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(1α,4aβ,9aβ)-4a-Acetoxy-9a-chloro-1-methoxy-3-trimethylsiloxy-1,4,4a,9,9a,10-hexahydroanthracene-9,10-dione

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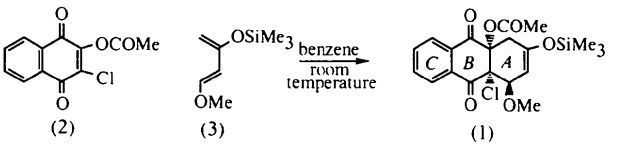
Abstract

The structure of the title compound, (1α,4aβ,9aβ)-9a-chloro-1-methoxy-9,10-dioxo-3-trimethylsiloxy-1,4,4a,9,-9a,10-hexahydro-4a-anthracenyl acetate, $C_{20}H_{23}ClO_6Si$, was determined in order to ascertain the regiochemistry of the Diels–Alder reaction between acetoxychloronaphthoquinone and a 1,3-dioxybutadiene.

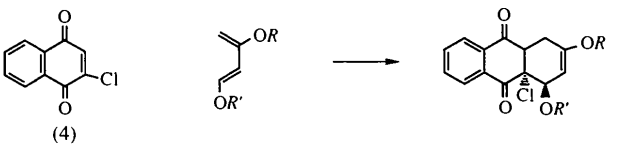
Comment

The title compound, (1), was the sole product obtained from the Diels–Alder cycloaddition reaction between acetoxychloronaphthoquinone (2) (Fries & Ochwat, 1923) and 1-methoxy-3-(trimethylsilyloxy)butadiene, (3) (Scheme I). The present structure analysis establishes that the regiochemistry of cycloaddition is such that the nucleophilic methylene terminus of the diene has attacked *ipso* to the acetoxy group; this is the same orientation with respect to the chloro group as for additions involving monochloro quinones, *e.g.* compound (4) (see Scheme II) (Cameron, Feutrill & Keep, 1989).



(2) (3) (1)

Scheme I



(4)

Scheme II

Compound (1) is a linear tricycle with a *cis* junction between rings *A* and *B*. Both rings *A* and *B* exist in slightly distorted half-chair conformations with approximate local axes of symmetry bisecting the C1—C6 and C3—C4 bonds for ring *A*, and the C1—C6 and C8—C13 bonds for ring *B*. The methoxy substituent occupies a pseudo-axial position [O6—C5—C4—C3 −98.7 (5)°] and is antiperiplanar to the chloro substituent [O6—C5—C6—Cl −163.6 (3)°]. The chloro substituent is pseudo-equatorial with respect to ring *B* and pseudo-axial with respect to ring *A*, while the acetoxy substituent is pseudo-axial with respect to ring *B* and pseudo-equatorial with respect to ring *A*.

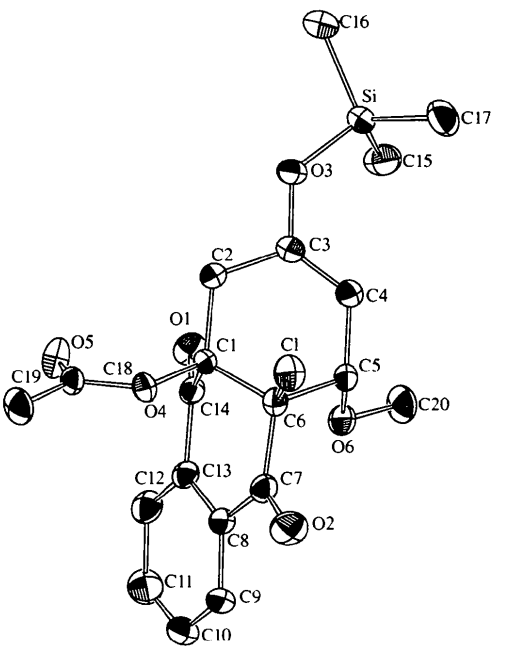


Fig. 1. *ZORTEP* (Zsolnai, 1994) diagram of (1). Displacement ellipsoids are plotted at the 30% probability level.

Experimental

Colourless rods of (1) (m.p. 397–402 K) were grown from petrol.

Crystal data

$C_{20}H_{23}ClO_6Si$
M_r = 422.94
Monoclinic
$P2_1$
a = 8.137 (2) Å
b = 12.363 (2) Å
c = 10.537 (2) Å
β = 98.77 (2)°
V = 1047.6 (4) Å^3
Z = 2
D_x = 1.341 Mg m^{-3}
D_m not measured

Cu $K\alpha$ (Ni-filtered) radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 25–30°
μ = 2.453 mm^{-1}
T = 293 (2) K
Rod
0.40 × 0.20 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*SHELX*76); Sheldrick, 1976)
T_{min} = 0.50, T_{max} = 0.72
1957 measured reflections
1832 independent reflections

1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.0511
θ_{max} = 63.96°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 14$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 160 min
intensity decay: <2%

Refinement

Refinement on F^2
$R(F)$ = 0.0397
$wR(F^2)$ = 0.1046
S = 1.235
1832 reflections
346 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.4093P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 1.010

$\Delta\rho_{max}$ = 0.238 e Å^{-3}
$\Delta\rho_{min}$ = −0.354 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0073 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.01 (3)

The title structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990). Refinement was performed with *SHELXL*93 (Sheldrick, 1993) using anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for the H atoms. All calculations were carried out on a VAXstation 4000VLC computer system.

Data collection: *CAD-4/VAX* (Enraf–Nonius, 1989). Cell refinement: *CAD-4/VAX*. Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: OA1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(1*R*,1*R*′)-2-*exo*-Mercapto-2′-thioxo-3-*exo*,3′-*exo*-bibornane, 2-Dehydro-2,2′-*exo*-epidithio-3,3′-bibornane and 2-*endo*,2′-*exo*-Epidithio-3,3′-bibornanylidene. Potential Antiviral Agents

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Abstract

The compounds 2,2′-*exo*-epidithio-1,1′,7,7,7′,7′-hexamethyl-3,3′-bibicyclo[2.2.1]hept-2-ene, $C_{20}H_{30}S_2$, (2), and 2-*endo*,2′-*exo*-epidithio-1,1′,7,7,7′,7′-hexamethyl-3,3′-bibicyclo[2.2.1]heptanylidene, $C_{20}H_{30}S_2$, (3), were prepared as potential antiviral agents and their structures were determined by X-ray diffraction. It has been shown previously that there is a relationship between the strain energy of a C—S—S—C group, as measured by its planarity and S—S bond length, and the antiviral activity of the compound containing this group. In