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2-Methyl-2-(trimethylsilyloxy)-3,4-dihydro-2*H*,5*H*-pyrano[3,2-*c*][1]benzopyran-5-one

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Key indicators

Single-crystal X-ray study
T = 288 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.152
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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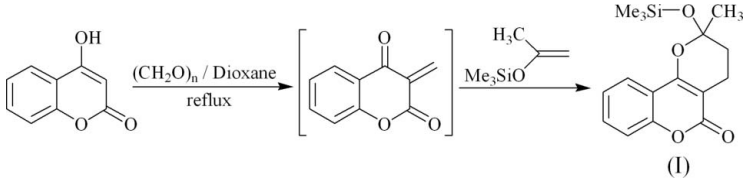
In the title compound, $C_{16}H_{20}O_4Si$, the coumarin moiety is essentially planar and the pyran ring adopts a half-chair conformation.

Comment

o-Quinone methides constitute a class of highly reactive intermediates and their cycloaddition chemistry has attracted considerable interest (Nair *et al.*, 2001). In our studies of the cycloaddition reactions of *o*-quinone methides derived from 4-hydroxycoumarin, we have found that the cycloaddition reaction between coumarin quinone methide and an excess amount of trimethyl[(1-methylethenyl)oxy]silane afforded the title compound, (I), as one of the products. As part of this study, we have undertaken the X-ray crystallographic analysis of (I) in order to elucidate the conformation and configuration of this cycloadduct product.



(I)

The bond lengths and angles in (I) (Fig. 1 and Table 1) are in good agreement with those found in related compounds (Savell *et al.*, 1989). The coumarin moiety is essentially planar, with atom O3 deviating from the mean plane by 0.152 (4) Å. The dihedral angle between the heterocyclic plane (C8/C9/O4/

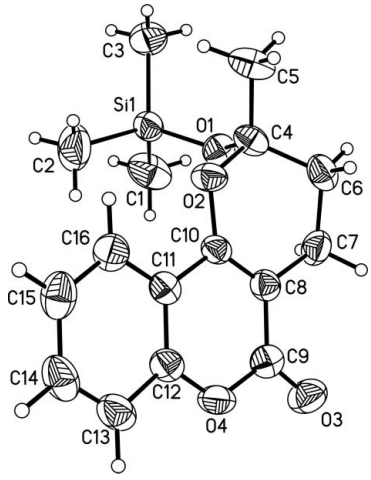


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

C12/C11/C10) and the fused benzene ring is 3.2 (3)°. The pyran ring (C4/C6–C8/C10/O2) adopts a half-chair conformation, with atoms C4 and C6 deviating from the C7/C8/C10/O2 plane by 0.237 (4) and −0.487 (4) Å, respectively.

In the crystal structure, the molecular packing is stabilized by intermolecular C—H···O interactions, as detailed in Table 2.

Experimental

Under argon, a mixture of 4-hydroxycoumarin, 3 equivalents of trimethyl[(1-methylethenyl)oxy]silane and an excess amount of paraformaldehyde was suspended in dry dioxane and refluxed for about 14 h. Compound (I) was isolated by column chromatography of the reaction mixture on silica gel after evaporation of the solvent, in 76% yield. Single crystals of (I) were obtained by slow evaporation of a petroleum ether–ethyl acetate (3:1 *v*/*v*) solution of (I).

Crystal data

$C_{16}H_{20}O_4Si$
M_r = 304.41
Monoclinic, $P2_1/c$
a = 11.960 (2) Å
b = 10.456 (2) Å
c = 13.039 (3) Å
β = 105.09 (3)°
V = 1574.4 (6) Å^3
Z = 4
D_x = 1.284 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.16 mm^{-1}
T = 288 (2) K
Block, colourless
0.40 × 0.31 × 0.28 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
T_{min} = 0.922, T_{max} = 0.956
3075 measured reflections
2930 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.5°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.152
S = 1.00
2930 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 1.48P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.391 (4)	C4—C6	1.507 (5)
O2—C4	1.464 (4)	C6—C7	1.519 (5)
O3—C9	1.215 (4)		
C4—O1—Si1	134.8 (2)	O1—C4—C5	113.0 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5*C*···O3^{i}	0.96	2.47	3.426 (5)	171
C13—H13···O3^{ii}	0.93	2.56	3.492 (4)	175

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+3, y-\frac{1}{2}, -z+\frac{3}{2}$.

H atoms were included in the riding-model approximation, with C—H distances of 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, and with $U_{iso}(H) = 1.2U_{eq}$(aromatic and methylene C) or $1.5U_{eq}$(methyl C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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