organic papers

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

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In the title compound, $C_{25}H_{30}O_4Si$, the coumarin system is essentially planar and the pyran ring adopts an envelope conformation. There are intramolecular C—H···O interactions and intermolecular π-π stacking interactions in the crystal structure.

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 recent research work on the cycloaddition reactions of *o*-quinone methides derived from 4-hydroxycoumarin, we have prepared the title compound, (I), which is obtained from the cycloaddition reaction between coumarin quinone methide and an excess amount of trimethyl[(1-phenylethenyl)oxy]silane. As part of this study, we have undertaken the X-ray crystallographic analysis of (I) in order to elucidate the conformation of this cycloadduct product.
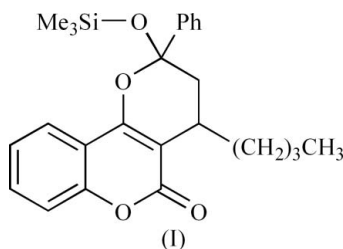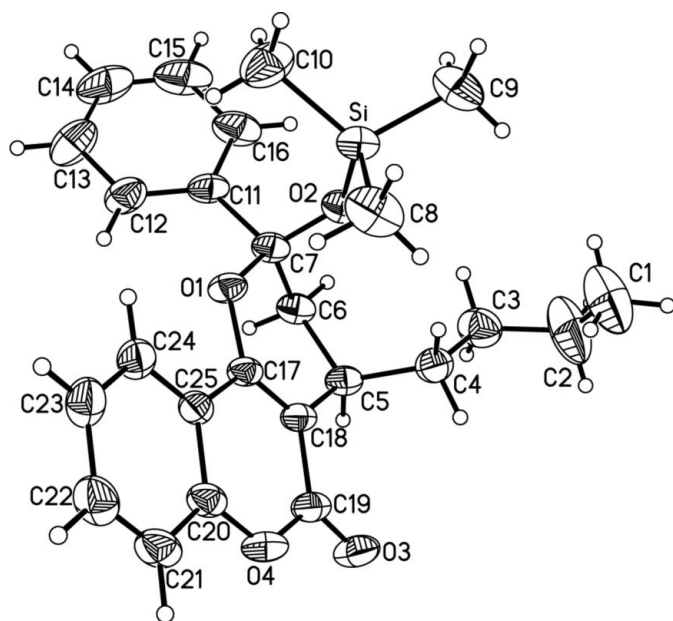

The bond lengths and angles in (I) (Fig. 1 and Table 1) are in good agreement with those found in the related compound 2-methyl-2-(trimethylsilyloxy)-3,4-dihydro-2 H,5H-pyrano-[3,2-c][1]benzopyran-5-one (Peng *et al.*, 2005). The coumarin system is essentially planar, with the atom O3 deviating from the mean plane by 0.101 (4) Å. The dihedral angle between the coumarin system and the C11–C16 benzene ring is 46.2 (3)°. The O1/C5–C7/C17/C18 pyran ring adopts an envelope conformation, with atom C6 deviating from the C5/C7/O1/C17/C18 plane by 0.598 (4) Å.

In the crystal structure, the molecular packing is stabilized by intramolecular C—H···O interactions (Table 2) and intermolecular π–π stacking interactions (Table 3).

Experimental

Under argon, a mixture of 4-hydroxycoumarin, 3 equivalents of trimethyl[(1-phenylethenyl)oxy]silane and an excess amount of *n*-pentanal were suspended in dry dioxane and refluxed for about 20 h. Compound (I) was isolated by column chromatography of the reac-

Table 1
Selected geometric parameters (Å, °).

O2—C7	1.387 (4)	O3—C19	1.205 (4)
O2—Si	1.664 (2)	C7—C11	1.532 (4)
C17—O1—C7	119.8 (2)	C20—O4—C19	122.3 (2)
C7—O2—Si	131.0 (2)	C7—C6—C5	114.0 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O2	0.97	2.52	3.064 (4)	115
C12—H12···O1	0.93	2.36	2.696 (4)	101

Table 3
π-π interactions (Å, °)..

CgI	CgJ	Symmetry code	Cg···Cg	Dihedral angle	Interplanar distance	Offset
A	A	$-x, 1-y, 1-z$	3.763 (2)	0.0 (2)	3.447 (3)	1.51

CgA denotes the centroid of the aromatic ring C20–C25. The offset is defined as the distance between CgI and the perpendicular projection of CgJ on ring I.

The H atoms were included in the riding-model approximation, with C—H distances of 0.93, 0.96, 0.97 and 0.98 Å for aromatic, methyl, methylene and methine H atoms, respectively, and with $U_{iso}(H) = 1.2U_{eq}(C)$ 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).

References

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Figure 1
The structure of (I) showing the atom numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

tion mixture on silica gel after evaporation of the solvent, in 65% yield. Single crystals of (I) were obtained by slow evaporation of a petroleum ether–acetone (1:3 *v/v*) solution of (I).

Crystal data

$C_{25}H_{30}O_4Si$	$Z = 4$
$M_r = 422.58$	$D_x = 1.174$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 12.138$ (2) Å	$\mu = 0.13$ mm^{-1}
$b = 12.874$ (3) Å	$T = 293$ (2) K
$c = 15.300$ (3) Å	Block, colourless
$\beta = 90.20$ (3)°	$0.40 \times 0.31 \times 0.28$ mm
$V = 2390.8$ (8) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	4208 independent reflections
$\omega/2\theta$ scans	2484 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)	$R_{int} = 0.078$
	$\theta_{max} = 25.0°$
	3 standard reflections
$T_{min} = 0.939$, $T_{max} = 0.966$	every 200 reflections
4420 measured reflections	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 1.79P]$
$R[F^2 > 2\sigma(F^2)] = 0.060$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.154$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.00$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
4208 reflections	$\Delta\rho_{min} = -0.35$ e Å^{-3}
266 parameters	Extinction correction: *SHELXL97*
H-atom parameters constrained	Extinction coefficient: 0.0192 (13)