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3-*tert*-Butyl-4-methyl-2-phenyl-3-(trimethylsilyloxy)oxetane and 2-(2-Benzyl-oxyphenyl)-3-*tert*-butyl-3-(trimethylsilyloxy)oxetane

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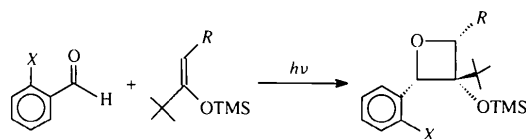
Abstract

In the structures of both of the title compounds, C₁₇H₂₈O₂Si, (I), and C₂₃H₃₂O₃Si, (II), the bulkiest substituents are *trans* with respect to one another, *i.e.* the phenyl group (or substituted phenyl) is *trans* with respect to the *tert*-butyl group. In compound (I), the methyl group at ring-position 4 is in a *trans* configuration relative to the *tert*-butyl group. The oxetane rings in both structures, especially (II), are non-planar. Both structures contain long C—O single bonds in the four-membered ring (1.44–1.46 Å), which is typical of oxetane structures.

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Comment

The Paternò–Büchi reaction represents the most convenient and straightforward access to functionalized oxetanes. It has been shown recently that the photocycloaddition of silyl enol ethers to various aldehydes proceeds with excellent regio- and diastereoselectivity (Bach, 1995a). The products formed are 3-silyloxy-substituted oxetanes, which represent versatile vehicles for further synthetic transformations (Bach, 1995b; Bach, Jödicke, Kather & Hecht, 1995). The relative configuration of these heterocycles is presumably controlled by a two-step pathway *via* an intermediate triplet 1,4-biradical (Freilich & Peters, 1981). Details of the synthetic work have been published elsewhere (Bach, 1995a). The crystals used in this work were obtained from an ether/pentane (1:1) solution. The photochemical reaction sequence is depicted in general terms below. The isolated yields were 82% for oxetane (I) and 56% for oxetane (II) after chromatographic purification.



- (I) X = H; R = Me (82%)
 (II) X = OCH₂Ph; R = H (56%)

The structural studies of compounds (I) and (II) were undertaken in order to establish the relative configurations of the oxetane rings. The X-ray crystal structures [shown in Figs. 1 and 2 for (I) and (II), respectively] confirm the expected *trans* relationship of the bulkiest substituents. This geometry was also strongly suggested by NMR measurements (difference Nuclear Overhauser Effect) performed earlier (Bach, 1995a). The oxetane rings are not planar and the puckering angles (defined as the dihedral angle between planes C—O—C and C—C—C) are 8.3 (3) and 20.7 (2)° for compounds (I) and (II), respectively. The non-planarity can also be seen from the ring torsion angles, which are clearly different from zero [O1—C2—C3—C4 5.8 (2) in (I) and –14.2 (2)° in (II)]. Previous single-crystal X-ray investigations on substituted oxetane derivatives (Seitz *et al.*, 1994; Khan, Morris, Smith & Walsh, 1991; Morris, Smith & Walsh, 1987; George & Gilardi, 1986; Hospital, Leroy, Bats & Moulines, 1978; Holan, Kowala & Wunderlich, 1973) show that the puckering angles can vary from 0 to 22.9 (2)°. Furthermore, the crystal structure of unsubstituted oxetane has been determined at temperatures of 90 and 140 K (Luger & Buschmann, 1984); the ring exhibits a non-planar structure with puckering angles of 10.7 (1)° at 90 K and 8.7 (2)° at 140 K.

The C—O bond distances [1.442 (3)–1.455 (2) Å] are larger than the average value for saturated heterocyclic C—O single bonds [1.426 (5) Å; Sutton, 1965], but this

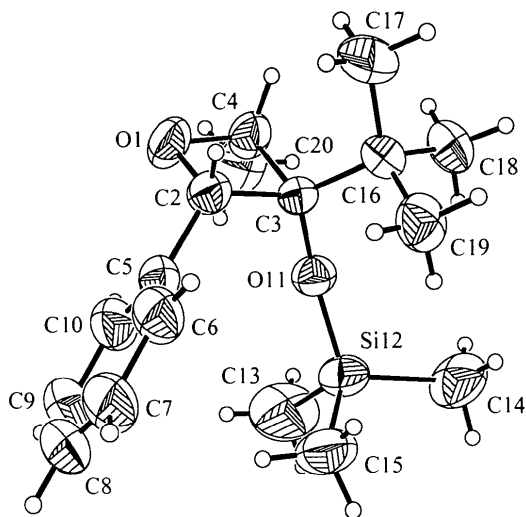


Fig. 1. *SHELXTL-Plus* (Sheldrick, 1990b) plot of (I) with the atomic numbering scheme and displacement ellipsoids shown at the 50% probability level.

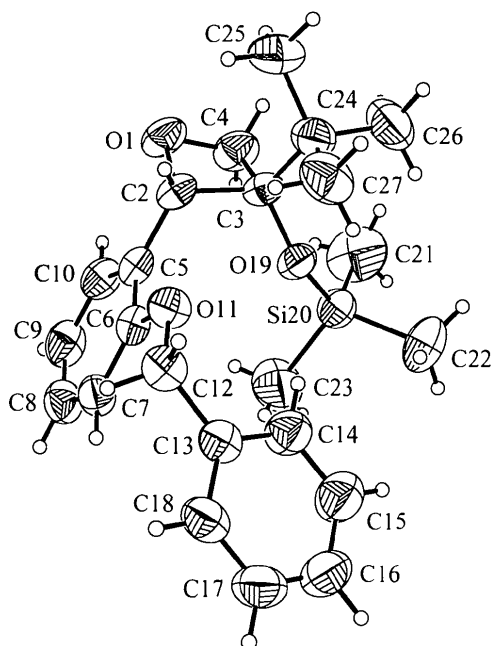


Fig. 2. *SHELXTL-Plus* (Sheldrick, 1990b) plot of (II) with the atomic numbering scheme and displacement ellipsoids shown at the 50% probability level.

is in agreement with the assumption that the C—O bond should be unusually long in four-membered rings (Mastryukov, Dorofeeva, Vilkov & Hargittai, 1976). The C—C—C angle is the smallest in the oxetane ring, with values of 84.8(2) for (I) and 84.1(2)° for (II), whereas the C—O—C and the O—C—C angles are in the range 89.7(2)–92.5(2)°. In previously determined oxetane structures, the C—C—C angle varies from 83.5

to 85.6°, and the corresponding range for the C—O—C and O—C—C angles is 89.9–92.5°. All phenyl groups are planar, with deviations from least-squares planes smaller than 0.012 Å. In compound (II), the dihedral angle between the phenyl plane and the benzyl plane is 89.0(1)°.

In the oxetane rings, the substituents are essentially in eclipsed positions and the conformations are determined by a balance between bond-angle and torsion-angle strains. The puckering of the oxetane ring releases some of this strain. The following torsion angles define the relative orientations of the substituents in the oxetane rings: C5—C2—C3—O11 14.7(2), C5—C2—C3—C16 –118.3(2), O11—C3—C4—C20 –8.3(3) and C16—C3—C4—C20 120.6(2)° in (I); C5—C2—C3—O19 –16.6(2) and C5—C2—C3—C24 –144.2(2)° in (II).

In the crystal packing, no intermolecular contacts are shorter than normal van der Waals separations.

Experimental

A quartz tube was charged with 1.5 mmol of aromatic aldehyde [(I): benzaldehyde (159 mg, 150 μ l); (II): 2-benzyloxybenzaldehyde (320 mg)], 3.0 mmol of silyl enol ether and 10 ml of benzene. The samples were irradiated at 300 nm (RPR 3000 Å) in an air-cooled merry-go-round unit (temperature *ca* 303 K). The reaction was monitored by thin-layer chromatography. After complete consumption of the aldehyde, irradiation was stopped. The solvent was evaporated *in vacuo* and the residue purified by flash chromatography using a cyclohexane/ethyl acetate solvent mixture (100/1 to 95/5). Crystals suitable for data collection were obtained by recrystallization from ether/pentane (1:1) solution.

Compound (I)

Crystal data

C₁₇H₂₈O₂Si

M_r = 292.48

Monoclinic

*P*2₁/*c*

a = 8.699(1) Å

b = 26.465(1) Å

c = 7.769(1) Å

β = 95.23(1)°

V = 1781.1(3) Å³

Z = 4

D_x = 1.091 Mg m⁻³

D_m not measured

Cu K α radiation

λ = 1.54178 Å

Cell parameters from 25 reflections

θ = 41.7–42.5°

μ = 1.15 mm⁻¹

T = 293(2) K

Block

0.6 × 0.4 × 0.4 mm

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

2 θ / ω scans

Absorption correction: none

3885 measured reflections

3617 independent reflections

3242 observed reflections

[*I* > 2 σ (*I*)]

R_{int} = 0.025

θ_{\max} = 74.1°

h = –10 → 10

k = 0 → 33

l = –9 → 0

3 standard reflections

orientation monitored

every 250 reflections

intensity check frequency:

120 min

intensity decay: 5%

Refinement

Refinement on F^2
 $R(F) = 0.055$
 $wR(F^2) = 0.181$
 $S = 1.075$
 3609 reflections
 188 parameters
 Methyl H atoms rigid, others riding
 $w = 1/[\sigma^2(F_o^2) + (0.1009P)^2 + 0.4515P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$
 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

Enraf-Nonius CAD-4 diffractometer
 $2\theta/\omega$ scans
 Absorption correction: none
 4009 measured reflections
 3850 independent reflections
 2942 observed reflections [$I > 2\sigma(I)$]
 $R_{\text{int}} = 0.051$

$\theta_{\max} = 24.6^\circ$
 $h = -17 \rightarrow 0$
 $k = 0 \rightarrow 11$
 $l = -17 \rightarrow 18$
 3 standard reflections orientation monitored every 250 reflections
 intensity check frequency: 120 min
 intensity decay: 4%

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (I)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Si12	0.20888 (6)	0.56554 (2)	0.36464 (7)	0.0547 (2)
O1	0.4945 (2)	0.66510 (6)	0.0469 (2)	0.0661 (4)
O11	0.3552 (2)	0.59702 (5)	0.3080 (2)	0.0480 (3)
C2	0.3894 (2)	0.68076 (7)	0.1716 (2)	0.0484 (4)
C3	0.4530 (2)	0.63864 (6)	0.3000 (2)	0.0403 (3)
C4	0.5718 (2)	0.62898 (8)	0.1667 (2)	0.0564 (5)
C5	0.2234 (2)	0.68175 (7)	0.1004 (2)	0.0498 (4)
C6	0.1249 (2)	0.71686 (8)	0.1628 (3)	0.0618 (5)
C7	-0.0299 (3)	0.71830 (11)	0.1015 (3)	0.0761 (7)
C8	-0.0865 (3)	0.68516 (13)	-0.0247 (3)	0.0827 (8)
C9	0.0101 (3)	0.65060 (13)	-0.0894 (3)	0.0826 (8)
C10	0.1662 (3)	0.64846 (10)	-0.0278 (3)	0.0656 (5)
C13	0.1658 (5)	0.51777 (13)	0.1925 (5)	0.1106 (11)
C14	0.2608 (3)	0.53150 (11)	0.5725 (4)	0.0865 (8)
C15	0.0354 (3)	0.60438 (11)	0.3902 (4)	0.0794 (7)
C16	0.5187 (2)	0.65620 (7)	0.4817 (2)	0.0499 (4)
C17	0.6283 (3)	0.70133 (11)	0.4747 (4)	0.0822 (7)
C18	0.6090 (3)	0.61218 (10)	0.5716 (3)	0.0681 (6)
C19	0.3867 (3)	0.67175 (9)	0.5861 (3)	0.0633 (5)
C20	0.5824 (4)	0.57799 (11)	0.0880 (4)	0.0910 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$) for (I)

O1—C2	1.452 (2)	C2—C3	1.563 (2)
O1—C4	1.455 (2)	C3—C4	1.550 (2)
O11—C3	1.396 (2)	C3—C16	1.545 (2)
C2—C5	1.498 (2)	C4—C20	1.488 (4)
C2—O1—C4	92.5 (1)	O11—C3—C2	114.2 (1)
O1—C2—C5	113.8 (2)	C16—C3—C2	116.7 (1)
O1—C2—C3	90.8 (1)	C4—C3—C2	84.8 (1)
C5—C2—C3	121.4 (1)	O1—C4—C20	112.1 (2)
O11—C3—C16	111.9 (1)	O1—C4—C3	91.3 (1)
O11—C3—C4	110.2 (1)	C20—C4—C3	119.6 (2)
C16—C3—C4	116.5 (1)		

Compound (II)

Crystal data

C₂₃H₃₂O₃Si
 $M_r = 384.58$
 Monoclinic
 $P2_1/c$
 $a = 14.701 (2) \text{ \AA}$
 $b = 10.153 (1) \text{ \AA}$
 $c = 15.420 (2) \text{ \AA}$
 $\beta = 96.26 (1)^\circ$
 $V = 2287.9 (5) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.117 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 10.0\text{--}18.0^\circ$

$\mu = 0.121 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Block

$1.0 \times 0.6 \times 0.5 \text{ mm}$

Colourless

Refinement

Refinement on F^2
 $R(F) = 0.046$
 $wR(F^2) = 0.147$
 $S = 1.052$
 3844 reflections
 250 parameters
 Methyl H atoms rigid, others riding
 $w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 0.4521P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (II)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Si20	0.37904 (4)	0.18526 (6)	0.72347 (4)	0.0570 (2)
O1	0.11600 (10)	0.3839 (2)	0.63835 (9)	0.0628 (4)
O11	0.10889 (9)	0.1254 (2)	0.84549 (8)	0.0487 (3)
O19	0.28860 (8)	0.2490 (2)	0.75901 (8)	0.0481 (4)
C2	0.12451 (13)	0.3032 (2)	0.71665 (12)	0.0464 (5)
C3	0.22518 (13)	0.3508 (2)	0.73805 (12)	0.0457 (5)
C4	0.2139 (2)	0.3994 (2)	0.64351 (14)	0.0623 (6)
C5	0.10917 (12)	0.1597 (2)	0.69613 (12)	0.0443 (5)
C6	0.10309 (12)	0.0703 (2)	0.76388 (11)	0.0417 (4)
C7	0.09038 (13)	-0.0627 (2)	0.74705 (13)	0.0495 (5)
C8	0.08098 (14)	-0.1067 (2)	0.66204 (14)	0.0570 (5)
C9	0.0847 (2)	-0.0194 (2)	0.59407 (14)	0.0610 (6)
C10	0.09925 (14)	0.1121 (2)	0.61167 (13)	0.0550 (5)
C12	0.09630 (14)	0.0415 (2)	0.91726 (12)	0.0504 (5)
C13	0.17753 (14)	-0.0442 (2)	0.94493 (11)	0.0471 (5)
C14	0.2644 (2)	-0.0094 (2)	0.9289 (2)	0.0624 (6)
C15	0.3384 (2)	-0.0854 (3)	0.9598 (2)	0.0784 (7)
C16	0.3262 (2)	-0.1978 (3)	1.0066 (2)	0.0771 (7)
C17	0.2406 (2)	-0.2337 (2)	1.0230 (2)	0.0735 (7)
C18	0.1666 (2)	-0.1582 (2)	0.9918 (2)	0.0601 (6)
C21	0.4267 (2)	0.2841 (4)	0.6382 (2)	0.1113 (12)
C22	0.4656 (2)	0.1762 (3)	0.8202 (2)	0.0939 (9)
C23	0.3494 (2)	0.0184 (3)	0.6826 (3)	0.1102 (12)
C24	0.2387 (2)	0.4599 (2)	0.8083 (2)	0.0578 (5)
C25	0.1680 (2)	0.5706 (2)	0.7908 (2)	0.0869 (8)
C26	0.3334 (2)	0.5205 (3)	0.8071 (2)	0.0861 (8)
C27	0.2299 (2)	0.4011 (3)	0.8977 (2)	0.0764 (7)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

O1—C2	1.453 (2)	C2—C3	1.558 (3)
O1—C4	1.442 (3)	C3—C4	1.531 (3)
O19—C3	1.405 (2)	C3—C24	1.547 (3)
C2—C5	1.502 (3)		

C4—O1—C2	91.2 (1)	C4—C3—C24	115.3 (2)
O1—C2—C5	112.0 (2)	O19—C3—C2	114.3 (2)
O1—C2—C3	89.7 (1)	C4—C3—C2	84.1 (2)
C5—C2—C3	117.6 (2)	C24—C3—C2	114.7 (2)
O19—C3—C4	117.2 (2)	O1—C4—C3	91.2 (1)
O19—C3—C24	109.5 (2)		

For both compounds, data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: own programs; program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990a); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990b); software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1113). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Pentafluorophenyl)diphenylphosphine

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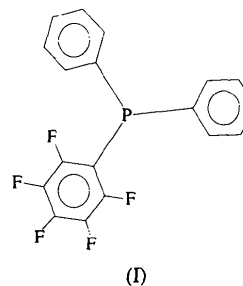
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Abstract

In solid C₁₈H₁₀F₅P the average P—C distance and C—P—C angle are 1.838 (2) Å and 102.9 (1)°, respectively. Intermolecular C—H···F hydrogen bonds and short F···F contacts are observed.

Comment

The title compound [(I), Fig. 1] has the expected geometry: the three P—C bonds are folded back from the lone-pair site, giving a slightly distorted tetrahedral geometry and C—P—C angles less than 109.5°.



The mean P—C bond length [1.838 (2) Å] is comparable with values of 1.828 (3) and 1.831 (2) Å for PPh₃ (Dunne & Orpen, 1991; Daly, 1964) and with the mean value of 1.828 Å for 2239 metal complexes containing PPh₃ (Orpen *et al.*, 1989). P—C12 is short [1.827 (2) Å] compared with P—C6 [1.846 (2) Å] and P—C18 [1.840 (2) Å]. A similar trend was observed in [(η⁶-C₆H₆)Cr(C₆F₅PPh₂)₂Rh(CO)Cl] (Faggiani, Hao, Lock, Sayer & McGlinchey, 1983). The average C—C distances are 1.375 (4) Å for the pentafluorophenyl ring and 1.382 (4) and 1.383 (4) Å for the two phenyl rings (C7—C12 and C13—C18).

The molecules are packed so that the lone pair of each P atom points along the shortest axis (*b* axis), a feature also observed in (2-methoxyphenyl)diphenylphosphine

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