

*Acta Cryst.* (1994). C50, 1820–1821

**endo-(5*R*,6*R*,11*S*,12*S*)-5,6,11,12-Tetrahydro-6-hydroxy-4,11,12-trimethoxy-9,13,13-trimethyl-5-[(trimethylsilyl)oxy]-6,10-methano-8(7*H*)-benzocyclodecenone**

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(Received 11 April 1994; accepted 21 June 1994)

**Abstract**

The X-ray analysis revealed that the title compound, C<sub>24</sub>H<sub>36</sub>O<sub>6</sub>Si, which was synthesized to obtain taxol (a congener of taxane diterpenes), has an unnatural absolute configuration.

**Comment**

The title compound, (2) in Fig. 1, was synthesized in the course of synthetic studies towards taxol (a congener of taxane diterpenes) by the Lewis-acid-promoted eight-membered ring cyclization of (1) between C9 and C10, followed by hydrolysis of the boronate and trimethylsilylation of the C2 hydroxy group. Details of the synthesis will be reported elsewhere (Nakamura, Waizumi,

Tsuruta, Horiguchi & Kuwajima, 1994). The absolute configuration originated from asymmetric dehydroxylation (Sharpless *et al.*, 1991) of the enol silyl ether (3). The structure analysis was carried out in order to determine the absolute stereochemistry.

One of the two independent molecules is shown in Fig. 2. The other has almost the same structure as that in Fig. 2, except for the torsion angle at the trimethylsilyl (TMS) group. The absolute configuration was determined from the *R* value and Flack's parameter (Flack, 1983).

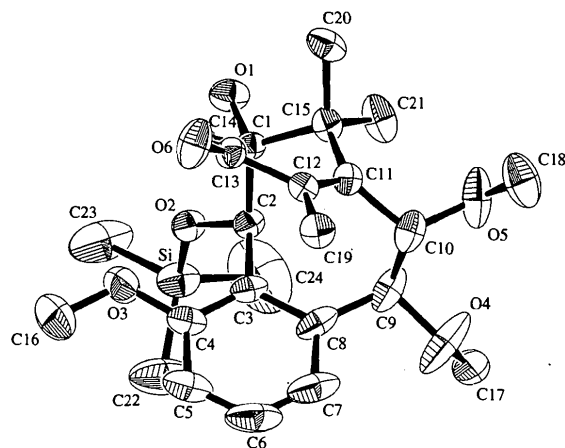


Fig. 2. View of C<sub>24</sub>H<sub>36</sub>O<sub>6</sub>Si showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level.

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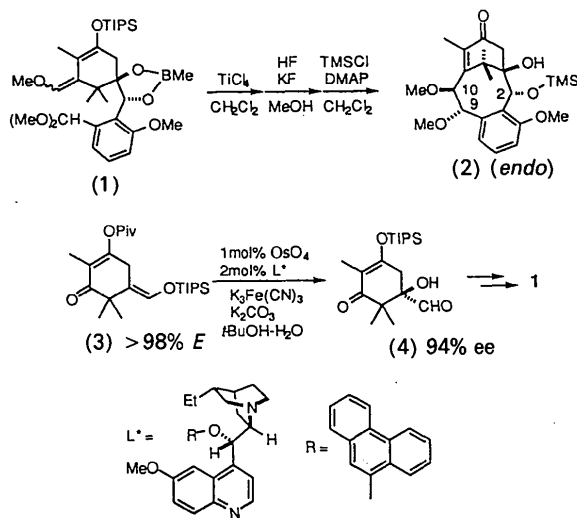


Fig. 1. Reaction scheme [TIPS = triisopropylsilyl, DMAP = 4-(dimethylamino)pyridine, TMS = trimethylsilyl].

**Experimental**

*Crystal data*

C<sub>24</sub>H<sub>36</sub>O<sub>6</sub>Si  
*M<sub>r</sub>* = 448.6  
 Orthorhombic  
 P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 15.818 (2) Å  
*b* = 30.390 (1) Å  
*c* = 10.5707 (9) Å  
*V* = 5081.3 (8) Å<sup>3</sup>  
*Z* = 8  
*D<sub>x</sub>* = 1.17 Mg m<sup>-3</sup>

Cu Kα radiation  
 λ = 1.5418 Å  
 Cell parameters from 25 reflections  
 θ = 27.5–30°  
 μ = 1.10 mm<sup>-1</sup>  
*T* = 293 K  
 Parallelepiped  
 0.50 × 0.35 × 0.30 mm  
 Colourless

*Data collection*

AFC-5 diffractometer  
 ω–2θ scans  
 Absorption correction: none  
 4526 measured reflections  
 4526 independent reflections  
 3173 observed reflections  
 [*I* > 2σ(*I*)]

θ<sub>max</sub> = 62.5°  
*h* = 0 → 18  
*k* = 0 → 34  
*l* = 0 → 12  
 3 standard reflections monitored every 100 reflections  
 intensity variation: 1.1%

*Refinement*

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.058  
*wR*(*F*<sup>2</sup>) = 0.147

(Δ/σ)<sub>max</sub> = 0.215  
 Δρ<sub>max</sub> = 0.243 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.320 e Å<sup>-3</sup>

$S = 1.131$   
 4092 reflections  
 582 parameters  
 All H-atom parameters  
 refined  
 $w = 1/[\sigma^2(F_o^2) + (0.1035P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none  
 Atomic scattering factors  
 from *International Tables*  
 for *Crystallography* (1992,  
 Vol. C, Tables 4.2.6.8 and  
 6.1.1.4)

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989).  
 Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990).  
 Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994).  
 Molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1985).  
 Software used to prepare material for publication: *SHELXL93*.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$U_{eq}$
Si	0.5975 (2)	0.23695 (6)	0.0623 (2)	0.082
O1	0.6155 (3)	0.1885 (1)	0.4087 (4)	0.071
O2	0.6256 (2)	0.2377 (1)	0.2126 (3)	0.063
O3	0.7329 (3)	0.3028 (1)	0.2451 (5)	0.085
O4	0.3709 (4)	0.3466 (2)	0.3817 (7)	0.153
O5	0.3778 (3)	0.2938 (2)	0.5976 (6)	0.113
O6	0.7477 (3)	0.3006 (2)	0.6419 (6)	0.097
C1	0.6077 (3)	0.2342 (2)	0.4397 (5)	0.051
C2	0.5809 (3)	0.2584 (2)	0.3141 (5)	0.054
C3	0.5906 (4)	0.3086 (2)	0.3092 (5)	0.059
C4	0.6639 (4)	0.3290 (2)	0.2688 (6)	0.070
C5	0.6680 (6)	0.3748 (2)	0.2516 (7)	0.101
C6	0.5968 (9)	0.3995 (3)	0.2791 (9)	0.128
C7	0.5289 (7)	0.3813 (2)	0.3236 (7)	0.107
C8	0.5187 (4)	0.3353 (2)	0.3411 (6)	0.074
C9	0.4395 (4)	0.3177 (2)	0.3991 (7)	0.084
C10	0.4511 (4)	0.3129 (2)	0.5441 (6)	0.072
C11	0.5310 (3)	0.2873 (2)	0.5708 (5)	0.052
C12	0.6009 (3)	0.3093 (2)	0.6040 (5)	0.049
C13	0.6843 (3)	0.2871 (2)	0.5885 (6)	0.057
C14	0.6910 (3)	0.2506 (2)	0.4936 (5)	0.056
C15	0.5376 (3)	0.2379 (2)	0.5413 (5)	0.057
C16	0.8085 (5)	0.3226 (3)	0.199 (1)	0.123
C17	0.3134 (4)	0.3361 (3)	0.2950 (9)	0.103
C18	0.3566 (6)	0.3081 (3)	0.7131 (9)	0.117
C19	0.6060 (4)	0.3558 (2)	0.6502 (6)	0.069
C20	0.5663 (5)	0.2139 (2)	0.6650 (6)	0.074
C21	0.4551 (4)	0.2144 (2)	0.5002 (7)	0.087
C22	0.6082 (9)	0.2898 (3)	-0.0184 (9)	0.156
C23	0.6668 (9)	0.1956 (4)	-0.0084 (9)	0.177
C24	0.4873 (9)	0.2200 (6)	0.052 (1)	0.245
Si'	0.5401 (2)	0.06524 (7)	0.2815 (2)	0.086
O1'	0.6288 (3)	0.1022 (1)	0.6106 (4)	0.066
O2'	0.5310 (3)	0.0735 (1)	0.4359 (3)	0.070
O3'	0.3724 (3)	0.0718 (2)	0.5052 (6)	0.110
O4'	0.5594 (3)	-0.0987 (1)	0.6087 (4)	0.088
O5'	0.6495 (3)	-0.0591 (1)	0.7978 (4)	0.077
O6'	0.3885 (4)	0.0787 (2)	0.8712 (6)	0.113
C1'	0.5702 (3)	0.0699 (2)	0.6536 (5)	0.051
C2'	0.5412 (4)	0.0425 (2)	0.5361 (5)	0.055
C3'	0.4642 (3)	0.0123 (2)	0.5475 (5)	0.053
C4'	0.3826 (4)	0.0270 (2)	0.5232 (7)	0.083
C5'	0.3146 (5)	-0.0016 (3)	0.5194 (9)	0.113
C6'	0.3263 (5)	-0.0452 (3)	0.5422 (9)	0.110
C7'	0.4036 (5)	-0.0610 (2)	0.5716 (7)	0.088
C8'	0.4736 (4)	-0.0329 (2)	0.5761 (5)	0.062
C9'	0.5572 (4)	-0.0518 (2)	0.6217 (5)	0.062
C10'	0.5701 (3)	-0.0429 (2)	0.7640 (5)	0.056
C11'	0.5537 (3)	0.0051 (2)	0.7918 (5)	0.047
C12'	0.4787 (3)	0.0184 (2)	0.8357 (5)	0.057
C13'	0.4518 (4)	0.0648 (2)	0.8168 (6)	0.070
C14'	0.4970 (4)	0.0924 (2)	0.7231 (6)	0.063
C15'	0.6178 (3)	0.0405 (2)	0.7497 (5)	0.053
C16'	0.2934 (5)	0.0884 (3)	0.467 (1)	0.132
C17'	0.5942 (8)	-0.1134 (2)	0.4964 (9)	0.130
C18'	0.6604 (5)	-0.0636 (3)	0.9305 (7)	0.101
C19'	0.4118 (4)	-0.0113 (2)	0.8916 (7)	0.085
C20'	0.6434 (4)	0.0681 (2)	0.8694 (6)	0.068
C21'	0.7023 (3)	0.0245 (2)	0.6948 (6)	0.068
C22'	0.6391 (6)	0.0907 (3)	0.2288 (8)	0.119
C23'	0.547 (1)	0.0066 (3)	0.241 (1)	0.196
C24'	0.4516 (9)	0.0944 (7)	0.208 (1)	0.321

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1994). **C50**, 1821–1823

## *anti,anti-N-(2,6-Dimethyl-1,3,5-trithian-1-ylidene)-4-methylbenzenesulfonamide*

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(Received 24 May 1994; accepted 21 June 1994)

## Abstract

The major product of the dimethylation of the tosylimide of 1,3,5-trithiane is shown to be the title compound,  $C_{12}H_{17}NO_2S_4$ , which has all the substituents in equatorial positions with respect to the 1,3,5-trithiane ring.

## Comment

We are currently investigating the potential of the tosylimides of 1,3-dithiolane (I), 1,3-dithiane (II) and 1,3,5-trithiane (III) as chiral acyl-anion equivalents in