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Key indicators

Single-crystal X-ray study
 $T = 190$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
Disorder in main residue
 R factor = 0.085
 wR factor = 0.119
Data-to-parameter ratio = 12.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Isopropyl 2,5-anhydro-4-(2,5-anhydro-4-azido-3-*O*-*tert*-butyldiphenylsilyl-4-deoxy- β -D-ribofuranosylamino)-3-*O*-*tert*-butyldiphenylsilyl-4-deoxy- β -D-ribofuranosylateThe crystal structure of the title compound, $\text{C}_{45}\text{H}_{56}\text{N}_4\text{O}_7\text{Si}_2$, shows a γ -turn conformation which is stabilized by an intramolecular hydrogen bond.

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Comment

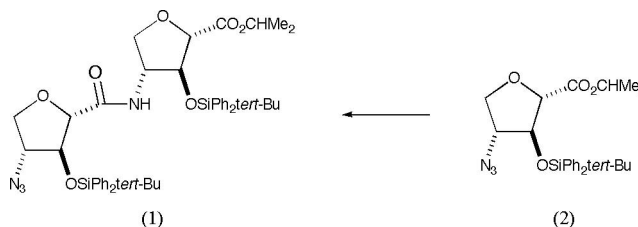
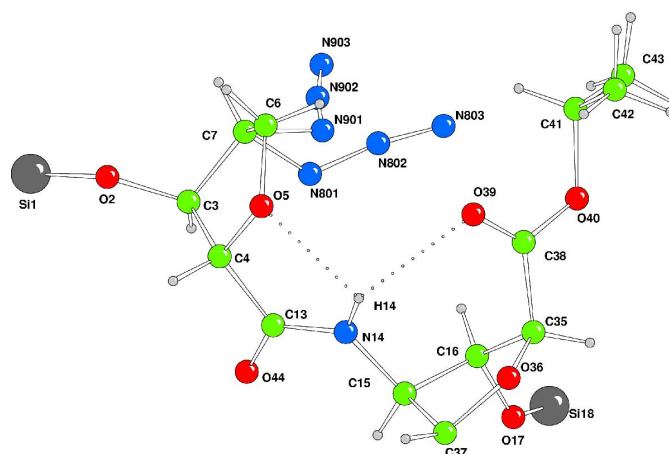
Tetrahydrofuran (THF)-derived sugar amino acids (SAA) have been extensively investigated as dipeptide isosteres (Chakraborty *et al.*, 2004; Grotenberg *et al.*, 2004). A multitude of peptidomimetics, including a number of δ -THF SAA scaffolds, induce β -turn-like structures (Smith *et al.*, 2003). However, there are relatively few examples of γ -turn conformations (Etzkorn *et al.*, 1999; Lindvall *et al.*, 1999; Belvisi *et al.*, 1999). In contrast to the extensive studies on β -peptides built from residues containing five- or six-membered rings (Wang *et al.*, 2000), there are only limited reports of γ -peptides based on cyclic templates (Curran *et al.*, 1996; Crisma *et al.*, 2001; Goswami & Moloney, 1999).This paper reports the structure of the γ -THF SAA compound, (1). The γ -turn conformation (Fig. 1) is stabilized by bifurcated intramolecular $\text{N14}-\text{H14}\cdots\text{O39}$ and $\text{N14}-\text{H14}\cdots\text{O5}$ hydrogen bonds. There is no intermolecular

Figure 1

The central section of the molecule, showing the γ -turn stabilized by the bifurcated internal hydrogen bond (dashed lines).

hydrogen bonding, thus leading to an open structure (calculated density = 1.219 Mg m⁻³) with a substantial opportunity for disorder and large atomic displacements (Fig. 2).

Experimental

Compound (1) was prepared by conventional peptide coupling procedures from the dipeptidomimetic compound (2) (Sanjayan *et al.*, 2003), and was crystallized from methanol.

Crystal data

C ₄₅ H ₅₆ N ₄ O ₇ Si ₂	$D_x = 1.219 \text{ Mg m}^{-3}$
$M_r = 821.13$	Mo K α radiation
Monoclinic, $P2_1$	Cell parameters from 4658 reflections
$a = 15.4548 (4) \text{ \AA}$	$\theta = 5\text{--}30^\circ$
$b = 9.0111 (2) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$c = 16.4767 (5) \text{ \AA}$	$T = 190 \text{ K}$
$\beta = 102.7868 (10)^\circ$	Prism, colourless
$V = 2237.72 (10) \text{ \AA}^3$	$0.80 \times 0.30 \times 0.20 \text{ mm}$
$Z = 2$	

Data collection

Nonius KappaCCD diffractometer	6788 independent reflections
ω scans	6788 reflections with $I > -3\sigma(I)$
Absorption correction: multi-scan (<i>DENZO/SCALEPACK</i> ;	$R_{\text{int}} = 0.057$
Otwinowski & Minor, 1997)	$\theta_{\text{max}} = 30.0^\circ$
$T_{\text{min}} = 0.96$, $T_{\text{max}} = 0.97$	$h = -21 \rightarrow 21$
23431 measured reflections	$k = -12 \rightarrow 8$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.085$	$w = 1/[\sigma^2(F^2) + 0.03 + 1.13P]$
$wR(F^2) = 0.119$	where $P = [\max(F_o^2, 0) + 2F_c^2]/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} = 0.001$
6788 reflections	$\Delta\rho_{\text{max}} = 0.57 \text{ e \AA}^{-3}$
550 parameters	$\Delta\rho_{\text{min}} = -0.48 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N14—H14 \cdots O39	0.84	2.39	3.057 (5)	137
N14—H14 \cdots O5	0.84	2.21	2.624 (5)	110

The disordered azide group could only be refined satisfactorily with distance and anisotropic displacement parameter (adp) similarity restraints. The small angle C3—C7—N801 [$93.1 (4)^\circ$] and the large angle C3—C7—N901 [$123.7 (5)^\circ$] suggests that the disorder probably extends into the ring system, but is accommodated by the adps. Atom H71 should also be represented by two partial atoms, but they could not be resolved. The other H atoms were all located in a difference map. Those attached to C atoms were repositioned geometrically. All H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H = 0.93–0.98, O—N = 0.86–0.89 and O—H = 0.82 \AA) and U_{iso} values (in the range 1.2–1.5 times U_{eq} of the parent atom), after which they were refined with riding constraints. The large adps in the phenyl groups are consistent with rigid-body librations (R_{TLS} in the range 2.5–5.0%). The large adps in the *tert*-butyl groups are not amenable to TLS analysis, but look consistent with simple libration. Both the large displacement parameters and the disorder in the azide are not unexpected, because there are no intermolecular hydrogen bonds to consolidate the crystal packing.

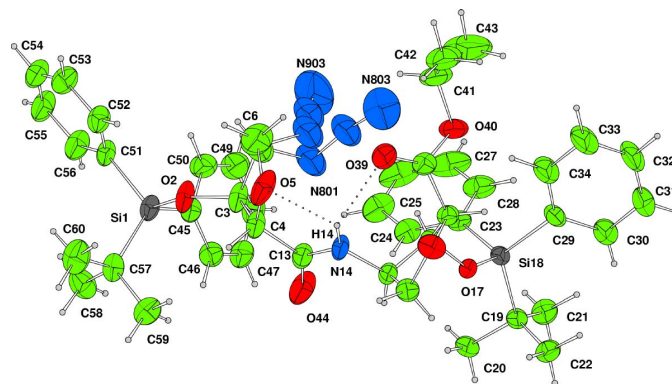


Figure 2

The complete molecule with displacement ellipsoids drawn at the 50% probability level. The cavity containing the disordered azide is evident, as are the large displacements of the atoms in the periphery. Some labels have been omitted for clarity.

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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