Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isopropyl 2,5-anhydro-4-(2,5-anhydro-4-azido-3-*O*-*tert*-butyldiphenylsilyl-4-deoxy-L-ribonyl-amino)-3-*O*-*tert*-butyldiphenylsilyl-4-deoxy-L-ribonate

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.085
wR factor = 0.119
Data-to-parameter ratio = 12.3

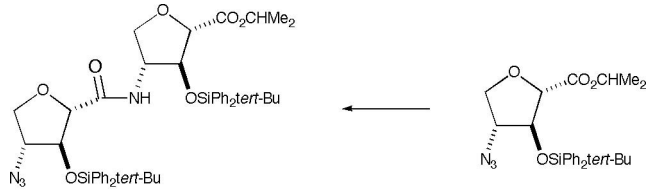
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $C_{45}H_{56}N_4O_7Si_2$, shows a γ-turn conformation which is stabilized by an intramolecular hydrogen bond.

Received 24 February 2005
Accepted 3 March 2005
Online 11 March 2005

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).



(1) (2)

This paper reports the structure of the γ-THF SAA compound, (1). The γ-turn conformation (Fig. 1) is stabilized by bifurcated intramolecular N14—H14···O39 and N14—H14···O5 hydrogen bonds. There is no intermolecular

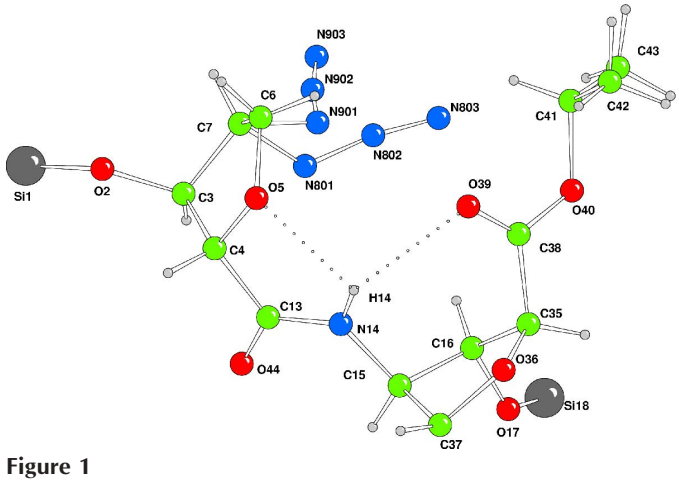


Figure 1
The central section of the molecule, showing the γ-turn stabilized by the bifurcated internal hydrogen bond (dashed lines.

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

$C_{45}H_{56}N_4O_7Si_2$
M_r = 821.13
Monoclinic, $P2_1$
a = 15.4548 (4) Å
b = 9.0111 (2) Å
c = 16.4767 (5) Å
β = 102.7868 (10)°
V = 2237.72 (10) Å^3
Z = 2
D_x = 1.219 Mg m^{-3}
Mo Kα radiation
Cell parameters from 4658 reflections
θ = 5–30°
μ = 0.13 mm^{-1}
T = 190 K
Prism, colourless
0.80 × 0.30 × 0.20 mm

Data collection

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

Refinement

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N14—H14···O39	0.84	2.39	3.057 (5)	137
N14—H14···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)°] and the large angle C3—C7—N901 [123.7 (5)°] 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 Å) 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.

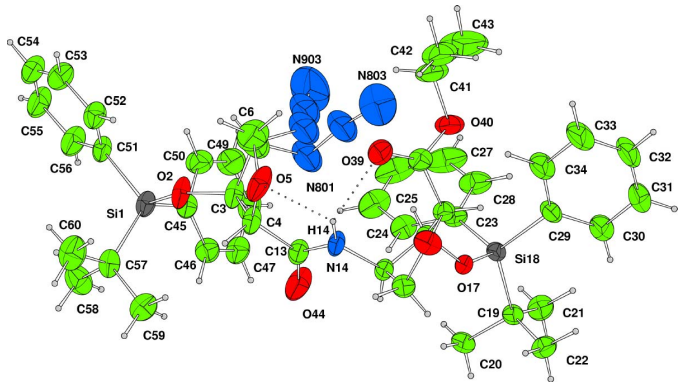


Figure 2
The complete molecule with displacement ellipsoids drawn at the 50% probability level. The cavity containing the disordered azide is evident, as are the large displacements of the atoms in the periphery. Some labels have been omitted for clarity.

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*.

Financial support to AAE for a post-doctoral fellowship from EPSRC (grant No. GR/S44105/01) and to GJS from DST, New Delhi, for a BOYCAST Fellowship, is gratefully acknowledged.

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supporting information

Acta Cryst. (2005). E**61**, o918–o919 [https://doi.org/10.1107/S1600536805006689]

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$Z = 2$
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$D_x = 1.219$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4658 reflections
$\theta = 5$–30°
$\mu = 0.13$ mm^{-1}
$T = 190$ K
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$\theta_{max} = 30.0°$, $\theta_{min} = 5.1°$
$h = -21 \rightarrow 21$
$k = -12 \rightarrow 8$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.085$
$wR(F^2) = 0.119$
$S = 0.99$
6788 reflections
550 parameters
44 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + 0.03 + 1.13P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Si1	0.14482 (6)	0.36845 (13)	0.25970 (5)	0.0362	

Si18	0.28747 (5)	0.47285 (12)	0.84360 (5)	0.0307
C3	0.2651 (2)	0.4364 (5)	0.40455 (19)	0.0467
C4	0.3032 (2)	0.5867 (5)	0.4362 (2)	0.0430
C6	0.4197 (3)	0.4315 (8)	0.4285 (3)	0.0948
C7	0.3418 (4)	0.3372 (7)	0.4321 (3)	0.0865
C13	0.2785 (2)	0.6316 (5)	0.5169 (2)	0.0446
C15	0.3333 (2)	0.7119 (4)	0.66015 (18)	0.0336
C16	0.36860 (18)	0.5938 (3)	0.72532 (17)	0.0289
C19	0.1894 (2)	0.5551 (4)	0.87813 (19)	0.0349
C20	0.1216 (2)	0.6032 (5)	0.7992 (2)	0.0490
C21	0.1462 (2)	0.4374 (5)	0.9232 (2)	0.0478
C22	0.2133 (2)	0.6925 (4)	0.9332 (2)	0.0457
C23	0.2496 (2)	0.3135 (4)	0.7724 (2)	0.0365
C24	0.2086 (2)	0.3349 (4)	0.6880 (2)	0.0454
C25	0.1747 (3)	0.2164 (5)	0.6373 (3)	0.0627
C26	0.1804 (3)	0.0739 (6)	0.6688 (4)	0.0761
C27	0.2187 (3)	0.0508 (5)	0.7508 (4)	0.0759
C28	0.2530 (3)	0.1678 (4)	0.8019 (3)	0.0576
C29	0.3823 (2)	0.4158 (4)	0.9299 (2)	0.0415
C30	0.3861 (3)	0.4411 (5)	1.0152 (2)	0.0595
C31	0.4627 (3)	0.4083 (6)	1.0747 (3)	0.0714
C32	0.5356 (3)	0.3520 (6)	1.0510 (3)	0.0677
C33	0.5317 (3)	0.3236 (8)	0.9700 (3)	0.0955
C34	0.4561 (3)	0.3576 (7)	0.9103 (3)	0.0764
C35	0.4674 (2)	0.6379 (4)	0.7527 (2)	0.0349
C37	0.3888 (2)	0.8455 (4)	0.6941 (2)	0.0433
C38	0.5238 (2)	0.5435 (4)	0.7081 (2)	0.0391
C41	0.6082 (3)	0.3190 (5)	0.7176 (3)	0.0688
C42	0.7008 (3)	0.3848 (7)	0.7285 (3)	0.0835
C43	0.6078 (5)	0.1813 (7)	0.7669 (5)	0.1347
C45	0.0883 (2)	0.2542 (4)	0.3279 (2)	0.0371
C46	0.0226 (2)	0.3088 (4)	0.3664 (2)	0.0442
C47	−0.0166 (2)	0.2214 (5)	0.4169 (2)	0.0461
C48	0.0097 (3)	0.0756 (5)	0.4313 (2)	0.0504
C49	0.0742 (3)	0.0193 (5)	0.3963 (3)	0.0606
C50	0.1126 (3)	0.1068 (5)	0.3437 (2)	0.0526
C51	0.1797 (2)	0.2446 (4)	0.18129 (19)	0.0401
C52	0.2645 (2)	0.2492 (5)	0.1659 (2)	0.0462
C53	0.2874 (3)	0.1602 (5)	0.1050 (2)	0.0587
C54	0.2272 (3)	0.0654 (5)	0.0587 (2)	0.0628
C55	0.1428 (3)	0.0575 (6)	0.0725 (2)	0.0629
C56	0.1200 (3)	0.1459 (5)	0.1330 (2)	0.0562
C57	0.0774 (2)	0.5299 (4)	0.2059 (2)	0.0433
C58	−0.0142 (3)	0.4788 (5)	0.1571 (3)	0.0600
C59	0.0653 (3)	0.6531 (5)	0.2669 (2)	0.0562
C60	0.1279 (3)	0.5981 (5)	0.1436 (3)	0.0672
O2	0.23825 (14)	0.4387 (3)	0.31647 (13)	0.0472
O5	0.39594 (17)	0.5793 (4)	0.44477 (16)	0.0661

O17	0.32402 (13)	0.6086 (2)	0.79167 (12)	0.0306	
O36	0.47512 (16)	0.7875 (3)	0.72919 (16)	0.0468	
O39	0.53825 (16)	0.5705 (3)	0.64113 (15)	0.0547	
O40	0.55231 (18)	0.4244 (3)	0.75264 (17)	0.0572	
O44	0.20056 (16)	0.6364 (5)	0.52043 (17)	0.0807	
N14	0.34587 (18)	0.6699 (3)	0.57860 (15)	0.0389	
N801	0.3204 (6)	0.3119 (13)	0.5206 (5)	0.0860	0.4308
N802	0.3745 (7)	0.2480 (12)	0.5756 (6)	0.0846	0.4321
N803	0.4213 (7)	0.2048 (14)	0.6321 (7)	0.1022	0.4321
N901	0.3658 (5)	0.2755 (9)	0.5127 (4)	0.0871	0.5692
N902	0.3353 (5)	0.1546 (9)	0.5120 (5)	0.0871	0.5679
N903	0.3137 (10)	0.0369 (10)	0.5186 (7)	0.1484	0.5679
H14	0.3979	0.6662	0.5705	0.0567*	
H31	0.2156	0.4073	0.4288	0.0666*	
H41	0.2805	0.6638	0.3945	0.0610*	
H61	0.4713	0.4017	0.4697	0.1317*	
H62	0.4323	0.4229	0.3736	0.1319*	
H71	0.3373	0.2510	0.3954	0.1191*	
H151	0.2710	0.7334	0.6569	0.0473*	
H161	0.3617	0.4943	0.7021	0.0408*	
H201	0.0704	0.6479	0.8153	0.0851*	
H202	0.1478	0.6748	0.7672	0.0853*	
H203	0.1016	0.5166	0.7650	0.0859*	
H211	0.0944	0.4798	0.9381	0.0897*	
H212	0.1877	0.4061	0.9743	0.0894*	
H213	0.1284	0.3527	0.8884	0.0898*	
H221	0.1607	0.7317	0.9477	0.0816*	
H222	0.2555	0.6685	0.9835	0.0818*	
H223	0.2376	0.7701	0.9029	0.0812*	
H241	0.2047	0.4306	0.6659	0.0643*	
H251	0.1484	0.2331	0.5815	0.0886*	
H261	0.1583	−0.0070	0.6345	0.1158*	
H271	0.2207	−0.0460	0.7715	0.1167*	
H281	0.2784	0.1516	0.8574	0.0871*	
H301	0.3350	0.4798	1.0327	0.0857*	
H311	0.4638	0.4238	1.1332	0.0998*	
H321	0.5882	0.3324	1.0900	0.0947*	
H331	0.5817	0.2813	0.9535	0.1379*	
H341	0.4551	0.3396	0.8523	0.1103*	
H351	0.4878	0.6242	0.8124	0.0492*	
H371	0.3643	0.8964	0.7368	0.0617*	
H372	0.3917	0.9152	0.6489	0.0621*	
H411	0.5820	0.3007	0.6581	0.1025*	
H421	0.7387	0.3125	0.7081	0.1486*	
H422	0.7237	0.4034	0.7882	0.1490*	
H423	0.6990	0.4780	0.6973	0.1492*	
H431	0.6491	0.1108	0.7523	0.2648*	
H432	0.6234	0.2051	0.8262	0.2649*	

H433	0.5473	0.1415	0.7523	0.2648*
H461	0.0048	0.4084	0.3561	0.0645*
H471	−0.0600	0.2605	0.4414	0.0677*
H481	−0.0176	0.0162	0.4647	0.0749*
H491	0.0917	−0.0796	0.4055	0.0899*
H501	0.1562	0.0655	0.3196	0.0781*
H521	0.3068	0.3167	0.1961	0.0669*
H531	0.3451	0.1673	0.0961	0.0887*
H541	0.2435	0.0030	0.0176	0.0914*
H551	0.1011	−0.0076	0.0423	0.0898*
H561	0.0628	0.1369	0.1429	0.0810*
H581	−0.0436	0.5629	0.1266	0.1018*
H582	−0.0492	0.4426	0.1953	0.1016*
H583	−0.0064	0.4024	0.1172	0.1012*
H591	0.0374	0.7392	0.2382	0.1018*
H592	0.1215	0.6831	0.3018	0.1015*
H593	0.0290	0.6155	0.3020	0.1015*
H601	0.0962	0.6844	0.1176	0.1247*
H602	0.1862	0.6269	0.1726	0.1244*
H603	0.1321	0.5240	0.1025	0.1246*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0301 (4)	0.0519 (5)	0.0278 (4)	−0.0106 (4)	0.0094 (3)	−0.0050 (4)
Si18	0.0261 (4)	0.0375 (4)	0.0287 (4)	0.0061 (4)	0.0064 (3)	0.0041 (4)
C3	0.0413 (18)	0.074 (3)	0.0255 (16)	−0.0136 (18)	0.0085 (14)	−0.0082 (17)
C4	0.0309 (16)	0.072 (2)	0.0262 (16)	−0.0068 (18)	0.0072 (13)	−0.0059 (17)
C6	0.047 (3)	0.154 (6)	0.077 (3)	0.026 (3)	−0.001 (2)	−0.034 (4)
C7	0.086 (3)	0.088 (3)	0.071 (2)	0.021 (2)	−0.013 (2)	0.005 (2)
C13	0.0343 (17)	0.068 (2)	0.0309 (17)	0.0036 (18)	0.0071 (14)	−0.0052 (17)
C15	0.0327 (16)	0.0425 (17)	0.0262 (15)	0.0021 (14)	0.0077 (12)	−0.0025 (13)
C16	0.0273 (14)	0.0320 (15)	0.0273 (14)	0.0003 (13)	0.0062 (11)	−0.0046 (12)
C19	0.0257 (14)	0.0460 (18)	0.0335 (16)	0.0061 (14)	0.0076 (12)	0.0022 (14)
C20	0.0342 (17)	0.066 (2)	0.0439 (19)	0.0204 (18)	0.0034 (15)	0.0020 (19)
C21	0.0384 (18)	0.060 (2)	0.050 (2)	−0.0010 (17)	0.0196 (16)	0.0018 (18)
C22	0.0429 (19)	0.051 (2)	0.046 (2)	0.0071 (17)	0.0144 (16)	−0.0057 (17)
C23	0.0319 (16)	0.0374 (16)	0.0434 (19)	0.0022 (14)	0.0154 (14)	−0.0011 (14)
C24	0.0378 (18)	0.047 (2)	0.052 (2)	−0.0059 (16)	0.0108 (16)	−0.0085 (17)
C25	0.042 (2)	0.071 (3)	0.074 (3)	−0.015 (2)	0.010 (2)	−0.028 (2)
C26	0.058 (3)	0.056 (3)	0.124 (5)	−0.018 (2)	0.041 (3)	−0.036 (3)
C27	0.076 (3)	0.034 (2)	0.133 (5)	−0.008 (2)	0.056 (3)	−0.007 (3)
C28	0.061 (3)	0.042 (2)	0.080 (3)	0.0063 (19)	0.038 (2)	0.008 (2)
C29	0.0333 (17)	0.054 (2)	0.0372 (18)	0.0125 (15)	0.0083 (13)	0.0156 (16)
C30	0.049 (2)	0.081 (3)	0.046 (2)	0.019 (2)	0.0052 (17)	0.005 (2)
C31	0.066 (3)	0.100 (4)	0.041 (2)	0.025 (3)	−0.0049 (19)	0.007 (2)
C32	0.042 (2)	0.108 (4)	0.047 (2)	0.021 (3)	−0.0032 (17)	0.023 (3)
C33	0.060 (3)	0.161 (6)	0.062 (3)	0.062 (4)	0.005 (2)	0.021 (3)

C34	0.054 (2)	0.123 (4)	0.051 (2)	0.043 (3)	0.010 (2)	0.017 (3)
C35	0.0313 (16)	0.0374 (17)	0.0359 (17)	0.0029 (14)	0.0072 (13)	−0.0033 (14)
C37	0.052 (2)	0.0348 (18)	0.0410 (19)	0.0035 (17)	0.0068 (16)	−0.0013 (15)
C38	0.0259 (15)	0.0484 (19)	0.044 (2)	−0.0010 (15)	0.0091 (14)	−0.0041 (16)
C41	0.078 (3)	0.059 (3)	0.080 (3)	0.031 (2)	0.041 (3)	0.002 (2)
C42	0.060 (3)	0.098 (4)	0.094 (4)	0.030 (3)	0.020 (2)	−0.023 (3)
C43	0.183 (7)	0.078 (4)	0.180 (7)	0.063 (5)	0.118 (6)	0.047 (5)
C45	0.0295 (16)	0.050 (2)	0.0320 (17)	−0.0049 (15)	0.0076 (13)	−0.0029 (15)
C46	0.0388 (18)	0.052 (2)	0.0434 (19)	−0.0007 (16)	0.0131 (15)	0.0021 (16)
C47	0.0363 (18)	0.064 (2)	0.042 (2)	−0.0079 (18)	0.0167 (15)	−0.0026 (18)
C48	0.056 (2)	0.059 (2)	0.0397 (19)	−0.017 (2)	0.0175 (17)	0.0032 (18)
C49	0.079 (3)	0.047 (2)	0.061 (3)	0.005 (2)	0.029 (2)	0.0083 (19)
C50	0.052 (2)	0.056 (2)	0.055 (2)	0.009 (2)	0.0238 (18)	0.0039 (19)
C51	0.0392 (18)	0.056 (2)	0.0262 (16)	−0.0044 (17)	0.0105 (13)	−0.0029 (15)
C52	0.0410 (19)	0.062 (2)	0.0381 (19)	−0.0062 (18)	0.0140 (15)	−0.0016 (17)
C53	0.056 (2)	0.078 (3)	0.051 (2)	0.006 (2)	0.0302 (19)	0.001 (2)
C54	0.089 (3)	0.063 (3)	0.040 (2)	0.009 (3)	0.023 (2)	−0.007 (2)
C55	0.070 (3)	0.077 (3)	0.041 (2)	−0.006 (2)	0.0107 (19)	−0.020 (2)
C56	0.049 (2)	0.077 (3)	0.043 (2)	−0.009 (2)	0.0116 (17)	−0.016 (2)
C57	0.0455 (19)	0.050 (2)	0.0340 (17)	−0.0123 (17)	0.0081 (15)	−0.0013 (16)
C58	0.058 (2)	0.053 (2)	0.059 (2)	−0.005 (2)	−0.0074 (19)	0.002 (2)
C59	0.062 (2)	0.054 (2)	0.055 (2)	−0.004 (2)	0.017 (2)	−0.009 (2)
C60	0.086 (3)	0.066 (3)	0.056 (3)	−0.019 (3)	0.029 (2)	0.007 (2)
O2	0.0366 (12)	0.0813 (19)	0.0247 (11)	−0.0217 (13)	0.0086 (9)	−0.0097 (12)
O5	0.0412 (14)	0.119 (3)	0.0437 (15)	−0.0256 (17)	0.0210 (12)	−0.0300 (17)
O17	0.0340 (11)	0.0350 (11)	0.0245 (10)	0.0047 (9)	0.0098 (8)	0.0008 (9)
O36	0.0402 (13)	0.0353 (12)	0.0605 (16)	−0.0061 (11)	0.0018 (11)	−0.0033 (11)
O39	0.0489 (14)	0.0752 (19)	0.0448 (14)	0.0122 (15)	0.0206 (12)	0.0038 (14)
O40	0.0635 (17)	0.0518 (15)	0.0650 (17)	0.0255 (14)	0.0327 (14)	0.0109 (13)
O44	0.0306 (13)	0.160 (4)	0.0494 (16)	0.0069 (18)	0.0052 (11)	−0.038 (2)
N14	0.0339 (14)	0.0620 (18)	0.0219 (13)	−0.0014 (13)	0.0082 (11)	−0.0049 (12)
N801	0.085 (3)	0.087 (3)	0.071 (2)	0.020 (2)	−0.013 (2)	0.007 (2)
N802	0.084 (3)	0.086 (3)	0.070 (2)	0.020 (3)	−0.012 (2)	0.008 (2)
N803	0.085 (7)	0.119 (10)	0.106 (8)	−0.027 (7)	0.028 (6)	0.016 (8)
N901	0.087 (3)	0.089 (3)	0.071 (2)	0.021 (2)	−0.013 (2)	0.006 (2)
N902	0.087 (3)	0.089 (3)	0.071 (2)	0.021 (2)	−0.013 (2)	0.006 (2)
N903	0.245 (15)	0.071 (6)	0.118 (8)	0.006 (8)	0.018 (8)	0.032 (6)

Geometric parameters (Å, º)

Si1—C45	1.876 (3)	C32—H321	0.934
Si1—C51	1.874 (3)	C33—C34	1.385 (6)
Si1—C57	1.892 (4)	C33—H331	0.953
Si1—O2	1.662 (2)	C34—H341	0.966
Si18—C19	1.884 (3)	C35—C38	1.520 (4)
Si18—C23	1.865 (3)	C35—O36	1.415 (4)
Si18—C29	1.874 (3)	C35—H351	0.972
Si18—O17	1.662 (2)	C37—O36	1.430 (4)

C3—C4	1.522 (5)	C37—H371	0.984
C3—C7	1.473 (6)	C37—H372	0.983
C3—O2	1.419 (4)	C38—O39	1.198 (4)
C3—H31	0.975	C38—O40	1.320 (4)
C4—C13	1.517 (4)	C41—C42	1.523 (7)
C4—O5	1.411 (4)	C41—C43	1.484 (7)
C4—H41	0.986	C41—O40	1.483 (4)
C6—C7	1.486 (8)	C41—H411	0.989
C6—O5	1.422 (7)	C42—H421	0.985
C6—H61	0.964	C42—H422	0.983
C6—H62	0.970	C42—H423	0.982
C7—N801	1.582 (8)	C43—H431	0.968
C7—H71	0.978	C43—H432	0.976
C7—N901	1.411 (7)	C43—H433	0.980
C7—H71	0.978	C45—C46	1.402 (5)
C13—O44	1.220 (4)	C45—C50	1.389 (5)
C13—N14	1.329 (4)	C46—C47	1.380 (5)
C15—C16	1.524 (4)	C46—H461	0.943
C15—C37	1.512 (5)	C47—C48	1.380 (6)
C15—N14	1.450 (4)	C47—H471	0.925
C15—H151	0.973	C48—C49	1.357 (6)
C16—C35	1.545 (4)	C48—H481	0.933
C16—O17	1.422 (3)	C49—C50	1.397 (5)
C16—H161	0.972	C49—H491	0.934
C19—C20	1.541 (4)	C50—H501	0.932
C19—C21	1.530 (5)	C51—C52	1.390 (4)
C19—C22	1.531 (5)	C51—C56	1.396 (5)
C20—H201	0.976	C52—C53	1.390 (5)
C20—H202	0.975	C52—H521	0.949
C20—H203	0.972	C53—C54	1.364 (6)
C21—H211	0.967	C53—H531	0.938
C21—H212	0.980	C54—C55	1.376 (6)
C21—H213	0.957	C54—H541	0.956
C22—H221	0.964	C55—C56	1.381 (6)
C22—H222	0.960	C55—H551	0.930
C22—H223	0.981	C56—H561	0.938
C23—C24	1.407 (5)	C57—C58	1.536 (5)
C23—C28	1.397 (5)	C57—C59	1.537 (5)
C24—C25	1.386 (5)	C57—C60	1.547 (5)
C24—H241	0.933	C58—H581	0.965
C25—C26	1.380 (7)	C58—H582	0.973
C25—H251	0.931	C58—H583	0.977
C26—C27	1.366 (7)	C59—H591	0.960
C26—H261	0.939	C59—H592	0.968
C27—C28	1.380 (6)	C59—H593	0.953
C27—H271	0.935	C60—H601	0.967
C28—H281	0.923	C60—H602	0.957
C29—C30	1.412 (5)	C60—H603	0.964

C29—C34	1.357 (5)	N14—H14	0.844
C30—C31	1.393 (5)	N801—N802	1.231 (8)
C30—H301	0.965	N802—N803	1.116 (8)
C31—C32	1.369 (6)	N901—N902	1.186 (8)
C31—H311	0.970	N902—N903	1.125 (8)
C32—C33	1.347 (6)		

C45—Si1—C51	109.19 (16)	C32—C33—C34	120.5 (4)
C45—Si1—C57	114.94 (15)	C32—C33—H331	119.8
C51—Si1—C57	110.32 (15)	C34—C33—H331	119.7
C45—Si1—O2	109.50 (13)	C33—C34—C29	122.5 (4)
C51—Si1—O2	105.56 (13)	C33—C34—H341	119.4
C57—Si1—O2	106.89 (15)	C29—C34—H341	118.1
C19—Si18—C23	109.35 (15)	C16—C35—C38	110.5 (3)
C19—Si18—C29	115.16 (14)	C16—C35—O36	107.6 (3)
C23—Si18—C29	110.50 (16)	C38—C35—O36	108.0 (3)
C19—Si18—O17	104.69 (13)	C16—C35—H351	110.0
C23—Si18—O17	109.76 (13)	C38—C35—H351	109.1
C29—Si18—O17	107.13 (13)	O36—C35—H351	111.7
C4—C3—C7	102.2 (3)	C15—C37—O36	105.1 (3)
C4—C3—O2	109.9 (3)	C15—C37—H371	111.0
C7—C3—O2	110.9 (3)	O36—C37—H371	110.7
C4—C3—H31	112.2	C15—C37—H372	110.2
C7—C3—H31	111.2	O36—C37—H372	110.3
O2—C3—H31	110.1	H371—C37—H372	109.5
C3—C4—C13	112.6 (3)	C35—C38—O39	125.1 (3)
C3—C4—O5	107.4 (3)	C35—C38—O40	110.0 (3)
C13—C4—O5	111.8 (3)	O39—C38—O40	124.8 (3)
C3—C4—H41	109.7	C42—C41—C43	112.3 (5)
C13—C4—H41	107.7	C42—C41—O40	108.5 (4)
O5—C4—H41	107.4	C43—C41—O40	104.5 (3)
C7—C6—O5	106.4 (4)	C42—C41—H411	110.4
C7—C6—H61	111.4	C43—C41—H411	110.7
O5—C6—H61	109.7	O40—C41—H411	110.3
C7—C6—H62	108.8	C41—C42—H421	108.5
O5—C6—H62	111.3	C41—C42—H422	107.8
H61—C6—H62	109.2	H421—C42—H422	110.1
C6—C7—C3	104.2 (5)	C41—C42—H423	110.8
C6—C7—N801	117.1 (6)	H421—C42—H423	109.7
C3—C7—N801	93.1 (4)	H422—C42—H423	109.9
C6—C7—H71	112.2	C41—C43—H431	109.3
C3—C7—H71	110.5	C41—C43—H432	109.5
N801—C7—H71	117.0	H431—C43—H432	111.3
C6—C7—C3	104.2 (5)	C41—C43—H433	106.9
C6—C7—N901	102.5 (5)	H431—C43—H433	110.1
C3—C7—N901	123.7 (5)	H432—C43—H433	109.8
C6—C7—H71	112.2	Si1—C45—C46	124.0 (3)
C3—C7—H71	110.5	Si1—C45—C50	119.7 (3)

N901—C7—H71	103.6	C46—C45—C50	116.3 (3)
C4—C13—O44	119.6 (3)	C45—C46—C47	122.1 (4)
C4—C13—N14	115.7 (3)	C45—C46—H461	117.7
O44—C13—N14	124.7 (3)	C47—C46—H461	120.1
C16—C15—C37	102.1 (2)	C46—C47—C48	119.7 (3)
C16—C15—N14	111.7 (3)	C46—C47—H471	120.1
C37—C15—N14	111.7 (3)	C48—C47—H471	120.2
C16—C15—H151	112.0	C47—C48—C49	120.0 (3)
C37—C15—H151	109.6	C47—C48—H481	119.6
N14—C15—H151	109.6	C49—C48—H481	120.5
C15—C16—C35	101.7 (2)	C48—C49—C50	120.4 (4)
C15—C16—O17	108.5 (2)	C48—C49—H491	120.0
C35—C16—O17	111.5 (2)	C50—C49—H491	119.5
C15—C16—H161	112.1	C49—C50—C45	121.5 (4)
C35—C16—H161	111.6	C49—C50—H501	119.0
O17—C16—H161	111.1	C45—C50—H501	119.5
Si18—C19—C20	107.3 (2)	Si1—C51—C52	122.3 (3)
Si18—C19—C21	109.9 (2)	Si1—C51—C56	121.2 (3)
C20—C19—C21	108.4 (3)	C52—C51—C56	116.4 (3)
Si18—C19—C22	113.1 (2)	C51—C52—C53	121.0 (4)
C20—C19—C22	107.6 (3)	C51—C52—H521	119.3
C21—C19—C22	110.3 (3)	C53—C52—H521	119.6
C19—C20—H201	109.1	C52—C53—C54	121.0 (4)
C19—C20—H202	110.9	C52—C53—H531	118.4
H201—C20—H202	109.4	C54—C53—H531	120.7
C19—C20—H203	109.5	C53—C54—C55	119.6 (4)
H201—C20—H203	108.4	C53—C54—H541	120.8
H202—C20—H203	109.7	C55—C54—H541	119.6
C19—C21—H211	109.0	C54—C55—C56	119.5 (4)
C19—C21—H212	110.1	C54—C55—H551	120.9
H211—C21—H212	108.4	C56—C55—H551	119.6
C19—C21—H213	111.2	C51—C56—C55	122.5 (4)
H211—C21—H213	108.8	C51—C56—H561	119.0
H212—C21—H213	109.3	C55—C56—H561	118.5
C19—C22—H221	109.7	Si1—C57—C58	111.3 (3)
C19—C22—H222	111.0	Si1—C57—C59	112.5 (2)
H221—C22—H222	108.5	C58—C57—C59	109.2 (3)
C19—C22—H223	110.1	Si1—C57—C60	108.0 (3)
H221—C22—H223	107.8	C58—C57—C60	108.3 (3)
H222—C22—H223	109.7	C59—C57—C60	107.4 (3)
Si18—C23—C24	121.7 (3)	C57—C58—H581	108.1
Si18—C23—C28	121.5 (3)	C57—C58—H582	109.9
C24—C23—C28	116.6 (3)	H581—C58—H582	109.6
C23—C24—C25	121.2 (4)	C57—C58—H583	109.1
C23—C24—H241	119.2	H581—C58—H583	108.6
C25—C24—H241	119.6	H582—C58—H583	111.6
C24—C25—C26	120.4 (5)	C57—C59—H591	111.5
C24—C25—H251	119.6	C57—C59—H592	111.6

C26—C25—H251	120.0	H591—C59—H592	108.2
C25—C26—C27	119.4 (4)	C57—C59—H593	108.2
C25—C26—H261	120.8	H591—C59—H593	109.0
C27—C26—H261	119.8	H592—C59—H593	108.2
C26—C27—C28	120.8 (4)	C57—C60—H601	109.5
C26—C27—H271	118.4	C57—C60—H602	109.6
C28—C27—H271	120.9	H601—C60—H602	109.4
C23—C28—C27	121.7 (4)	C57—C60—H603	108.2
C23—C28—H281	118.0	H601—C60—H603	110.6
C27—C28—H281	120.3	H602—C60—H603	109.5
Si18—C29—C30	124.3 (3)	C3—O2—Si1	126.2 (2)
Si18—C29—C34	118.9 (3)	C6—O5—C4	109.0 (3)
C30—C29—C34	116.6 (3)	C16—O17—Si18	127.19 (19)
C29—C30—C31	120.6 (4)	C37—O36—C35	109.4 (2)
C29—C30—H301	120.0	C41—O40—C38	117.1 (3)
C31—C30—H301	119.4	C15—N14—C13	122.2 (3)
C30—C31—C32	120.1 (4)	C15—N14—H14	119.0
C30—C31—H311	119.6	C13—N14—H14	118.7
C32—C31—H311	120.2	C7—N801—N802	119.9 (7)
C31—C32—C33	119.6 (4)	N801—N802—N803	170.7 (14)
C31—C32—H321	121.2	C7—N901—N902	109.4 (6)
C33—C32—H321	119.1	N901—N902—N903	172.5 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14···O39	0.84	2.39	3.057 (5)	137
N14—H14···O5	0.84	2.21	2.624 (5)	110