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# 5-*O*-*tert*-Butyldiphenylsilyl-2-*C*-hydroxymethyl-2,3-*O*-isopropylidene-2′-*O*-trifluoromethanesulfonyl-D-ribono-1,4-lactone

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**Key indicators**

Single-crystal X-ray study
*T* = 150 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.033
*wR* factor = 0.033
Data-to-parameter ratio = 13.6

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

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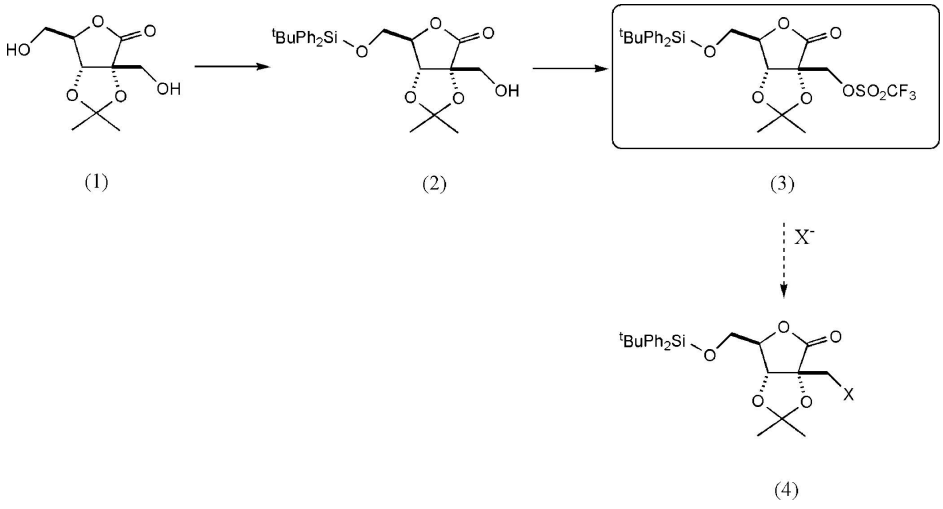
The title compound, $C_{26}H_{31}F_3O_8SSi$, provides a unique example of the crystal structure of an organic trifluoromethanesulfonate attached to a primary C atom. The absolute configuration is determined by the use of D-ribose as the starting material.

## Comment

Sulfonate esters provide a wide range of leaving groups for nucleophilic substitution reactions in organic chemistry (Bentley, 1991). A β-oxygen substituent very substantially retards either $S_N1$ or $S_N2$ reactions (Shaik, 1983); in carbohydrate chemistry, where there is always a β-oxygen, nucleophilic substitutions at secondary carbons are usually too slow if a mesylate or a tosylate is used as a leaving group (Richardson, 1969). However trifluoromethanesulfonate (Howells & McCown, 1977; Rakita, 2004) is an excellent leaving group with a rate increase of around $10^5$ in comparison to tosylate in $S_N1$ (Takeuchi *et al.*, 1988) and $S_N2$ reactions (Streitwieser *et al.*, 1968), and in decarboxylative eliminations (Fleming & Ramarao, 2004). Trifluoromethanesulfonates are relatively unstable; few crystal structures of organic trifluoromethanesulfonates have been reported. The first crystal structure of a secondary trifluoromethanesulfonate was reported by Barnes *et al.* (1996) and a further two have been reported (Hung *et al.*, 2001; Tremmel *et al.*, 2003). Although two crystal structures of primary trifluoromethanesulfonates of carboranes have been published (Herzog *et al.*, 1999; Kalinin *et al.*, 2005), the present paper reports the first example of the crystal structure of a primary trifluoromethanesulfonate, (3).




(1) (2) (3) (4)

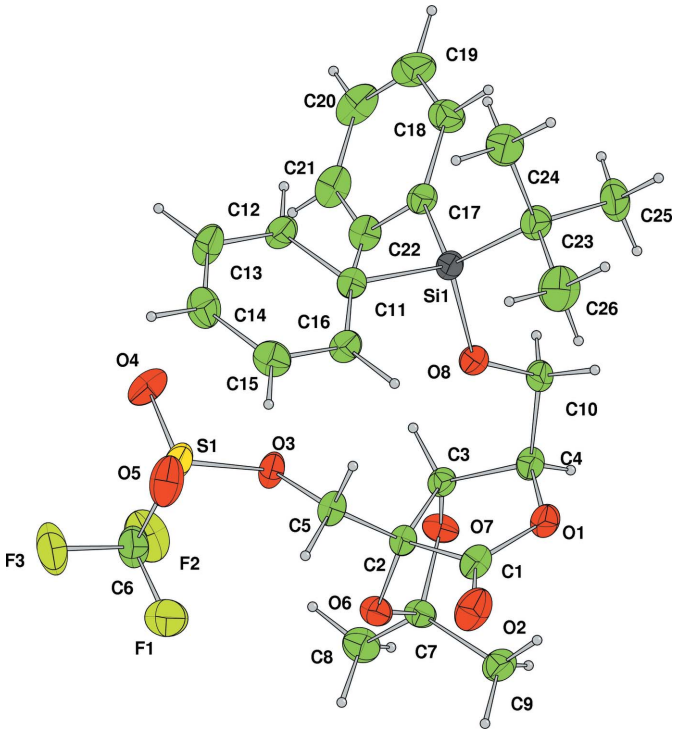



**Figure 1**
The molecular structure of (3), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

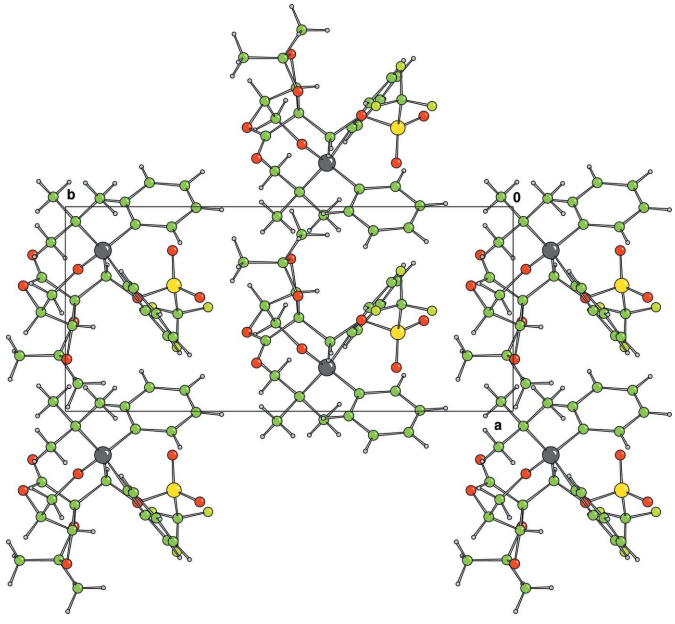



**Figure 2**
Packing diagram of (3) viewed along the *c* axis, showing the columns of molecules lying parallel to *a*.

In a study of secondary structures of novel peptides (Jockusch *et al.*, 2006), the synthesis of a number of carbon-branched sugar amino acids (Simone *et al.*, 2005) required displacements by nucleophiles ($X^-$ = $N_3^-$, $I^-$) of the leaving group in the very hindered neopentyl trifluoromethanesulfonate (3), yielding (4). D-Ribose was converted to the protected hamamelonolactone (1) (Ho, 1979, 1985) as previously described. The less hindered primary alcohol in (1) was selectively protected as the very bulky *tert*-butyldiphenylsilyl ether (2). Esterification of the remaining neopentyl alcohol in (2) with trifluoromethanesulfonic (triflic) anhydride gave the trifluoromethanesulfonate (3) as a stable crystalline compound, allowing the first X-ray crystallographic analysis of a primary organic trifluoromethanesulfonate. The crystal structure of (3) confirmed the relative stereochemistry and the integrity of the trifluoromethanesulfonate functional group; the absolute configuration of (3) was determined by the use of D-ribose as the starting material.

There are no unusual bond lengths or angles in the structure (Fig. 1), the largest differences from the Mogul norms (Bruno *et al.*, 2004) being O8—Si1 (0.02 Å; Mogul s.u. 0.01 Å) and S1—O5—O4 (5.9°; Mogul s.u. 3.7°). The Flack parameter refined to −0.04 (7), enabling the absolute configuration of the molecule to be assigned with confidence.

The crystal structure consists of discrete molecules without any specific strong interactions between them. The molecules are well separated in the *b* and *c* directions, giving the appearance of columns in close contact, parallel to *a* (Fig. 2).

## Experimental

Triflic anhydride (97 µl, 0.58 mmol) was added dropwise to a stirred solution of the silyl ether (2) (203 mg, 0.44 mmol) in dichloromethane (1.7 ml) containing dry pyridine (79 µl) at 243 K under an atmosphere of argon. After 20 min, thin layer chromatography (ethyl acetate/cyclohexane, 1:4) indicated the presence of a major UV-active product ($R_f$ = 0.45) and complete consumption of the starting material ($R_f$ = 0.11). The reaction mixture was diluted with dichloromethane (20 ml), and washed with aqueous hydrochloric acid solution (1*M*, 2.0 ml), then with a buffer solution [pH 7, $K_2H_2PO_4$ (0.51 *M*)/NaOH (0.38 *M*), 1.0 ml]. The organic layers were dried (magnesium sulfate) and filtered, and the filtrate was concentrated *in vacuo* to give a residue which was purified by flash column chromatography (ethyl acetate/cyclohexane, 1:6 to 1:3), to yield the trifluoromethanesulfonate (3) (239 mg, 91% yield) as a colourless oil which crystallized on standing. M.p. 367–370 K; $[\alpha]_D^{25}$ +9.0 (*c*, 0.94 in acetonitrile); $\nu_{max}$ (thin film): 1785 (*s*, C═O) cm$^{-1}$. A sample of (3), suitable for X-ray crystallographic analysis, was obtained *via* solvent evaporation (ethyl acetate/cyclohexane).

*Crystal data*

$C_{26}H_{31}F_3O_8SSi$
$M_r$ = 588.67
Orthorhombic, $P2_12_12_1$
$a$ = 7.7889 (1) Å
$b$ = 17.0479 (3) Å
$c$ = 21.2824 (3) Å
$V$ = 2825.97 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.384 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 150 K
Block, colourless
0.32 × 0.24 × 0.20 mm

*Data collection*

Nonius KappaCCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min}$ = 0.93, $T_{max}$ = 0.96
29606 measured reflections
6416 independent reflections
4788 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.055
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F$
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.033$
$S = 1.06$
4788 reflections
353 parameters
H-atom parameters not refined
Chebychev polynomial with three parameters (Carruthers & Watkin, 1979) 0.297, 0.0573 and 0.0793
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$
Absolute structure: Flack (1983), 2791 Friedel pairs
Flack parameter: −0.04 (7)

All H atoms were found in difference Fourier maps, but were repositioned geometrically after each cycle of refinement; C—H = 1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2001).; cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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# supporting information

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$Z = 4$
$F(000) = 1232$
$D_x = 1.384$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 29606 reflections
$\theta = 5$–28°
$\mu = 0.22$ mm$^{-1}$
$T = 150$ K
Block, colourless
0.32 × 0.24 × 0.20 mm

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$T_{min} = 0.93$, $T_{max} = 0.96$
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6416 independent reflections
4788 reflections with $I > 3\sigma(I)$
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$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -10 \rightarrow 10$
$k = -22 \rightarrow 22$
$l = -27 \rightarrow 27$

*Refinement*

Refinement on $F$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.033$
$S = 1.06$
4788 reflections
353 parameters
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined
Chebychev polynomial *w*ith three parameters (Carruthers & Watkin, 1979) 0.297, 0.0573 and 0.0793
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$
Absolute structure: Flack (1983), 2791 Friedel pairs
Absolute structure parameter: −0.04 (7)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.1132 (2) | −0.09395 (8) | 0.30408 (7) | 0.0304 |

| | | | | |
|---|---|---|---|---|
| C1 | 0.1544 (3) | −0.05246 (12) | 0.35539 (11) | 0.0290 |
| C2 | 0.0421 (3) | 0.02148 (11) | 0.36056 (9) | 0.0236 |
| C3 | −0.0810 (3) | 0.01558 (11) | 0.30473 (9) | 0.0232 |
| C4 | −0.0154 (3) | −0.05425 (11) | 0.2658 (1) | 0.0266 |
| O2 | 0.2640 (2) | −0.07288 (9) | 0.39182 (9) | 0.0408 |
| C5 | 0.1602 (2) | 0.09126 (12) | 0.3645 (1) | 0.0265 |
| O3 | 0.05389 (19) | 0.16164 (8) | 0.35441 (7) | 0.0291 |
| S1 | 0.11475 (8) | 0.24270 (3) | 0.37960 (3) | 0.0328 |
| O4 | 0.0571 (3) | 0.3005 (1) | 0.33682 (9) | 0.0519 |
| O5 | 0.2860 (2) | 0.2394 (1) | 0.40186 (9) | 0.0470 |
| C6 | −0.0229 (3) | 0.25115 (14) | 0.44861 (11) | 0.0389 |
| F1 | 0.0080 (2) | 0.1936 (1) | 0.48786 (7) | 0.0566 |
| F2 | −0.18620 (19) | 0.24919 (11) | 0.43224 (8) | 0.0570 |
| F3 | 0.0084 (3) | 0.31886 (9) | 0.47665 (8) | 0.0614 |
| O6 | −0.06340 (17) | 0.01953 (8) | 0.41459 (6) | 0.0262 |
| O7 | −0.24526 (17) | 0.00347 (8) | 0.33208 (7) | 0.0273 |
| C7 | −0.2262 (3) | −0.01392 (12) | 0.39767 (9) | 0.0249 |
| C8 | −0.3641 (3) | 0.02811 (14) | 0.43398 (11) | 0.0350 |
| C9 | −0.2287 (3) | −0.10197 (12) | 0.40774 (11) | 0.0328 |
| C10 | 0.0686 (3) | −0.02992 (12) | 0.2051 (1) | 0.0286 |
| O8 | 0.19443 (18) | 0.02857 (8) | 0.21883 (6) | 0.0260 |
| Si1 | 0.28595 (7) | 0.08423 (3) | 0.16397 (3) | 0.0229 |
| C11 | 0.4132 (3) | 0.15675 (11) | 0.21083 (9) | 0.0240 |
| C12 | 0.4015 (3) | 0.23743 (12) | 0.1981 (1) | 0.0296 |
| C13 | 0.4952 (3) | 0.29198 (14) | 0.23258 (12) | 0.0382 |
| C14 | 0.6027 (3) | 0.26699 (14) | 0.28012 (11) | 0.0380 |
| C15 | 0.6171 (3) | 0.18799 (14) | 0.29389 (11) | 0.0344 |
| C16 | 0.5230 (3) | 0.13321 (12) | 0.2595 (1) | 0.0282 |
| C17 | 0.1131 (3) | 0.13991 (11) | 0.1223 (1) | 0.0268 |
| C18 | 0.1020 (3) | 0.15241 (14) | 0.0574 (1) | 0.0339 |
| C19 | −0.0220 (3) | 0.20302 (16) | 0.03243 (12) | 0.0437 |
| C20 | −0.1368 (3) | 0.24101 (15) | 0.07124 (13) | 0.0415 |
| C21 | −0.1304 (3) | 0.22856 (13) | 0.13519 (12) | 0.0373 |
| C22 | −0.0074 (3) | 0.17878 (13) | 0.16037 (11) | 0.0302 |
| C23 | 0.4279 (3) | 0.02309 (12) | 0.1115 (1) | 0.0331 |
| C24 | 0.5407 (3) | 0.07733 (15) | 0.07131 (13) | 0.0450 |
| C25 | 0.3262 (4) | −0.03198 (15) | 0.06794 (13) | 0.0462 |
| C26 | 0.5478 (4) | −0.02684 (17) | 0.15282 (14) | 0.0520 |
| H31 | −0.0879 | 0.0623 | 0.2764 | 0.0278* |
| H41 | −0.1155 | −0.0882 | 0.2547 | 0.0319* |
| H51 | 0.2157 | 0.0935 | 0.4069 | 0.0318* |
| H52 | 0.2509 | 0.0877 | 0.3314 | 0.0318* |
| H81 | −0.4788 | 0.0056 | 0.4228 | 0.0421* |
| H82 | −0.3433 | 0.0214 | 0.4800 | 0.0421* |
| H83 | −0.3620 | 0.0852 | 0.4232 | 0.0421* |
| H91 | −0.3436 | −0.1232 | 0.3955 | 0.0394* |
| H92 | −0.2064 | −0.1138 | 0.4530 | 0.0394* |
| H93 | −0.1378 | −0.1270 | 0.3813 | 0.0394* |

| | | | | |
|---|---|---|---|---|
| H101 | 0.1250 | −0.0763 | 0.1851 | 0.0343* |
| H102 | −0.0197 | −0.0080 | 0.1759 | 0.0343* |
| H121 | 0.3243 | 0.2560 | 0.1636 | 0.0356* |
| H131 | 0.4847 | 0.3492 | 0.2229 | 0.0459* |
| H141 | 0.6704 | 0.3062 | 0.3047 | 0.0456* |
| H151 | 0.6949 | 0.1702 | 0.3284 | 0.0413* |
| H161 | 0.5340 | 0.0762 | 0.2697 | 0.0338* |
| H181 | 0.1836 | 0.1249 | 0.0286 | 0.0407* |
| H191 | −0.0273 | 0.2117 | −0.0140 | 0.0524* |
| H201 | −0.2244 | 0.2774 | 0.0531 | 0.0498* |
| H211 | −0.2144 | 0.2554 | 0.1635 | 0.0448* |
| H221 | −0.0044 | 0.1703 | 0.2068 | 0.0362* |
| H241 | 0.6161 | 0.0450 | 0.0435 | 0.0540* |
| H242 | 0.6137 | 0.1106 | 0.0994 | 0.0540* |
| H243 | 0.4660 | 0.1118 | 0.0449 | 0.0540* |
| H251 | 0.4078 | −0.0628 | 0.0414 | 0.0554* |
| H252 | 0.2555 | −0.0687 | 0.0939 | 0.0554* |
| H253 | 0.2490 | −0.0002 | 0.0403 | 0.0554* |
| H261 | 0.6237 | −0.0595 | 0.1254 | 0.0625* |
| H262 | 0.6200 | 0.0084 | 0.1795 | 0.0625* |
| H263 | 0.4775 | −0.0618 | 0.1804 | 0.0625* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0374 (8) | 0.0213 (7) | 0.0324 (8) | 0.0046 (7) | 0.0044 (7) | −0.0000 (6) |
| C1 | 0.0265 (11) | 0.024 (1) | 0.0366 (12) | −0.0007 (8) | 0.0010 (9) | 0.0024 (9) |
| C2 | 0.0252 (9) | 0.0197 (9) | 0.026 (1) | 0.0007 (8) | 0.0002 (8) | −0.0007 (8) |
| C3 | 0.024 (1) | 0.0204 (9) | 0.025 (1) | −0.0022 (8) | 0.0036 (8) | 0.0006 (8) |
| C4 | 0.0279 (11) | 0.021 (1) | 0.0307 (11) | −0.0018 (8) | 0.0011 (9) | −0.0017 (8) |
| O2 | 0.0355 (9) | 0.0310 (8) | 0.0559 (11) | 0.0043 (7) | −0.0122 (8) | 0.0044 (8) |
| C5 | 0.024 (1) | 0.0207 (9) | 0.0345 (11) | 0.0005 (8) | −0.0006 (8) | −0.0013 (9) |
| O3 | 0.0336 (8) | 0.0175 (7) | 0.0361 (8) | 0.0004 (6) | −0.0073 (6) | −0.0029 (6) |
| S1 | 0.0389 (3) | 0.0194 (2) | 0.0401 (3) | −0.0035 (2) | −0.0035 (2) | −0.0006 (2) |
| O4 | 0.0789 (14) | 0.0265 (8) | 0.050 (1) | 0.0023 (8) | −0.0019 (11) | 0.0130 (8) |
| O5 | 0.0369 (9) | 0.0346 (9) | 0.0696 (12) | −0.0085 (8) | −0.0065 (9) | −0.0110 (9) |
| C6 | 0.0456 (14) | 0.0291 (12) | 0.0421 (13) | 0.0050 (11) | −0.0066 (11) | −0.0084 (11) |
| F1 | 0.0821 (12) | 0.0480 (9) | 0.0398 (8) | 0.0122 (9) | 0.0073 (8) | 0.0050 (7) |
| F2 | 0.0407 (8) | 0.0683 (11) | 0.062 (1) | 0.0083 (8) | 0.0004 (7) | −0.0157 (9) |
| F3 | 0.0839 (13) | 0.0421 (9) | 0.058 (1) | 0.0051 (9) | −0.0043 (9) | −0.0248 (8) |
| O6 | 0.0255 (7) | 0.0303 (8) | 0.0229 (7) | −0.0035 (6) | 0.0004 (6) | −0.0003 (6) |
| O7 | 0.0227 (7) | 0.0373 (8) | 0.0219 (7) | 0.0005 (6) | −0.0003 (6) | 0.0025 (6) |
| C7 | 0.0248 (9) | 0.029 (1) | 0.0204 (9) | −0.0032 (8) | −0.0005 (8) | 0.0004 (8) |
| C8 | 0.0318 (12) | 0.0400 (13) | 0.0334 (12) | 0.001 (1) | 0.006 (1) | −0.001 (1) |
| C9 | 0.0373 (12) | 0.0280 (11) | 0.0332 (12) | −0.0058 (9) | −0.001 (1) | 0.0040 (9) |
| C10 | 0.0369 (12) | 0.022 (1) | 0.026 (1) | −0.0082 (9) | 0.0043 (9) | −0.0037 (8) |
| O8 | 0.0292 (8) | 0.0235 (7) | 0.0252 (7) | −0.0048 (6) | 0.0011 (6) | 0.0004 (6) |
| Si1 | 0.0250 (2) | 0.0192 (2) | 0.0244 (3) | −0.0008 (2) | 0.0023 (2) | 0.0000 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C11 | 0.022 (1) | 0.0234 (9) | 0.027 (1) | −0.0001 (8) | 0.0018 (8) | −0.0003 (8) |
| C12 | 0.0323 (11) | 0.023 (1) | 0.0336 (11) | −0.0007 (9) | −0.004 (1) | 0.0039 (8) |
| C13 | 0.0421 (13) | 0.023 (1) | 0.0499 (15) | −0.005 (1) | −0.0059 (12) | −0.001 (1) |
| C14 | 0.0355 (12) | 0.0378 (12) | 0.0406 (13) | −0.0091 (11) | −0.007 (1) | −0.008 (1) |
| C15 | 0.0290 (11) | 0.0413 (13) | 0.0330 (12) | −0.002 (1) | −0.005 (1) | −0.000 (1) |
| C16 | 0.0275 (11) | 0.0257 (11) | 0.0313 (11) | −0.0008 (9) | −0.0026 (9) | 0.0026 (9) |
| C17 | 0.029 (1) | 0.0244 (9) | 0.027 (1) | −0.0079 (9) | −0.0007 (9) | 0.0011 (8) |
| C18 | 0.0353 (12) | 0.0389 (12) | 0.0275 (11) | −0.005 (1) | −0.003 (1) | −0.0002 (9) |
| C19 | 0.0471 (15) | 0.0461 (14) | 0.0378 (13) | −0.0104 (12) | −0.0190 (12) | 0.0129 (11) |
| C20 | 0.0305 (12) | 0.0347 (12) | 0.0594 (15) | −0.005 (1) | −0.0136 (11) | 0.0123 (12) |
| C21 | 0.0293 (11) | 0.0308 (12) | 0.0518 (15) | 0.0004 (9) | −0.0043 (11) | 0.001 (1) |
| C22 | 0.027 (1) | 0.0280 (11) | 0.0350 (11) | 0.0002 (8) | −0.002 (1) | −0.0008 (9) |
| C23 | 0.0385 (12) | 0.026 (1) | 0.0347 (12) | −0.0003 (9) | 0.010 (1) | −0.0015 (9) |
| C24 | 0.0440 (14) | 0.0402 (14) | 0.0509 (15) | −0.0037 (12) | 0.0219 (12) | −0.0045 (12) |
| C25 | 0.0615 (18) | 0.0327 (12) | 0.0443 (15) | −0.0076 (12) | 0.0170 (13) | −0.0145 (11) |
| C26 | 0.0543 (16) | 0.0439 (15) | 0.0579 (17) | 0.0234 (13) | 0.0083 (13) | −0.0010 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.340 (3) | Si1—C17 | 1.871 (2) |
| O1—C4 | 1.458 (3) | Si1—C23 | 1.886 (2) |
| C1—C2 | 1.538 (3) | C11—C12 | 1.405 (3) |
| C1—O2 | 1.205 (3) | C11—C16 | 1.401 (3) |
| C2—C3 | 1.530 (3) | C12—C13 | 1.391 (3) |
| C2—C5 | 1.506 (3) | C12—H121 | 1.000 |
| C2—O6 | 1.414 (2) | C13—C14 | 1.381 (3) |
| C3—C4 | 1.538 (3) | C13—H131 | 1.000 |
| C3—O7 | 1.421 (2) | C14—C15 | 1.383 (3) |
| C3—H31 | 1.0000 | C14—H141 | 1.000 |
| C4—C10 | 1.506 (3) | C15—C16 | 1.395 (3) |
| C4—H41 | 1.000 | C15—H151 | 1.000 |
| C5—O3 | 1.474 (2) | C16—H161 | 1.000 |
| C5—H51 | 1.000 | C17—C18 | 1.399 (3) |
| C5—H52 | 1.000 | C17—C22 | 1.407 (3) |
| O3—S1 | 1.5563 (14) | C18—C19 | 1.400 (3) |
| S1—O4 | 1.4146 (18) | C18—H181 | 1.000 |
| S1—O5 | 1.4166 (18) | C19—C20 | 1.379 (4) |
| S1—C6 | 1.824 (3) | C19—H191 | 1.000 |
| C6—F1 | 1.310 (3) | C20—C21 | 1.378 (4) |
| C6—F2 | 1.319 (3) | C20—H201 | 1.000 |
| C6—F3 | 1.322 (3) | C21—C22 | 1.387 (3) |
| O6—C7 | 1.436 (2) | C21—H211 | 1.000 |
| O7—C7 | 1.435 (2) | C22—H221 | 1.000 |
| C7—C8 | 1.505 (3) | C23—C24 | 1.536 (3) |
| C7—C9 | 1.516 (3) | C23—C25 | 1.539 (3) |
| C8—H81 | 1.000 | C23—C26 | 1.539 (3) |
| C8—H82 | 1.000 | C24—H241 | 1.000 |
| C8—H83 | 1.000 | C24—H242 | 1.000 |

| | | | |
|---|---|---|---|
| C9—H91 | 1.000 | C24—H243 | 1.000 |
| C9—H92 | 1.000 | C25—H251 | 1.000 |
| C9—H93 | 1.000 | C25—H252 | 1.000 |
| C10—O8 | 1.428 (2) | C25—H253 | 1.000 |
| C10—H101 | 1.000 | C26—H261 | 1.000 |
| C10—H102 | 1.000 | C26—H262 | 1.000 |
| O8—Si1 | 1.6648 (14) | C26—H263 | 1.000 |
| Si1—C11 | 1.872 (2) | | |

| | | | |
|---|---|---|---|
| C1—O1—C4 | 112.00 (15) | C10—O8—Si1 | 123.29 (12) |
| O1—C1—C2 | 110.79 (18) | O8—Si1—C11 | 103.27 (8) |
| O1—C1—O2 | 122.78 (19) | O8—Si1—C17 | 108.29 (9) |
| C2—C1—O2 | 126.4 (2) | C11—Si1—C17 | 107.38 (9) |
| C1—C2—C3 | 104.29 (16) | O8—Si1—C23 | 110.57 (9) |
| C1—C2—C5 | 107.68 (16) | C11—Si1—C23 | 111.7 (1) |
| C3—C2—C5 | 118.56 (17) | C17—Si1—C23 | 114.9 (1) |
| C1—C2—O6 | 111.69 (16) | Si1—C11—C12 | 120.62 (15) |
| C3—C2—O6 | 105.43 (15) | Si1—C11—C16 | 121.83 (15) |
| C5—C2—O6 | 109.16 (16) | C12—C11—C16 | 117.55 (19) |
| C2—C3—C4 | 105.13 (16) | C11—C12—C13 | 121.3 (2) |
| C2—C3—O7 | 104.81 (15) | C11—C12—H121 | 119.4 |
| C4—C3—O7 | 114.04 (16) | C13—C12—H121 | 119.4 |
| C2—C3—H31 | 116.77 | C12—C13—C14 | 119.9 (2) |
| C4—C3—H31 | 107.99 | C12—C13—H131 | 120.1 |
| O7—C3—H31 | 108.30 | C14—C13—H131 | 120.1 |
| O1—C4—C3 | 106.67 (16) | C13—C14—C15 | 120.3 (2) |
| O1—C4—C10 | 107.98 (17) | C13—C14—H141 | 119.8 |
| C3—C4—C10 | 113.14 (16) | C15—C14—H141 | 119.8 |
| O1—C4—H41 | 113.58 | C14—C15—C16 | 119.9 (2) |
| C3—C4—H41 | 108.45 | C14—C15—H151 | 120.1 |
| C10—C4—H41 | 107.16 | C16—C15—H151 | 120.1 |
| C2—C5—O3 | 106.94 (15) | C11—C16—C15 | 121.12 (19) |
| C2—C5—H51 | 110.10 | C11—C16—H161 | 119.4 |
| O3—C5—H51 | 110.10 | C15—C16—H161 | 119.4 |
| C2—C5—H52 | 110.10 | Si1—C17—C18 | 126.14 (18) |
| O3—C5—H52 | 110.10 | Si1—C17—C22 | 116.46 (16) |
| H51—C5—H52 | 109.47 | C18—C17—C22 | 117.1 (2) |
| C5—O3—S1 | 120.09 (13) | C17—C18—C19 | 120.7 (2) |
| O3—S1—O4 | 107.4 (1) | C17—C18—H181 | 119.6 |
| O3—S1—O5 | 111.51 (9) | C19—C18—H181 | 119.6 |
| O4—S1—O5 | 122.81 (12) | C18—C19—C20 | 120.6 (2) |
| O3—S1—C6 | 99.7 (1) | C18—C19—H191 | 119.7 |
| O4—S1—C6 | 106.06 (12) | C20—C19—H191 | 119.7 |
| O5—S1—C6 | 106.70 (11) | C19—C20—C21 | 119.7 (2) |
| S1—C6—F1 | 110.25 (17) | C19—C20—H201 | 120.2 |
| S1—C6—F2 | 110.61 (16) | C21—C20—H201 | 120.2 |
| F1—C6—F2 | 109.1 (2) | C20—C21—C22 | 120.1 (2) |
| S1—C6—F3 | 108.90 (18) | C20—C21—H211 | 120.0 |

| | | | |
|---|---|---|---|
| F1—C6—F3 | 109.4 (2) | C22—C21—H211 | 120.0 |
| F2—C6—F3 | 108.6 (2) | C17—C22—C21 | 121.7 (2) |
| C2—O6—C7 | 108.58 (14) | C17—C22—H221 | 119.1 |
| C3—O7—C7 | 109.59 (14) | C21—C22—H221 | 119.1 |
| O6—C7—O7 | 104.67 (14) | Si1—C23—C24 | 109.42 (15) |
| O6—C7—C8 | 108.21 (16) | Si1—C23—C25 | 113.07 (16) |
| O7—C7—C8 | 109.10 (17) | C24—C23—C25 | 109.1 (2) |
| O6—C7—C9 | 111.66 (17) | Si1—C23—C26 | 108.83 (16) |
| O7—C7—C9 | 109.92 (16) | C24—C23—C26 | 107.7 (2) |
| C8—C7—C9 | 112.92 (17) | C25—C23—C26 | 108.6 (2) |
| C7—C8—H81 | 109.5 | C23—C24—H241 | 109.5 |
| C7—C8—H82 | 109.5 | C23—C24—H242 | 109.5 |
| H81—C8—H82 | 109.5 | H241—C24—H242 | 109.5 |
| C7—C8—H83 | 109.47 | C23—C24—H243 | 109.5 |
| H81—C8—H83 | 109.5 | H241—C24—H243 | 109.5 |
| H82—C8—H83 | 109.5 | H242—C24—H243 | 109.5 |
| C7—C9—H91 | 109.5 | C23—C25—H251 | 109.5 |
| C7—C9—H92 | 109.47 | C23—C25—H252 | 109.5 |
| H91—C9—H92 | 109.5 | H251—C25—H252 | 109.5 |
| C7—C9—H93 | 109.47 | C23—C25—H253 | 109.5 |
| H91—C9—H93 | 109.5 | H251—C25—H253 | 109.5 |
| H92—C9—H93 | 109.5 | H252—C25—H253 | 109.5 |
| C4—C10—O8 | 108.39 (16) | C23—C26—H261 | 109.5 |
| C4—C10—H101 | 109.74 | C23—C26—H262 | 109.5 |
| O8—C10—H101 | 109.74 | H261—C26—H262 | 109.5 |
| C4—C10—H102 | 109.74 | C23—C26—H263 | 109.5 |
| O8—C10—H102 | 109.74 | H261—C26—H263 | 109.5 |
| H101—C10—H102 | 109.47 | H262—C26—H263 | 109.5 |