

## ORGANIC COMPOUNDS

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**First X-ray Determination of an Oxaziridinium Salt: (1*S*,2*R*,3*R*,4*S*)-2,3-Dimethyl-4-phenyl-1,2,3,4-tetrahydro-1,2-epoxy-isoquinolinium Tetrafluoroborate and (1*S*,2*R*,3*R*,4*S*)-3-Methyl-4-phenyl-1,2,3,4-tetrahydro-2,3-epoxyisoquinoline**

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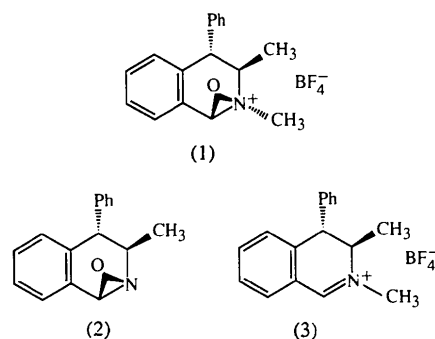
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**Abstract**

The chiral oxaziridinium salt (1),  $C_{17}H_{18}NO^+ \cdot BF_4^-$ , transfers the O atom enantioselectively to nucleophilic substrates. The configuration (1*S*,2*R*) for the O atom has been established. This is the first X-ray determination for an oxaziridinium salt. Its geometry is quite superimposable with that of the parent oxaziridine (2),  $C_{16}H_{15}NO$ . The N—O bond length of 1.468 (8) Å in the oxaziridinium salt is shortened compared with the mean bond length of 1.508 (16) Å observed in 40 X-ray structures of oxaziridine. The oxaziridine ring is perpendicular to the isoquinoline ring, the former making a dihedral angle of 97° with the mean plane of the latter in (1), and dihedral angles of 94 and 93° in the two independent molecules of (2).

**Comment**

Preliminary results have shown that the chiral oxaziridinium salt (1) transfers the O atom enantioselectively to nucleophilic substrates (Bohé, Hanquet, Lusinchi & Lusinchi, 1993). This salt has been obtained either by methylation of the corresponding chiral oxaziridine (2) or by action of a peracid on the chiral iminium salt (3) (Hanquet, Lusinchi & Milliet, 1991). This last reaction is stereospecific, allowing the use of the oxaziridinium salt as a chiral intermediate in a catalytic cycle. As the  $\alpha$  (1*R*,2*S*) or  $\beta$  (1*S*,2*R*) configuration for the O atom cannot be determined by analysis of the NMR data, the structure of (1) was established by an X-ray analysis. This study being the first X-ray determination of an oxaziridinium salt, it was of interest to compare its geometry with that of the parent oxaziridine (2).



The structures of the oxaziridinium salt (1) and the oxaziridine (2) are depicted with the same orientation in Figs. 1 and 2, respectively. In the asymmetric unit of (2) there are two independent molecules of identical conformation. These molecules are practically superimposable with the oxaziridinium ion of (1). The Cambridge Structural Database (CSD; Allen & Kennard, 1993) was searched for the oxaziridine fragment. 34 X-ray determinations were found (38 independent molecules). The two independent molecules of the present X-ray structure (2) were added to give 40 observations. The mean values of the pertinent bond distances compared with those of the oxaziridinium salt are as follows:

	C—N	N—O	C—O
Mean bond distance	1.449	1.508	1.417
e.s.d. (40 values)	0.017	0.016	0.012
Oxaziridinium (1)	1.436 (9)	1.468 (8)	1.433 (9)

The shortening of the N—O bond length in the oxaziridinium salt is noteworthy. In (1), the four atoms C1, N2, C3 and the methyl C17 are approximately in the same mean plane, which forms a dihedral angle of 91° with the plane formed by C1, N2 and O9. More precisely, the central N2 atom is 0.147 Å from the plane defined by the other three atoms C1, C3 and C17. In the CSD search, there are 24 structures where C1 of the oxaziridine ring is substituted by two C atoms:  $C^\alpha$  and  $C^\beta$  (the other structures where C1 is bonded to an H atom were excluded). In these structures the mean distance of C1 to the plane formed by N,  $C^\alpha$  and  $C^\beta$  is 0.25 (2) Å (range 0.20–0.30 Å), so, when tetra-substituted by C, N or O atoms, C1 of the oxaziridine ring appears more tetrahedral than the quaternary N atom.

The conformation adopted by the six-membered ring C1—C8a is 1,3 diplanar (Bucourt & Hainault, 1965), the phenyl and methyl substituents being in equatorial positions while the oxaziridine ring is axial. The phenyl ring at C4 forms a dihedral angle of 95° with the mean plane of the isoquinolinium ring in (1), while this angle is 92 and 98° in the two independent molecules of the oxaziridine (2). The oxaziridine ring is perpendicular

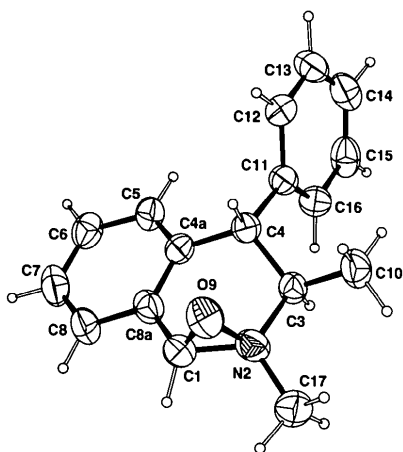


Fig. 1. Perspective view of the oxaziridinium salt (1). Displacement ellipsoids are shown at the 30% probability level.

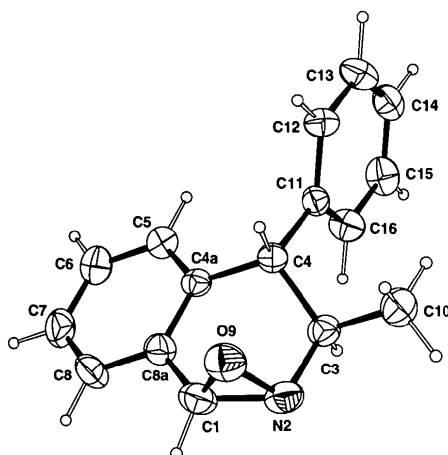


Fig. 2. Perspective view of (2). Displacement ellipsoids are shown at the 30% probability level.

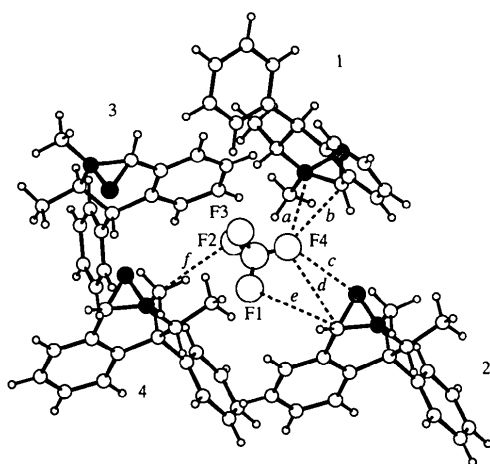


Fig. 3. Environment of the BF<sub>4</sub><sup>-</sup> anion in the crystal of (1). Pertinent distances (with e.s.d.'s):  $a = 3.22$  (1),  $b = 3.13$  (1),  $c = 2.97$  (1),  $d = 3.29$  (1),  $e = 3.39$  (1) and  $f = 3.18$  (1) Å. Symmetry operators: 1 ( $x, y, z$ ); 2 ( $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$ ); 3 ( $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$ ); 4 ( $-x, y - \frac{1}{2}, \frac{1}{2} - z$ ).

to the isoquinoline ring, the former making a dihedral angle of 97° with the mean plane of the latter in (1), and dihedral angles of 94 and 93° in (2). The CSD search revealed only one structure having an isoquinoline nucleus that forms a dihedral angle of 91° with the oxaziridine ring.

In the crystal of the oxaziridinium salt, the BF<sub>4</sub><sup>-</sup> anion is surrounded by four molecules of (1). The shortest contacts occur with the positively charged portion of (1) (Fig. 3).

## Experimental

### Compound (1)

#### Crystal data

C<sub>17</sub>H<sub>18</sub>NO<sup>+</sup>.BF<sub>4</sub><sup>-</sup>

$M_r = 339.14$

Orthorhombic

$P2_12_12_1$

$a = 8.448$  (2) Å

$b = 9.383$  (3) Å

$c = 21.140$  (7) Å

$V = 1675.7$  (8) Å<sup>3</sup>

$Z = 4$

$D_x = 1.34$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation

$\lambda = 1.5418$  Å

Cell parameters from 25

reflections

$\theta = 8.2$ – $21.4^\circ$

$\mu = 0.94$  mm<sup>-1</sup>

$T = 293$  K

Prism

$0.40 \times 0.25 \times 0.15$  mm

Colourless

#### Data collection

Nonius CAD-4 diffractometer

$\theta/2\theta$  scans

Absorption correction:

none

3241 measured reflections

2979 independent reflections

2323 observed reflections

$[I > 3.0\sigma(I)]$

$R_{\text{int}} = 0.071$

$\theta_{\text{max}} = 67.03^\circ$

$h = -10 \rightarrow 10$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 25$

3 standard reflections

frequency: 60 min

intensity decay: none

#### Refinement

Refinement on  $F$

$R = 0.097$

$wR = 0.083$

$S = 1.05$

2318 reflections

218 parameters

H-atom parameters not

refined

Unit weights applied

$(\Delta/\sigma)_{\text{max}} = 0.17$

$\Delta\rho_{\text{max}} = 0.63$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.70$  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 equivalent isotropic displacement parameters (Å<sup>2</sup>) for (1)

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

	$x$	$y$	$z$	$U_{\text{eq}}$
C1	0.4660 (8)	0.5275 (8)	0.2958 (4)	0.085 (9)
N2	0.3001 (7)	0.5608 (6)	0.2978 (3)	0.083 (7)
C3	0.2147 (7)	0.5671 (7)	0.3604 (3)	0.066 (6)
C4	0.3308 (7)	0.5960 (7)	0.4141 (3)	0.066 (6)
C4a	0.4784 (7)	0.5072 (7)	0.4115 (3)	0.068 (6)
C5	0.5571 (9)	0.4599 (7)	0.4652 (3)	0.083 (8)
C6	0.7039 (9)	0.3911 (9)	0.4594 (4)	0.093 (9)
C7	0.7666 (9)	0.3655 (9)	0.4019 (5)	0.101 (10)

C8	0.6870 (9)	0.4073 (8)	0.3480 (4)	0.093 (9)
C8a	0.5457 (7)	0.4797 (7)	0.3533 (3)	0.072 (7)
O9	0.4189 (6)	0.6737 (6)	0.2902 (2)	0.099 (6)
C10	0.0871 (8)	0.6752 (8)	0.3542 (4)	0.092 (9)
C11	0.2439 (7)	0.5874 (7)	0.4778 (3)	0.067 (6)
C12	0.2606 (8)	0.6970 (8)	0.5209 (3)	0.086 (8)
C13	0.1877 (11)	0.6876 (10)	0.5796 (4)	0.100 (10)
C14	0.0909 (9)	0.5741 (11)	0.5934 (3)	0.095 (10)
C15	0.0708 (9)	0.4652 (10)	0.5514 (4)	0.095 (10)
C16	0.1513 (9)	0.4712 (8)	0.4938 (3)	0.082 (8)
C17	0.2039 (9)	0.5427 (10)	0.2406 (3)	0.105 (11)
B	0.2131 (11)	0.1512 (14)	0.3180 (6)	0.097 (13)
F1	0.2195 (13)	0.0264 (7)	0.3042 (5)	0.274 (18)
F2	0.0920 (12)	0.2286 (10)	0.3023 (6)	0.29 (2)
F3	0.179 (2)	0.1713 (14)	0.3733 (5)	0.39 (3)
F4	0.3282 (10)	0.2184 (10)	0.3056 (8)	0.34 (3)

$\theta/2\theta$  scans  $h = -10 \rightarrow 10$   
 Absorption correction:  $k = 0 \rightarrow 17$   
 none  $l = 0 \rightarrow 11$   
 2522 measured reflections 3 standard reflections  
 2377 independent reflections frequency: 166 min  
 1399 observed reflections intensity decay: none  
 $[I > 2.5\sigma(I)]$

**Refinement**

Refinement on  $F$   $(\Delta/\sigma)_{\max} = 0.02$   
 $R = 0.045$   $\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$   
 $wR = 0.060$   $\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$   
 $S = 0.81$  Extinction correction: none  
 1396 reflections Atomic scattering factors  
 163 parameters from *International Tables*  
 H-atom parameters not for *X-ray Crystallography*  
 refined (1974, Vol. IV, Table  
 $w = 1/[\sigma^2(F) + 0.0034F^2]$  2.2B)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (1)

C1—N2	1.436 (9)	C4a—C5	1.388 (9)
C1—C8a	1.461 (10)	C4a—C8a	1.380 (9)
C1—O9	1.433 (9)	C5—C6	1.403 (11)
N2—C3	1.509 (8)	C6—C7	1.349 (13)
N2—O9	1.468 (8)	C7—C8	1.379 (12)
N2—C17	1.466 (9)	C8—C8a	1.378 (10)
C3—C4	1.526 (8)	B—F1	1.208 (15)
C3—C10	1.486 (10)	B—F2	1.298 (15)
C4—C4a	1.500 (9)	B—F3	1.218 (16)
C4—C11	1.535 (8)	B—F4	1.188 (14)
N2—C1—C8a	119.5 (6)	C5—C4a—C8a	118.1 (6)
N2—C1—O9	61.5 (4)	C4a—C5—C6	120.0 (7)
C8a—C1—O9	119.3 (6)	C5—C6—C7	120.5 (7)
C1—N2—C3	120.0 (5)	C6—C7—C8	120.1 (8)
C1—N2—O9	59.1 (4)	C7—C8—C8a	119.7 (7)
C1—N2—C17	119.5 (6)	C1—C8a—C4a	119.7 (6)
C3—N2—O9	113.2 (5)	C1—C8a—C8	118.9 (6)
C3—N2—C17	117.5 (5)	C4a—C8a—C8	121.4 (6)
O9—N2—C17	111.9 (5)	C1—O9—N2	59.3 (4)
N2—C3—C4	110.7 (5)	C4—C11—C12	119.4 (6)
N2—C3—C10	107.3 (5)	C4—C11—C16	121.7 (6)
C4—C3—C10	114.2 (5)	F1—B—F2	121.1 (11)
C3—C4—C4a	114.1 (5)	F1—B—F3	113.1 (12)
C3—C4—C11	109.7 (5)	F1—B—F4	115.1 (12)
C4a—C4—C11	113.6 (5)	F2—B—F3	88.4 (11)
C4—C4a—C5	123.1 (6)	F2—B—F4	107.0 (11)
C4—C4a—C8a	118.6 (5)	F3—B—F4	108.8 (13)
C8a—C1—N2—C3	8.8 (6)	C4—C3—N2—O9	-43.3 (5)
C1—N2—C3—C4	23.4 (5)	C10—C3—N2—O9	82.0 (6)
N2—C3—C4—C4a	-45.8 (5)	C1—N2—C3—C10	148.6 (7)
C3—C4—C4a—C8a	38.4 (6)	C3—C4—C11—C12	-129.7 (7)
C4—C4a—C8a—C1	-4.8 (6)	C4a—C4—C11—C16	-77.5 (6)
C4a—C8a—C1—N2	-19.6 (6)	C8a—C1—N2—C17	-151.2 (8)
C4a—C8a—C1—O9	52.4 (7)	C4—C3—N2—C17	-176.2 (7)
C8—C8a—C1—O9	-127.9 (8)		

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (2)

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

	x	y	z	$U_{eq}$
C1A	0.4714 (6)	0.6709 (4)	0.1106 (6)	0.067 (7)
N2A	0.3164 (5)	0.6774 (3)	0.1275 (5)	0.064 (5)
C3A	0.2353 (5)	0.5913 (4)	0.1190 (5)	0.052 (5)
C4A	0.3335 (5)	0.5075 (4)	0.1535 (4)	0.046 (5)
C4aA	0.4652 (5)	0.5084 (4)	0.0764 (4)	0.044 (5)
C5A	0.5242 (6)	0.4333 (4)	0.0259 (6)	0.061 (6)
C6A	0.6494 (6)	0.4381 (5)	-0.0369 (7)	0.077 (7)
C7A	0.7175 (6)	0.5174 (6)	-0.0525 (7)	0.082 (8)
C8A	0.6583 (7)	0.5938 (5)	-0.0039 (7)	0.076 (7)
C8aA	0.5346 (6)	0.5897 (4)	0.0615 (5)	0.057 (6)
O9A	0.4445 (4)	0.6745 (3)	0.2533 (4)	0.070 (4)
C10A	0.1203 (7)	0.5987 (4)	0.2151 (7)	0.072 (7)
C11A	0.2404 (5)	0.4231 (3)	0.1276 (5)	0.045 (5)
C12A	0.2467 (6)	0.3606 (4)	0.2338 (5)	0.055 (5)
C13A	0.1644 (7)	0.2831 (4)	0.2129 (6)	0.072 (7)
C14A	0.0742 (6)	0.2669 (4)	0.0848 (7)	0.069 (7)
C15A	0.0660 (6)	0.3285 (5)	-0.0225 (6)	0.070 (7)
C16A	0.1499 (6)	0.4056 (4)	-0.0024 (5)	0.063 (6)
C1B	-0.0686 (6)	1.0610 (4)	0.3814 (7)	0.066 (6)
N2B	0.0785 (5)	1.0814 (3)	0.4530 (6)	0.071 (6)
C3B	0.1703 (5)	1.0051 (4)	0.5157 (6)	0.058 (5)
C4B	0.1265 (5)	0.9136 (3)	0.4490 (5)	0.045 (5)
C4aB	-0.0393 (5)	0.8986 (3)	0.4198 (5)	0.043 (5)
C5B	-0.1024 (6)	0.8150 (4)	0.4180 (5)	0.056 (6)
C6B	-0.2556 (7)	0.8036 (5)	0.3824 (6)	0.073 (7)
C7B	-0.3469 (6)	0.8771 (5)	0.3527 (7)	0.077 (8)
C8B	-0.2857 (6)	0.9599 (5)	0.3534 (7)	0.071 (7)
C8aB	-0.1334 (5)	0.9710 (4)	0.3870 (5)	0.054 (5)
O9B	0.0403 (4)	1.0725 (3)	0.2929 (4)	0.075 (5)
C10B	0.3326 (6)	1.0274 (5)	0.5100 (8)	0.087 (8)
C11B	0.2130 (5)	0.8421 (3)	0.5409 (5)	0.045 (5)
C12B	0.3154 (5)	0.7894 (4)	0.4886 (6)	0.057 (6)
C13B	0.4018 (6)	0.7282 (4)	0.5738 (7)	0.072 (7)
C14B	0.3817 (6)	0.7155 (4)	0.7135 (7)	0.069 (7)
C15B	0.2769 (6)	0.7662 (4)	0.7645 (5)	0.069 (7)
C16B	0.1929 (5)	0.8287 (4)	0.6805 (5)	0.057 (6)

**Compound (2)**

*Crystal data*

$C_{16}H_{15}NO$   
 $M_r = 237.30$   
 Monoclinic  
 $P2_1$   
 $a = 9.130 (7) \text{\AA}$   
 $b = 15.082 (12) \text{\AA}$   
 $c = 9.506 (7) \text{\AA}$   
 $\beta = 100.36 (3)^\circ$   
 $V = 1287.6 (17) \text{\AA}^3$   
 $Z = 4$   
 $D_x = 1.22 \text{ Mg m}^{-3}$

**Cu K $\alpha$  radiation**

$\lambda = 1.5418 \text{\AA}$   
 Cell parameters from 25 reflections  
 $\theta = 6.9\text{--}16.3^\circ$   
 $\mu = 0.56 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Prism  
 $0.50 \times 0.07 \times 0.05 \text{ mm}$   
 Colourless

*Data collection*

Nonius CAD-4 diffractometer

$R_{int} = 0.031$   
 $\theta_{\max} = 66.93^\circ$

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (2)

C1A—N2A	1.456 (7)	C1B—N2B	1.426 (7)
C1A—C8aA	1.466 (8)	C1B—C8aB	1.486 (8)
C1A—O9A	1.423 (7)	C1B—O9B	1.424 (7)
N2A—C3A	1.490 (7)	N2B—C3B	1.485 (7)
N2A—O9A	1.515 (6)	N2B—O9B	1.505 (6)
C3A—C4A	1.550 (7)	C3B—C4B	1.540 (7)

C3A—C10A	1.514 (8)	C3B—C10B	1.530 (8)
C4A—C4aA	1.517 (6)	C4B—C4aB	1.506 (6)
C4A—C11A	1.525 (7)	C4B—C11B	1.517 (7)
C4aA—C5A	1.377 (8)	C4aB—C5B	1.386 (7)
C4aA—C8aA	1.398 (8)	C4aB—C8aB	1.390 (7)
C5A—C6A	1.383 (8)	C5B—C6B	1.389 (8)
C6A—C7A	1.368 (11)	C6B—C7B	1.386 (10)
C7A—C8A	1.386 (11)	C7B—C8B	1.368 (10)
C8A—C8aA	1.384 (8)	C8B—C8aB	1.379 (7)
N2A—C1A—C8aA	122.6 (5)	N2B—C1B—C8aB	121.8 (5)
N2A—C1A—O9A	63.5 (3)	N2B—C1B—O9B	63.8 (4)
C8aA—C1A—O9A	118.7 (5)	C8aB—C1B—O9B	117.2 (5)
C1A—N2A—C3A	114.7 (4)	C1B—N2B—C3B	116.0 (5)
C1A—N2A—O9A	57.2 (3)	C1B—N2B—O9B	58.1 (3)
C3A—N2A—O9A	108.8 (4)	C3B—N2B—O9B	110.3 (4)
N2A—C3A—C4A	115.9 (4)	N2B—C3B—C4B	116.3 (4)
N2A—C3A—C10A	107.2 (4)	N2B—C3B—C10B	107.2 (5)
C4A—C3A—C10A	111.7 (4)	C4B—C3B—C10B	111.4 (5)
C3A—C4A—C4aA	111.6 (4)	C3B—C4B—C4aB	112.9 (4)
C3A—C4A—C11A	111.2 (4)	C3B—C4B—C11B	109.2 (4)
C4aA—C4A—C11A	113.4 (4)	C4aB—C4B—C11B	113.7 (4)
C4A—C4aA—C5A	123.7 (4)	C4B—C4aB—C5B	122.7 (4)
C4A—C4aA—C8aA	118.1 (4)	C4B—C4aB—C8aB	119.0 (4)
C5A—C4aA—C8aA	118.2 (5)	C5B—C4aB—C8aB	118.3 (4)
C4aA—C5A—C6A	120.8 (5)	C4aB—C5B—C6B	120.9 (5)
C5A—C6A—C7A	121.3 (6)	C5B—C6B—C7B	119.5 (6)
C6A—C7A—C8A	118.6 (6)	C6B—C7B—C8B	120.0 (6)
C7A—C8A—C8aA	120.7 (6)	C7B—C8B—C8aB	120.4 (6)
C1A—C8aA—C4aA	119.2 (5)	C1B—C8aB—C4aB	119.4 (5)
C1A—C8aA—C8A	120.3 (5)	C1B—C8aB—C8B	119.6 (5)
C4aA—C8aA—C8A	120.4 (5)	C4aB—C8aB—C8B	120.9 (5)
C1A—O9A—N2A	59.3 (3)	C1B—O9B—N2B	58.2 (3)
C4A—C11A—C12A	120.3 (4)	C4B—C11B—C12B	121.1 (4)
C4A—C11A—C16A	122.0 (4)	C4B—C11B—C16B	120.6 (4)
C8aA—C1A—N2A—C3A	11.4 (5)		
C1A—N2A—C3A—C4A	23.2 (4)		
N2A—C3A—C4A—C4aA	-46.8 (4)		
C3A—C4A—C4aA—C8aA	37.1 (4)		
C4A—C4aA—C8aA—C1A	-4.6 (5)		
C4aA—C8aA—C1A—N2A	-22.0 (5)		
C4aA—C8aA—C1A—O9A	53.3 (5)		
C8A—C8aA—C1A—O9A	-129.2 (7)		
C4A—C3A—N2A—O9A	-38.5 (4)		
C10A—C3A—N2A—O9A	86.9 (5)		
C1A—N2A—C3A—C10A	148.7 (6)		
C3A—C4A—C11A—C12A	-126.4 (5)		
C4aA—C4A—C11A—C16A	-71.9 (5)		
C8aB—C1B—N2B—C3B	8.4 (5)		
C1B—N2B—C3B—C4B	23.7 (5)		
N2B—C3B—C4B—C4aB	-42.8 (4)		
C3B—C4B—C4aB—C8aB	30.5 (4)		
C4B—C4aB—C8aB—C1B	0.1 (5)		
C4aB—C8aB—C1B—N2B	-21.6 (5)		
C4aB—C8aB—C1B—O9B	53.0 (5)		
C8B—C8aB—C1B—O9B	-124.1 (7)		
C4B—C3B—N2B—O9B	-39.8 (4)		
C10B—C3B—N2B—O9B	85.6 (5)		
C1B—N2B—C3B—C10B	149.0 (6)		
C3B—C4B—C11B—C12B	-115.6 (5)		
C4aB—C4B—C11B—C16B	-63.5 (5)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1171). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## A 10-Deacetylbaecatin III Derivative: 11,12-Dihydro-7-triethylsilyl-10-deacetyl- baecatin III

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## Abstract

The title compound (systematic name: 2a,3,4,4a,5,6,7-, 8,9,10,11,12,12a,12b-tetradecahydro-6,9,11-trihydroxy-4a,8,13,13-tetramethyl-4-triethylsiloxy-7,11-methano-1H-cyclodeca[3,4]benz[1,2b]oxete-12,12b-diyl 12b-acetate 12-benzoate), C<sub>35</sub>H<sub>52</sub>O<sub>10</sub>Si, was obtained from 10-deacetylbaecatin III by the reduction of the C11=C12 double bond of the 13-keto derivative. The saturation of the C11=C12 bond results in a large change in the orientation of the C13—OH hydroxy group. The

For (1), very high thermal motion was observed for the BF<sub>4</sub> anion. Isotropic displacement parameters of H atoms were set equal to 1.30 × U<sub>eq</sub> of the bonded atom. For (2), two large blocks were used in the refinement; thus, the origin of the space group was fixed. Isotropic displacement parameters of H atoms were set equal to 1.10 × U<sub>eq</sub> of the bonded atom.

For both compounds, data collection: *CAD-4 Software* (Enraf-Nonius, 1989); data reduction: *NONIUS* (Riche, 1989); program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELX76* (Sheldrick, 1976); molecular graphics: *R3M* (Riche, 1983), *ORTEP* (Johnson, 1965); software used to prepare material for publication: *ACTACIF* (Riche, 1992).