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p-Bromobenzoate Derivative of 3-(*tert*-Butyldimethylsilyl)-5-hydroxy-2(5*H*)-furanone

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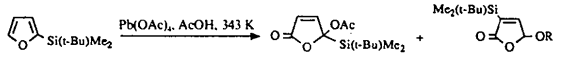
Abstract

In 3-(*tert*-butyldimethylsilyl)-2-oxo-5*H*-5-furyl 4-bromobenzoate, the furan ring is almost planar with a maximum deviation of 0.047 (6) Å, while the benzene ring is essentially planar [maximum deviation 0.012 (5) Å]; the best mean planes of the two rings are inclined at an angle of 73.7°. The average bond distances are: Si—C 1.865 (7), C_{sp^3}—C_{sp^3} 1.523 (9), C_{sp^2}—C_{sp^2} 1.487 (7), C—C_{arom} 1.381 (7), C_{sp^3}—O 1.423 (6), C_{sp^2}—O 1.362 (6) and C=O 1.204 (6) Å.

Comment

Treatment of 2-(*tert*-butyldimethylsilyl)furan (1) with lead tetraacetate in acetic acid at 343 K for 2 h provided compounds (2) and (3) in a 2:1 ratio. The



(1) (2) (3) R = Ac (4) R = COC_6H_4Br

major compound (2) was identified as 5-(*tert*-butyldimethylsilyl)-5-acetoxy-2(5*H*)-furanone by NMR, IR and MS spectra. The minor compound was identified by converting the acetate of compound (3) into a *p*-bromobenzoate (4) and performing an X-ray structure determination. The structure consists of discrete molecules separated by normal van der Waals distances.

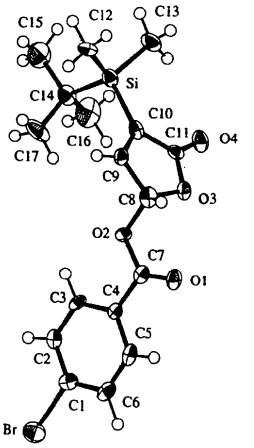


Fig. 1. *ORTEP* (Johnson, 1976) drawing of the title compound showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels; H atoms are assigned arbitrary radii.

Experimental

Crystal data

$C_{17}H_{21}BrO_4Si$
M_r = 397.34
Triclinic
$P\bar{1}$
a = 7.350 (4) Å
b = 19.906 (10) Å
c = 6.726 (5) Å
α = 93.93 (6)°
β = 109.10 (6)°
γ = 87.59 (5)°
V = 927 (1) Å^3
Z = 2
D_x = 1.423 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10–15°
μ = 2.27 mm^{-1}
T = 152 (1) K
Block
0.50 × 0.40 × 0.30 mm
Colourless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans, width (1.78 + 0.35tanθ)°, rate 8.0° min^{-1} in ω
Absorption correction: empirical (North, Phillips & Mathews, 1968)
T_{min} = 0.723, T_{max} = 0.999
3560 measured reflections
3279 independent reflections
2289 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.0255
θ_{max} = 25°
h = 0 → 8
k = −23 → 23
l = −7 → 7
3 standard reflections
frequency: 100 min
intensity variation: <0.7%

Refinement

Refinement on F
Final R = 0.048
wR = 0.033
S = 3.49
2289 reflections
271 parameters
Only coordinates of H atoms refined
$w = 1/(\sigma^2 F)$
$(\Delta/\sigma)_{max}$ = 0.05
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (8\pi^2/3)[(aa^*)^2U_{11} + (bb^*)^2U_{22} + (cc^*)^2U_{33} + (2aa^*bb^*\cos\gamma)U_{12} + (2aa^*cc^*\cos\beta)U_{13} + (2bb^*cc^*\cos\alpha)U_{23}]$.

	x	y	z	B_{eq}
Br	−0.0747 (1)	0.3478 (1)	0.2192 (1)	0.0405 (4)
Si	0.2622 (2)	0.8764 (1)	0.2976 (2)	0.028 (1)
O(1)	0.6436 (5)	0.5627 (2)	0.2602 (5)	0.031 (2)
O(2)	0.4188 (5)	0.6344 (2)	0.3208 (5)	0.024 (2)
O(3)	0.5209 (5)	0.7036 (2)	0.1153 (5)	0.026 (2)
O(4)	0.3812 (5)	0.7850 (2)	−0.0984 (5)	0.035 (3)
C(1)	0.0930 (8)	0.4189 (3)	0.2263 (7)	0.025 (4)
C(2)	0.0399 (8)	0.4824 (3)	0.2661 (8)	0.024 (3)
C(3)	0.1642 (8)	0.5348 (3)	0.2815 (8)	0.023 (4)
C(4)	0.3447 (7)	0.5196 (3)	0.2575 (7)	0.020 (3)
C(5)	0.3925 (8)	0.4543 (3)	0.2127 (8)	0.026 (4)
C(6)	0.2681 (9)	0.4025 (3)	0.1948 (8)	0.029 (4)
C(7)	0.4865 (8)	0.5727 (3)	0.2780 (7)	0.024 (4)
C(8)	0.5384 (8)	0.6891 (3)	0.3255 (9)	0.030 (4)
C(9)	0.4639 (7)	0.7501 (3)	0.4129 (8)	0.023 (3)
C(10)	0.3936 (7)	0.7950 (3)	0.2696 (8)	0.024 (3)
C(11)	0.4241 (7)	0.7639 (3)	0.0747 (8)	0.022 (4)
C(12)	0.3853 (10)	0.9129 (3)	0.5655 (10)	0.035 (4)
C(13)	0.2723 (9)	0.9342 (3)	0.1005 (10)	0.039 (4)
C(14)	0.0030 (7)	0.8550 (3)	0.2581 (9)	0.035 (4)
C(15)	−0.1001 (10)	0.9171 (4)	0.3215 (13)	0.057 (6)
C(16)	−0.0978 (10)	0.8343 (4)	0.0286 (12)	0.061 (6)
C(17)	−0.0033 (10)	0.7982 (4)	0.3969 (13)	0.055 (5)

Table 2. *Geometric parameters* (Å, °)

Br—C(1)	1.904 (6)	Si—C(10)	1.881 (6)
Si—C(12)	1.845 (7)	Si—C(13)	1.836 (7)
Si—C(14)	1.898 (6)	O(1)—C(7)	1.206 (6)
O(2)—C(7)	1.355 (6)	O(2)—C(8)	1.420 (6)
O(3)—C(8)	1.426 (6)	O(3)—C(11)	1.368 (6)
O(4)—C(11)	1.202 (6)	C(1)—C(2)	1.343 (7)
C(1)—C(6)	1.393 (9)	C(2)—C(3)	1.391 (9)
C(3)—C(4)	1.406 (7)	C(4)—C(5)	1.372 (7)
C(4)—C(7)	1.484 (7)	C(5)—C(6)	1.380 (8)
C(8)—C(9)	1.476 (7)	C(9)—C(10)	1.321 (7)
C(10)—C(11)	1.490 (7)	C(14)—C(15)	1.531 (9)
C(14)—C(16)	1.513 (9)	C(14)—C(17)	1.526 (11)
C(10)—Si—C(12)	106.4 (3)	C(10)—Si—C(13)	110.5 (3)
C(10)—Si—C(14)	107.2 (3)	C(12)—Si—C(13)	110.1 (3)
C(12)—Si—C(14)	112.1 (3)	C(13)—Si—C(14)	110.3 (3)
C(7)—O(2)—C(8)	115.6 (4)	C(8)—O(3)—C(11)	107.6 (4)
Br—C(1)—C(2)	118.8 (5)	Br—C(1)—C(6)	118.4 (5)
C(2)—C(1)—C(6)	122.8 (6)	C(1)—C(2)—C(3)	119.7 (6)
C(2)—C(3)—C(4)	118.8 (5)	C(3)—C(4)—C(5)	119.9 (5)
C(3)—C(4)—C(7)	121.7 (5)	C(5)—C(4)—C(7)	118.4 (5)
C(4)—C(5)—C(6)	121.2 (5)	C(1)—C(6)—C(5)	117.5 (6)
O(1)—C(7)—O(2)	123.5 (6)	O(1)—C(7)—C(4)	124.8 (6)
O(2)—C(7)—C(4)	111.7 (5)	O(2)—C(8)—O(3)	109.4 (5)
O(2)—C(8)—C(9)	108.6 (5)	O(3)—C(8)—C(9)	105.1 (5)
C(8)—C(9)—C(10)	111.8 (5)	Si—C(10)—C(9)	127.5 (4)
Si—C(10)—C(11)	127.3 (4)	C(9)—C(10)—C(11)	104.9 (5)
O(3)—C(11)—O(4)	120.0 (5)	O(3)—C(11)—C(10)	110.2 (5)
O(4)—C(11)—C(10)	129.8 (5)	Si—C(14)—C(15)	109.2 (5)
Si—C(14)—C(16)	109.6 (5)	Si—C(14)—C(17)	110.1 (4)
C(15)—C(14)—C(16)	109.3 (6)	C(15)—C(14)—C(17)	108.4 (6)
C(16)—C(14)—C(17)	110.2 (6)		

Space group $P\bar{1}$ or $P1$; the former was assumed and confirmed by successful analysis. Lorentz-polarization corrections were applied but not extinction corrections. The structure was solved by the heavy-atom method and refined by full-matrix least squares with the non-H atoms anisotropic. The H atoms were located from the ΔF map and allowed to refine with fixed isotropic temperature factors. The scattering factors were taken from Cromer & Waber (1974) and Stewart, Davidson & Simpson (1965); allowance was made for anomalous dispersion (Ibers & Hamilton, 1964). All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1992) on a Silicongraphics Personal Iris D/35 computer. A search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) yielded only one hit of a similar compound, a diphenylmethylsilylbenzofuran (de Perez, Fuentes, Larson, Barnes & Heeg, 1986).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55823 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1021]

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Structure of Methyl 2-(Nitrooxy)ethyl 1,4-Dihydro-2,6-dimethyl-4-(3-nitrophenyl)-3,5-pyridinedicarboxylate

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Abstract

The orientations of the carbonyl groups at C3 and C5 are different. The phenyl ring linked to C4 is perpendicular to the dihydropyridine ring. Some other structural features have also been elucidated.

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