ O9 = 2.01 Å, O5—H $\cdots$ O9 = 153.1°.

These chains of molecules are bound to chains of identical molecules of symmetry ( $1 - x, \frac{1}{2} + y, 2 - z$ ) by means of two other hydrogen bonds, O10—H $\cdots$ O1 and O1—H $\cdots$ O13, according to the following scheme: O10—H $\cdots$ O1H( $2 - x, \frac{1}{2} + y, 2 - z$ ) = 2.88(2), H(O10) $\cdots$ O1 = 2.04 Å, O10—H $\cdots$ O1 = 139.5°; O1—H $\cdots$ O13( $1 - x, y - \frac{1}{2}, 2 - z$ ) = 2.66(2), H(O1) $\cdots$ O13 = 1.71 Å, O1—H $\cdots$ O13 = 155.7°.

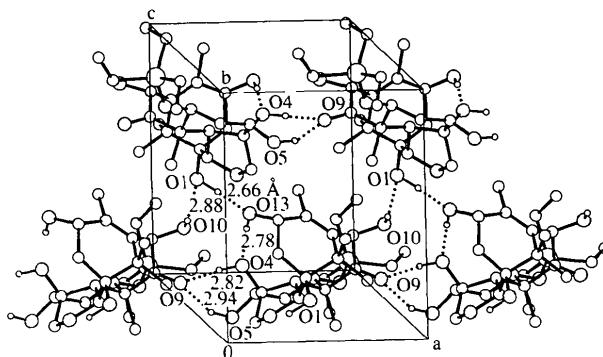


Fig. 2. Packing of the molecules showing the hydrogen-bond network.

### Experimental

(1) was obtained as described in the literature (Wahl, Guéritte-Voegelein, Guénard, Le Goff & Potier, 1992; Farina & Huang, 1992). (1) has m.p. 401–403 K (CH<sub>2</sub>Cl<sub>2</sub>) and  $[\alpha]_D = -42$  (EtOH,  $c = 1.04$ ). Peaks at  $m/z$  535 [ $M + Na$ ]<sup>+</sup>,

513 [M + H]<sup>+</sup> and 495 [M + H - H<sub>2</sub>O] can be found in the FAB mass spectrum. The IR spectrum shows absorption at  $\nu = 3430, 2960, 1695, 1600 \text{ cm}^{-1}$ . The chemical shifts,  $\delta$  p.p.m., of the protons in the <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub> + 10% C<sub>5</sub>D<sub>5</sub>N) are: 0.37 (6H, *q*, *J* = 8, CH<sub>2</sub>Si), 0.78 (9H, *t*, *J* = 8, CH<sub>3</sub>CH<sub>2</sub>Si), 0.85 (3H, *s*, C16H<sub>3</sub>), 0.98 (3H, *s*, C17H<sub>3</sub>), 1.23 (3H, *s*, C19H<sub>3</sub>), 1.75 (1H, *m*, C6H $\beta$ ), 1.91 (3H, *s*, C18H<sub>3</sub>), 2.13 (1H, *m*, C6H $\alpha$ ), 2.41 (1H, *dd*, *J* = 9, *J'* = 15, C14H), 2.58 (1H, *dd*, *J* = 1, *J'* = 15, C14H), 3.49 (1H, *d*, *J* = 7, C3H), 3.70 and 3.85 (2H, *dd*, *J* = 10, C20H<sub>2</sub>), 3.76 (1H, *dd*, *J* = 11, *J'* = 5, C7H), 4.04 (1H, *d*, *J* = 7, C2H), 4.26 (1H, *t*, *J* = 9, C5H), 4.34 (1H, *d* large, *J* = 9, C13H), 4.93 (1H, *s*, C10H). The chemical shifts,  $\delta$  p.p.m., of the C atoms in the <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>) are: 4.87 (CH<sub>2</sub>Si), 6.71 (CH<sub>3</sub>CH<sub>2</sub>Si), 14.83 (C19), 16.89 (C18), 18.65 (C16), 28.26 (C17), 38.07 (C6), 38.07 (C14), 42.41 (C15), 52.47 (C3), 56.60 (C8), 68.59 (C13), 73.61 (C5), 71.86 (C7), 74.46 (C20), 75.84 (C1), 76.48 (C10), 84.51 (C4), 84.92 (C2), 139.32 (C11), 141.12 (C12), 211.49 (C9).

#### Crystal data

C<sub>26</sub>H<sub>44</sub>O<sub>8</sub>Si

*M<sub>r</sub>* = 512.72

Monoclinic

*P*2<sub>1</sub>

*a* = 8.323 (4) Å

*b* = 10.569 (5) Å

*c* = 17.022 (10) Å

$\beta$  = 100.15 (2)°

*V* = 1473.9 (13) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.16 Mg m<sup>-3</sup>

Cu *K* $\alpha$  radiation

$\lambda$  = 1.5418 Å

Cell parameters from 25

reflections

$\theta$  = 13.4–22.2°

$\mu$  = 1.03 mm<sup>-1</sup>

*T* = 293 K

Prism

0.30 × 0.20 × 0.13 mm

Colourless

#### Data collection

Nonius CAD-4 diffractometer

$\theta/2\theta$  scans

Absorption correction:

none

2766 measured reflections

2674 independent reflections

1630 observed reflections

[*I* > 2.5 $\sigma$ (*I*)]

*R*<sub>int</sub> = 0.079

$\theta_{\text{max}}$  = 65.68°

*h* = -9 → 9

*k* = 0 → 12

*l* = 0 → 20

3 standard reflections

frequency: 166 min

intensity decay: none

#### Refinement

Refinement on *F*

*R* = 0.105

*wR* = 0.145

*S* = 1.25

1615 reflections

285 parameters

H-atom parameters not

refined

*w* = 1/[ $\sigma^2(F) + 0.163230F^2$ ]

( $\Delta/\sigma$ )<sub>max</sub> = 0.04

$\Delta\rho_{\text{max}}$  = 0.53 e Å<sup>-3</sup>

$\Delta\rho_{\text{min}}$  = -0.56 e Å<sup>-3</sup>

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol IV, Table 2.2B)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> / <i>U</i> <sub>eq</sub>
C1	0.7446 (12)	-0.0157 (13)	0.9630 (7)	0.044 (11)
C2	0.6801 (12)	-0.0723 (14)	0.8826 (6)	0.041 (11)
C3	0.6388 (11)	0.0138 (12)	0.8073 (6)	0.037 (10)

*U*<sub>iso</sub> for C21–C26; *U*<sub>eq</sub> = (1/3)Σ<sub>*i*</sub>Σ<sub>*j*</sub>*U*<sub>*ij*</sub>*a*<sub>*i*</sub><sup>\*</sup>*a*<sub>*j*</sub><sup>\*</sup> for other atoms.

C4	0.4554 (13)	-0.0163 (14)	0.7747 (7)	0.047 (11)
C5	0.3929 (13)	0.0032 (14)	0.6865 (9)	0.054 (13)
C6	0.5218 (13)	-0.0052 (17)	0.6354 (8)	0.056 (14)
C7	0.6778 (13)	0.0625 (15)	0.6691 (7)	0.053 (12)
C8	0.7618 (13)	-0.0040 (13)	0.7481 (7)	0.045 (11)
C9	0.9228 (11)	0.0660 (11)	0.7784 (6)	0.036 (9)
C10	0.9349 (11)	0.1860 (12)	0.8330 (6)	0.036 (9)
C11	0.8630 (11)	0.1721 (12)	0.9086 (6)	0.035 (9)
C12	0.7356 (12)	0.2423 (12)	0.9166 (7)	0.040 (10)
C13	0.6175 (14)	0.2139 (17)	0.9723 (8)	0.060 (14)
C14	0.6232 (14)	0.0724 (16)	0.9955 (8)	0.058 (13)
C15	0.9073 (11)	0.0581 (12)	0.9659 (6)	0.036 (9)
C16	1.0429 (13)	-0.0259 (12)	0.9452 (8)	0.051 (12)
C17	0.9750 (15)	0.1107 (14)	1.0475 (7)	0.054 (13)
C18	0.6803 (17)	0.3622 (15)	0.8631 (10)	0.068 (15)
C19	0.8045 (14)	-0.1407 (14)	0.7299 (8)	0.055 (13)
C20	0.4331 (14)	-0.1443 (14)	0.8085 (6)	0.046 (11)
O1	0.7820 (9)	-0.1210 (10)	1.0167 (5)	0.056 (9)
O2	0.5346 (11)	-0.1429 (11)	0.8863 (5)	0.065 (10)
O4	0.3753 (9)	0.0781 (12)	0.8168 (6)	0.064 (10)
O5	0.2616 (10)	-0.0798 (12)	0.6550 (5)	0.064 (10)
O7	0.7870 (9)	0.0609 (9)	0.6145 (5)	0.062 (9)
O9	1.0440 (8)	0.0325 (10)	0.7546 (5)	0.052 (8)
O10	1.0986 (9)	0.2254 (9)	0.8459 (5)	0.053 (9)
O13	0.4630 (10)	0.2522 (12)	0.9393 (6)	0.069 (11)
Si	0.8044 (6)	0.1623	0.5424 (3)	0.099 (6)
C21	1.0141 (12)	0.1475 (15)	0.5218 (8)	0.083 (5)
C22	1.0581 (17)	0.221 (2)	0.4567 (10)	0.097 (6)
C23	0.761 (2)	0.3239 (9)	0.5752 (12)	0.216 (18)
C24	0.885 (4)	0.383 (2)	0.634 (2)	0.200 (16)
C25	0.6503 (19)	0.1201 (18)	0.4530 (8)	0.23 (2)
C26	0.661 (4)	-0.0049 (26)	0.4196 (18)	0.201 (16)

Table 2. Selected geometric parameters (Å, °)

C1—C2	1.50 (2)	C9—O9	1.20 (1)
C1—C14	1.55 (2)	C10—C11	1.52 (1)
C1—C15	1.56 (1)	C10—O10	1.40 (1)
C1—O1	1.44 (2)	C11—C12	1.32 (2)
C2—C3	1.56 (2)	C11—C15	1.55 (2)
C2—O2	1.43 (2)	C12—C13	1.51 (2)
C3—C4	1.56 (1)	C12—C18	1.58 (2)
C3—C8	1.57 (2)	C13—C14	1.55 (2)
C4—C5	1.51 (2)	C13—O13	1.37 (2)
C4—C20	1.49 (2)	C15—C16	1.53 (2)
C4—O4	1.46 (2)	C15—C17	1.51 (2)
C5—C6	1.50 (2)	C20—O2	1.44 (1)
C5—O5	1.43 (2)	O7—Si	1.66 (1)
C6—C7	1.50 (2)	Si—C21	1.85 (1)
C7—C8	1.57 (2)	Si—C23	1.85 (1)
C7—O7	1.41 (1)	Si—C25	1.86 (1)
C8—C9	1.54 (2)	C21—C22	1.45 (2)
C8—C19	1.53 (2)	C23—C24	1.45 (4)
C9—C10	1.56 (2)	C25—C26	1.45 (3)
C2—C1—C14	114.4 (10)	C10—C9—O9	117.9 (9)
C2—C1—C15	113.4 (10)	C9—C10—C11	115.5 (9)
C2—C1—O1	105.9 (9)	C9—C10—O10	107.0 (8)
C14—C1—C15	107.9 (10)	C11—C10—O10	114.4 (9)
C14—C1—O1	108.7 (10)	C10—C11—C12	118.4 (10)
C15—C1—O1	106.2 (9)	C10—C11—C15	121.5 (9)
C1—C2—C3	120.5 (10)	C12—C11—C15	118.5 (10)
C1—C2—O2	109.6 (10)	C11—C12—C13	125.1 (11)
C3—C2—O2	105.9 (9)	C11—C12—C18	123.2 (11)
C2—C3—C4	103.5 (9)	C13—C12—C18	111.4 (11)
C2—C3—C8	112.5 (9)	C12—C13—C14	111.0 (11)
C4—C3—C8	117.1 (9)	C12—C13—O13	110.6 (11)
C3—C4—C5	117.9 (10)	C14—C13—O13	112.0 (12)
C3—C4—C20	103.2 (10)	C1—C14—C13	119.0 (11)
C3—C4—O4	100.9 (9)	C1—C15—C11	105.1 (9)
C5—C4—C20	117.5 (11)	C1—C15—C16	112.1 (9)
C5—C4—O4	106.7 (10)	C1—C15—C17	113.2 (10)
C20—C4—O4	109.4 (10)	C11—C15—C16	114.2 (9)
C4—C5—C6	114.3 (11)	C11—C15—C17	107.5 (9)
C4—C5—O5	113.3 (11)	C16—C15—C17	104.8 (9)
C6—C5—O5	108.9 (11)	C4—C20—O2	104.6 (10)
C5—C6—C7	114.1 (11)	C2—O2—C20	109.1 (9)
C6—C7—C8	109.8 (11)	C7—O7—Si	129.2 (8)

C6—C7—O7	110.9 (11)	O7—Si—C21	107.2 (5)
C8—C7—O7	108.6 (10)	O7—Si—C23	109.1 (6)
C3—C8—C7	104.7 (9)	O7—Si—C25	108.2 (6)
C3—C8—C9	110.9 (9)	C21—Si—C23	112.1 (7)
C3—C8—C19	116.4 (10)	C21—Si—C25	111.3 (7)
C7—C8—C9	107.6 (10)	C23—Si—C25	109.0 (8)
C7—C8—C19	109.4 (10)	Si—C21—C22	118.3 (11)
C9—C8—C19	107.6 (10)	Si—C23—C24	117.1 (16)
C8—C9—C10	123.4 (9)	Si—C25—C26	117.2 (15)
C8—C9—O9	118.5 (10)		
C15—C1—C2—C3	-59.3 (11)	C3—C2—O2—C20	21.0 (10)
C1—C2—C3—C8	109.7 (12)	C2—O2—C20—C4	-36.5 (11)
C2—C3—C8—C9	-77.7 (11)	O2—C20—C4—C3	35.8 (11)
C3—C8—C9—C10	-27.4 (9)	C20—C4—C3—C2	-22.7 (10)
C8—C9—C10—C11	53.4 (10)	C4—C3—C2—O2	2.0 (10)
C9—C10—C11—C15	49.8 (10)	C8—C3—C4—C5	-29.7 (10)
C10—C11—C15—C1	-115.7 (11)	C3—C4—C5—C6	25.1 (11)
C11—C15—C1—C2	65.1 (10)	C4—C5—C6—C7	-43.4 (12)
C14—C1—C15—C11	-62.8 (11)	C5—C6—C7—C8	65.7 (12)
C1—C15—C11—C12	49.7 (10)	C6—C7—C8—C3	-63.8 (11)
C15—C11—C12—C13	-6.3 (11)	C7—C8—C3—C4	46.9 (10)
C11—C12—C13—C14	-20.9 (12)	C20—C4—C5—O5	26.2 (11)
C12—C13—C14—C1	1.7 (11)	O4—C4—C5—O5	-96.9 (12)
C13—C14—C1—C15	39.9 (11)		

Because of the large thermal motion of the triethylsilyl group, the three ethyl chains have been refined with constraints on bond distances and angles, and have been kept isotropic, conferring to the high *R* factor and consequently a lack of precision in the results. However, all the H atoms of the hydroxyl groups were located in successive difference Fourier maps, allowing the building of the hydrogen-bond network. Their positional parameters were fixed (C—H = 1.00 Å). Isotropic temperature factors of the H atoms were assigned as  $1.10 \times U_{eq}$  of the bonded atom.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Data reduction: *NONIUS* (Riche, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *R3M* (Riche, 1983), *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *ACTACIF* (Riche, 1992).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Appendino, G., Gariboldi, P., Gabetta, B., Pace, R., Bombardelli, E. & Viterbo, D. (1992). *J. Chem. Soc. Perkin Trans. 1*, pp. 2925–2929.
- Begley, M. J., Frecknall, E. A. & Pattenden, G. (1984). *Acta Cryst. C* **40**, 1745–1747.
- Castellano, E. E. & Hodder, O. J. R. (1973). *Acta Cryst. B* **29**, 2566–2570.
- Chauvière, G., Guénard, D., Picot, F., Senilh, V. & Potier, P. (1981). *C. R. Acad. Sci. Paris Ser. II*, **294**, 501–503.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Farina, V. & Huang, S. (1992). *Tetrahedron Lett.* **33**, 3979–3982.
- Guénard, D., Guéritte-Voegelein, F. & Potier, P. (1993). *Acc. Chem. Res.* **26**, 160–167.
- Guéritte-Voegelein, F., Guénard, D., Mangatal, L., Potier, P., Guilhem, J., Cesario, M. & Pascard, C. (1990). *Acta Cryst. C* **46**, 781–784.
- Gunawardana, G. P., Premachandran, U., Burren, N. S., Whittern, D. N., Henry, R., Spanton, S. & McAlpine, J. B. (1992). *J. Nat. Prod.* **55**, 1686–1689.

- Horwitz, S. B. (1992). *TIPS*, **13**, 134–136.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Mangatal, L., Adeline, M. T., Guénard, D., Guéritte-Voegelein, F. & Potier, P. (1989). *Tetrahedron*, **42**, 4177–4190.
- Riche, C. (1983). *R3M. Représentation et Manipulation de Modèles Moléculaires*. Institut de Chimie des Substances Naturelles du CNRS, Gif sur Yvette, France.
- Riche, C. (1989). *NONIUS. Program for Data Reduction for Nonius CAD-4 Diffractometer*. Institut de Chimie des Substances Naturelles du CNRS, Gif sur Yvette, France.
- Riche, C. (1992). *ACTACIF. Logiciel de préparation des fichiers CIF*. Institut de Chimie des Substances Naturelles du CNRS, Gif sur Yvette, France.
- Rose, W. C. (1992). *Anticancer Drugs* **3**, 311–319.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- There, H. (1993). *Biofutur*, pp. 22–25.
- Wahl, A., Guéritte-Voegelein, F., Guénard, D., Le Goff, M. T. & Potier, P. (1992). *Tetrahedron*, **48**, 6965–6974.
- Wani, M. C., Taylor, H. L., Wall, M. E., Coggon, P. & McPhail, A. T. (1971). *J. Am. Chem. Soc.* **93**, 2325–2327.

*Acta Cryst.* (1995). **C51**, 468–471

## The *endo-endo-syn* Diels–Alder Diadduct of Tetrachlorodimethoxycyclopentadiene with 1,6-Dioxacyclodeca-3,8-diene

J. G. GARCIA

Lawrence Berkeley Laboratory, UC Berkeley,  
MS 55-121, Berkeley, CA 94720, USA

F. R. FRONCZEK

Department of Chemistry, Louisiana State University,  
Baton Rouge, Louisiana 70803-1804, USA

(Received 31 January 1994; accepted 25 July 1994)

## Abstract

In the title compound, *endo-endo-syn*-1,7,8,9,10,16,17,18-octachloro-19,19,20,20-tetramethoxy-4,13-dioxapentacyclo[14.2.1.1<sup>7,10</sup>.0<sup>2,15</sup>.0<sup>6,11</sup>]icosa-8,17-diene, C<sub>22</sub>H<sub>24</sub>Cl<sub>8</sub>O<sub>6</sub>, the ten-membered ring adopts a chair–chair conformation. The near-zero endocyclic torsion angles of this ring at the ring fusion bonds have values of 0.0 (6) and –0.8 (4)°. The norbornene C=C bonds have lengths 1.320 (4) and 1.311 (5) Å. The four dioxolane O—C bonds have an average length of 1.413 (2) Å.

## Comment

The crystal structures of a number of Diels–Alder-substituted cycloocta(e)nes and cyclohexa(e)nes have