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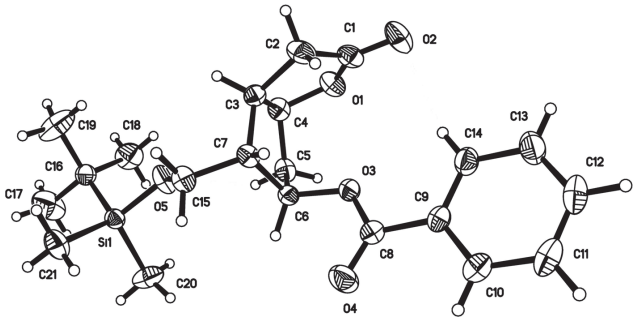
A derivative of the Corey lactone – crystal structure of (3a*R*,4*S*,5*R*,6a*S*)-4-(((*tert*-butyldimethylsilyl)oxy)methyl)-2-oxohexahydro-2*H*-cyclopenta[*b*]furan-5-yl benzoate, $C_{21}H_{30}O_5Si$



https://doi.org/10.1515/ncrs-2017-0184
Received July 7, 2017; accepted November 25, 2017; available online December 16, 2017

Abstract

$C_{21}H_{30}O_5Si$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 8.5017(6)$ Å, $b = 11.4370(7)$ Å, $c = 22.2285(12)$ Å, $V = 2161.4(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0499$, $wR_{ref}(F^2) = 0.0972$, $T = 296(2)$ K.

CCDC no.: 1587576

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The compound we report here, can be synthesized following the procedures given in a known patent [3]. Evaporation of a methanol solution at room temperature yielded colourless crystals.

Experimental details

The hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 23, 13, 43, 0 or 137 option) [2].

Discussion

The title compound, we report here is an important derivative of a Corey lactone. Corey lactones are the most

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.40 × 0.37 × 0.35 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.14 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω-scans
θ_{max}, completeness:	25.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25865, 3965, 0.085
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2970
$N(param)_{refined}$:	249
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0122(6)	1.1086(3)	0.72001(18)	0.0531(11)
C2	0.1357(6)	1.0178(4)	0.72813(16)	0.0609(12)
H2A	0.2261	1.0347	0.7029	0.073*
H2B	0.0952	0.9413	0.7175	0.073*
C3	0.1819(4)	1.0215(3)	0.79473(15)	0.0443(10)
H3	0.2963	1.0274	0.7992	0.053*
C4	0.1008(5)	1.1332(3)	0.81780(17)	0.0481(10)
H4	0.1779	1.1914	0.8311	0.058*
C5	−0.0063(5)	1.0966(3)	0.86881(16)	0.0498(10)
H5A	0.0437	1.1109	0.9074	0.060*
H5B	−0.1045	1.1396	0.8673	0.060*
C6	−0.0349(4)	0.9678(3)	0.85986(15)	0.0422(9)
H6	−0.0605	0.9288	0.8979	0.051*
C7	0.1169(4)	0.9202(3)	0.83271(15)	0.0398(9)
H7	0.0906	0.8552	0.8058	0.048*
C8	−0.2490(5)	0.8581(3)	0.81921(18)	0.0457(10)
C9	−0.3591(5)	0.8449(3)	0.76816(17)	0.0427(9)
C10	−0.4689(5)	0.7557(4)	0.7699(2)	0.0575(12)
H10	−0.4730	0.7065	0.8032	0.069*
C11	−0.5725(5)	0.7387(4)	0.7228(3)	0.0724(15)
H11	−0.6471	0.6794	0.7246	0.087*
C12	−0.5645(6)	0.8099(5)	0.6735(2)	0.0729(14)
H12	−0.6335	0.7985	0.6415	0.088*
C13	−0.4552(6)	0.8981(5)	0.6708(2)	0.0713(14)
H13	−0.4500	0.9458	0.6370	0.086*
C14	−0.3527(5)	0.9163(4)	0.71816(17)	0.0549(11)
H14	−0.2794	0.9766	0.7163	0.066*
C15	0.2303(5)	0.8763(3)	0.88019(16)	0.0488(10)
H15A	0.1801	0.8152	0.9036	0.059*
H15B	0.3220	0.8429	0.8607	0.059*
C16	0.4945(5)	1.0699(3)	0.99722(16)	0.0490(10)
C17	0.5536(8)	1.0765(6)	1.0623(2)	0.115(2)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H17A	0.6088	1.0058	1.0722	0.172*
H17B	0.4657	1.0859	1.0890	0.172*
H17C	0.6233	1.1420	1.0666	0.172*
C18	0.4219(7)	1.1878(4)	0.9822(2)	0.0821(16)
H18A	0.4993	1.2481	0.9872	0.123*
H18B	0.3347	1.2024	1.0087	0.123*
H18C	0.3857	1.1873	0.9413	0.123*
C19	0.6300(6)	1.0494(5)	0.9540(3)	0.109(2)
H19A	0.5899	1.0412	0.9139	0.164*
H19B	0.6850	0.9794	0.9653	0.164*
H19C	0.7010	1.1146	0.9556	0.164*
C20	0.1779(6)	0.9726(4)	1.0409(2)	0.0794(15)
H20A	0.1418	1.0520	1.0386	0.119*
H20B	0.2122	0.9560	1.0811	0.119*
H20C	0.0936	0.9207	1.0302	0.119*
C21	0.4303(6)	0.8051(4)	0.9990(2)	0.0731(14)
H21A	0.3511	0.7467	0.9926	0.110*
H21B	0.4703	0.7987	1.0393	0.110*
H21C	0.5146	0.7939	0.9708	0.110*
O1	0.0048(4)	1.1778(2)	0.76851(12)	0.0583(8)
O2	−0.0718(4)	1.1252(3)	0.67801(14)	0.0782(10)
O3	−0.1612(3)	0.9548(2)	0.81594(10)	0.0493(6)
O4	−0.2388(4)	0.7890(3)	0.85975(14)	0.0776(10)
O5	0.2783(3)	0.9675(2)	0.91904(11)	0.0574(8)
Si1	0.34377(13)	0.95170(9)	0.98809(4)	0.0414(3)

important educts for the synthesis of prostaglandins [4]. The prostaglandins (PGs) are a group of physiologically active lipid compounds having diverse hormone-like effects in animals. Prostaglandins have been found in almost every tissue in humans and animals. They are derived enzymatically from fatty acids. Every prostaglandin contains 20 carbon atoms, including a membered 5-carbon ring. They are a subclass of eicosanoids and of the prostanoid class of fatty acid derivatives. They are powerful locally acting vasodilators and inhibit the aggregation of blood platelets.

The molecular structure of the title compound is shown in the figure. All bond lengths and angles are within normal ranges [5, 6]. There are 3 rings: ring A (C9—C10—C11—C12—C13—C14), ring B (O1—C1—C2—C3—C4) and ring C (C3—C4—C5—C6—C7), which are not coplanar. The dihedral angles between the benzene ring A and ring B is 75(2)°. The dihedral angles between the benzene ring A and ring C is 85.3(3)°. The dihedral angles between the ring C and ring B is 72.6(2)°. In the crystal structure, there may be some weak π-π stacking interactions, as the centroid to centroid distance between the two nearest rings is less than 5 Å.

Acknowledgements: This work is supported by Jiangsu Provincial Natural Science Foundation for Youths (Grant No. BK20170118).

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