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(2*S*,3*R*,4*R*,5*R*)-2-(*tert*-Butyldimethylsilyloxymethyl)-2,4,5-trimethyltetrahydrofuran-3,4-diol

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.012 Å
R factor = 0.067
wR factor = 0.182
Data-to-parameter ratio = 8.8

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

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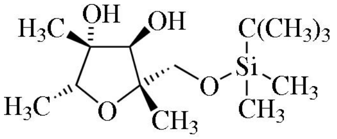
In the title compound, $C_{14}H_{30}O_4Si$, the bond angles around Si are in the range 105.4 (3)–112.1 (4)° and the tetrahydrofuran ring adopts a distorted envelope conformation. There is one intramolecular and one intermolecular O—H···O hydrogen bond in the crystal structure. A spiral of molecules forms around the 4_1 axis (*c* axis) of the crystal structure *via* the intermolecular O—H···O hydrogen bond.

Comment

The title compound, (I), is an intermediate in the enantioselective synthesis of the natural products citreoviral and citreoviridin. The compound was obtained in a multistep sequence involving a gold-catalysed cycloisomerization (Hoffmann-Röder & Krause, 2001). The crystal structure proves the relative configuration of the four stereogenic centres within the tetrahydrofuran (THF) ring. The five-membered ring has the shape of a slightly distorted envelope with the torsion angles 31.8 (8), −44.5 (7), 40.3 (8), −18.9 (8) and −9.6 (8)°.



(I)

Experimental

The synthesis of (I) will be described elsewhere (Fan & Krause, 2005). It was dissolved in a small amount of THF and hexane, and crystals were obtained by vapour diffusion of hexane.

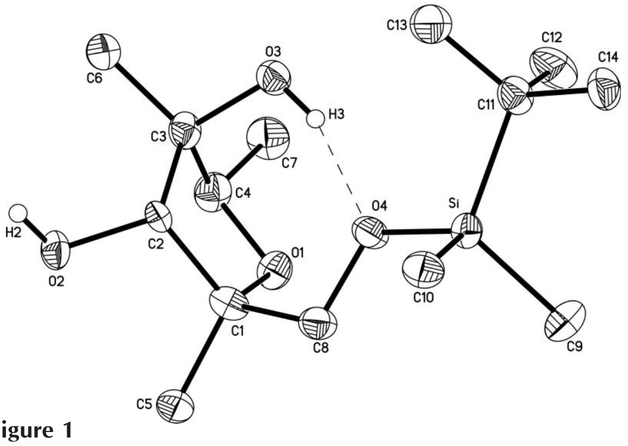


Figure 1
The molecular structure of the title compound, showing the labelling of all non-H atoms and of the H atoms involved in O—H···O hydrogen bonds. The remaining H atoms have been omitted for clarity. The dashed line indicates the intramolecular hydrogen bond. Displacement ellipsoids are shown at the 30% probability level.

Crystal data

$C_{14}H_{30}O_4Si$
$M_r = 290.47$
Tetragonal, $P4_1$
$a = 12.3616$ (12) Å
$c = 11.1685$ (11) Å
$V = 1706.6$ (3) Å^3
$Z = 4$
$D_x = 1.130$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20589 reflections
$\theta = 3.0$–25.4°
$\mu = 0.14$ mm^{-1}
$T = 173$ (1) K
Needle, colourless
0.36 × 0.08 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: none
20589 measured reflections
1607 independent reflections
1227 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\rm max} = 25.4°$
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.182$
$S = 1.15$
1607 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 4.0462P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3$^{\rm i}$	0.82	2.02	2.834 (7)	170
O3—H3···O4	0.82	1.97	2.750 (8)	158

Symmetry code: (i) $-y+1, x, z+\frac{1}{4}$.

In the absence of significant anomalous scattering effects, Friedel pairs were merged. H atoms were placed in calculated positions, with C—H = 0.96–0.98 Å and O—H = 0.82 Å, and were refined as riding, with $U_{\rm iso}$(H) values of $1.5U_{\rm eq}$(C,O) for methyl and hydroxy groups, and $1.2U_{\rm eq}$(C) for others; the methyl and hydroxy groups were allowed to rotate but not to tip.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*, *PARST95* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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