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A fused [3.3.0]-neoglycoside lactone derived from glucuronic acid

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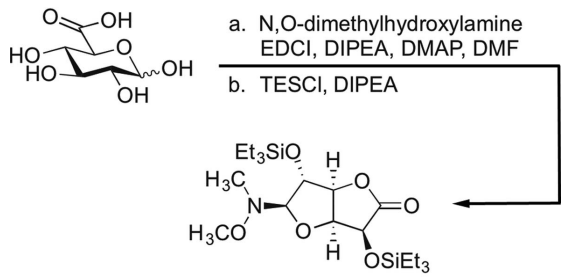
The bridged next-generation aminoglycoside (neoglycoside), 1-deoxy-1-[(methoxy)methylamino)]-2,5-di-*O*-triethylsilyl-β-D-glucofuranurono-γ-lactone {systematic name: (3*S*,3a*S*,5*R*,6*R*,6a*S*)-5-[methoxy(methyl)amino]-3,6-bis[(triethylsilyl)oxy]-2,3,3a,5,6,6a-hexahydrofuro[3,2-*b*]furan-2-one}, $C_{20}H_{41}NO_6Si_2$, was synthesized in a one-pot manner from commercially available D-glucuronic acid. This structure supports the properties associated with the anomeric effect for furanosides and can be employed to provide insight into the mechanisms by which alkoxyamine-appended natural products derive their enhanced biological activity. To the best of our knowledge, this is the first published crystal structure of a bicyclic neoglycoside and is the first neoglycoside to be completely and unambiguously characterized.

Keywords: crystal structure; neoglycoside.

1. Introduction

In 1944, Selman Waksman isolated the first aminoglycoside drug, streptomycin, from bacteria (Jones *et al.*, 1944). Since its discovery, this class of natural products has been used extensively as antibiotics. Unfortunately, mistreatment has resulted in the emergence of resistant strains. This is largely due to the actions of aminoglycoside-modifying enzymes (AMEs) (Armstrong & Miller, 2010). One way to combat resistance is through the use of neoglycosides (Griffith *et al.*, 2007). It is well known that the conjugation of carbohydrates to xenobiotics can significantly impact the pharmacokinetic and pharmacodynamic properties (Ahmed *et al.*, 2006) of a molecule. Neoglycosylation has been successfully employed for the activation of prodrugs (Thorson *et al.*, 2001; Weymouth-Wilson, 1997) to modulate the specificity, toxicity and potency of parent molecules (Goff & Thorson, 2010).

Indeed, the title [3.3.0]-bicyclic neoglycoside, 1-deoxy-1-[(methoxy)methylamino)]-2,5-di-*O*-triethylsilyl-β-D-glucofuranurono-γ-lactone, (I), reported herein is very similar in structure to isoavenaciolide and other naturally occurring fused lactones (Aldridge & Turner, 1971). The densely functionalized bis-lactone framework has served as the central building block for a number of synthetic endeavors designed to enhance antimicrobial and antifungal activities (Burke *et al.*, 1992). As such, (I) may serve as a scaffold for further derivatization of these chiral biologically relevant and naturally occurring lactones. Furthermore, the X-ray crystal structure reported here may shed light on the structure and activity relationship between the antimicrobial and antifungal activity



(I)

of this class of reagents and the ability of its members to inhibit certain phosphatases (Ueda *et al.*, 2002). While investigating conditions for rapid access to biologically relevant glycosides, (I) was obtained in a one-pot manner from α-D-glucuronic acid (see Scheme below). The synthesis provides a straightforward route to a single [3.3.0]-bicyclic lactone containing five points of chirality. Herein we describe the synthesis, characterization and crystal structure of this bridged neoglycoside.

2. Experimental

2.1. Synthesis and characterization

The reaction was carried out on commercially available D-glucuronic acid (Sigma–Aldrich), which exists as a mixture of α and β anomers (see Scheme). For a full list of the general materials and equipment used for this project, see the *Supplementary materials*.

The synthesis of (I) was initiated by suspending glucuronic acid (1.0 g, 5.15 mmol, 1 equivalent) and 4-(dimethylamino)-pyridine (DMAP; 10 mol%) in *N,N*-dimethylformamide (DMF, 10 ml) under an argon atmosphere. *N,N*-Diisopropylethylamine (DIPEA; 3.59 ml, 20.6 mmol, 4 equivalents) was added to the reaction mixture, which became transparent and homogenous within 10 min of addition. *N*-Methylhydroxymethylamine (1 g, 10.3 mmol, 2 equivalents) and 3-[3-(dimethylamino)propyl]-1-ethylcarbodiimide (EDCI; 1.97 g, 10.3 mmol, 2 equivalents) were added and the solution was stirred at room temperature under an argon atmosphere. While the exact mechanism for this transformation is not known, we postulate that once the activated EDC ester is formed, lactonization should be a facile process. Subsequent imine formation and intramolecular ring closure affords fused neoglycoside (I) after protection of the two remaining *cis*-hydroxy groups. As such, once thin-layer chromatography (TLC) revealed complete consumption of the

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Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{41}NO_6Si_2$
M_r	447.72
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	90
a, b, c (Å)	12.0523 (8), 28.1562 (19), 14.5558 (10)
β (°)	90.022 (3)
V (Å^3)	4939.5 (6)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.58
Crystal size (mm)	0.60 × 0.28 × 0.10
Data collection	
Diffractometer	Bruker DUO APEXII CCD diffractometer
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008)
T_{min}, T_{max}	0.451, 0.853
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27457, 16785, 16549
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
R(all data), $wR(F^2)$, S	0.047, 0.116, 1.04
No. of reflections	16785
No. of parameters	1085
No. of restraints	7
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.03, −0.30
Absolute structure	Flack (1983), 7580 Friedel pairs
Absolute structure parameter	0.004 (16)

Computer programs: *APEX2* (Bruker, 2012), *SAINT* (Bruker, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *SHELXTL* (Sheldrick, 2008).

starting materials (48 h), the mixture was cooled to 273 K. Next, a second portion of DIPEA (3.59 ml, 20.6 mmol, 4 equivalents) was added, followed by the addition of chlorotriethylsilane (3.46 ml, 20.6, 4 equivalents). The reaction mixture was stirred and gradually returned to room temperature. After 36 h, the reaction was quenched by the addition of ice water. The solution was next extracted into hexanes (5 × 50 ml). The combined organics were washed with deionized water (25 ml) and then brine (25 ml). The collected organic phase was dried over Na_2SO_4, filtered and the solvent removed *in vacuo* to afford a brown oil. The crude oil was purified using flash column chromatography with a gradient mobile phase consisting of 1–20% EtOAc in hexanes. The structure of (I) was unambiguously assigned using one- and two-dimensional NMR techniques, IR, MS, melting point and optical rotation. In agreement with the literature, the reaction proceeds with complete β-stereoselectivity (Goff & Thorson, 2009). Finally, concentration *in vacuo* afforded compound (I) as a crystalline solid, 592 mg, 26% yield, R_F = 0.47 (hexane–EtOAc = 9:1 *v*/*v*), m.p. 382 K, $[\alpha]_D^{20}$ +37.0° (*c* = 1.0, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 4.84 (*dd*, *J* = 4.8, 6.0 Hz, 1H, H-4), 4.68 (*d*, *J* = 4.8, 1H, H-5), 4.46 (*s*, 1H, H-2), 4.30 (*appt*, *J* = 6.0 Hz, 2H, H-1, H-3), 3.49 (*s*, 3H, OCH_3), 2.57 (*s*, 3H, NCH_3), 0.99 (*dt*, *J* = 7.8, 11.3, 18H), 0.74–0.62 (*m*, 12H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 174.0, 104.1, 84.3, 78.4, 77.4, 70.3, 59.6, 40.3, 6.77, 6.75, 4.76, 4.67. ESI–HRMS calculated for $C_{20}H_{41}NO_6Si_2$ $[M + Na]^+$: 470.2365; found: 470.2357. IR (neat, cm^{-1}): 2955.83 (*s*), 2914.62 (*s*), 2878.22 (*s*), 1793.62 (*s*), 1782.21 (C═O, str, *s*) 1458.28 (*w*), 1414.80 (*w*), 1381.35 (*w*), 1331.29 (*w*). In addition, X-ray crystallography allowed for the assignment of the absolute and relative configuration about all five stereogenic centers.

2.2. Crystal structure determination, refinement and computing details

Crystals of fused neoglycoside (I) were difficult to grow, presumably due to the lipophilic nature conferred by the two triethylsilyl (TES) protecting groups. Initially only very thin needle-like crystals were obtained and they did not provide an adequate diffraction pattern. After several recrystallization attempts, slow evaporation from *n*-hexanes and ethyl acetate (1:8 *v*/*v*) provided large rectangular colorless crystals that all appeared to be twinned under the microscope. One such crystal was then cut to suitable dimensions for data collection. The structure was determined by direct methods, with the successful location of four molecules of identical composition. The central cores of all four molecules are essentially identical, while the terminal TES groups have minor rotational deviations at each end of each molecule. For one of the molecules, one of these TES groups was disordered and was optimized with the final occupancies of the two components determined to be 0.69 and 0.31. Due to the twinning, the twin matrix 100 0$\bar{1}$0 00$\bar{1}$ was required and the two components were refined and found to be 0.53:0.47. H atoms were idealized throughout the final refinement process. The final difference-Fourier map was featureless; the largest peak was 1.03 e Å^{-3}, within 0.80 Å of atom Si1, indicating that the structure is both correct and complete. An empirical correction for extinction was also attempted but was found to be negative and therefore not applied. The absolute structure parameter, Flack *x* (Flack, 1983), was refined and found to be 0.004 (16), indicating that the absolute configuration has been reliably determined. Selected crystallographic experimental parameters and details are shown in Table 1.

3. Results and discussion

The dihedral angle [118.7 (5)°] between the two fused ring systems of (I) is similar to that of other known β-D-glucofuransidurono-6,3-lactone crystal structures. The bond angles about the Csp^3 atoms of the lactone ring (C1*A*, C2*A*, C3 and C4) range from 102.9 (3) to 107.4 (3)°, while that of sp^2-hybridized carbonyl atom C3 is slightly larger [108.9 (3)°]. Similar results are observed for the furanose ring C atoms. The bond angles about the Csp^3 atoms of the furanose ring (C1, C2, C1*A* and C2*A*) range from 102.5 (3) to 106.3 (3)° and are within standard values observed for these systems (Table 2).

Neoglycoside (I) has a similar C—O bond length to the bridging C atoms (C1*A* and C2*A*) reported for other fused lactone systems (Ružić-Toroš *et al.*, 1986). The C—C bond lengths of the core bicyclic system vary from 1.526 (5) to 1.550 (5) Å and are not significantly different from the mean value of 1.532 (12) Å for all C—C bonds. Bridghead atoms C1*A* and C2*A* were found to have the longest C—C bond length [1.550 (5) Å]; however, the difference beteween this

and the other C—C bonds is not significant. The three C—O bonds of the furanose ring, *viz.* C1*A*—O1, C1—O1 and C2*A*—O2 (Table 2), average 1.422 (10) Å. The C2—C4 (C—OSi) and C4—O6 (C—OSi) bond lengths are in agreement with typical values. Both the O4—Si1 and O6—Si2 bond lengths are in agreement with related silyl ethers (Chiaroni *et al.*, 1995). The C3═O5 double bond is within the expected range for a carbonyl group. The two C—O bonds of the γ-lactone ring, *viz.* C3—O2 and C2*A*—O2, differ by 0.10 (5) Å (Table 2). Differences of approximately 0.1 Å have been observed for similar systems containing a planar lactone group (Jeffrey *et al.*, 1967).

Interestingly, the C1*A*—O1 and C1—O1 bond lengths are not equivalent and average 1.422 (10) Å between them. The difference is a result of the anomeric effect (Fig. 3), which is known to cause a slight contraction of the C1—O4 bond in nucleosides (Kirby, 1983). This phenomenon is also responsible for the elongated C1—N1 bond, which is much longer than most C—N bonds [1.475 (5) Å] and is unique to this structure and presumably to *N*-linked glycosides. The C5—N1 bond length is very close to the value expected for a C—N bond of methylamine [1.463 (5) Å].

The stabilizing influence of the anomeric effect in furanosides can be attributed to dipole–dipole interactions, stabi-

Table 2
Selected geometric parameters (Å, °).

C—C (average)	1.52 (3)	C1*A*—O1	1.425 (4)
C1*A*—C2*A*	1.550 (5)	C1—O1	1.409 (4)
C1*A*—C4	1.516 (5)	C2*A*—O2	1.433 (5)
C1—C2	1.526 (5)	C2—O4	1.408 (5)
C2—C2*A*	1.526 (5)	C4—O6	1.403 (4)
C3—C4	1.531 (6)	C3—O2	1.338 (5)
C7—C8	1.520 (5)	C3—O5	1.193 (5)
C9—C10	1.562 (5)	C—N (average)	1.47 (1)
C11—C12	1.479 (6)	C1—N1	1.475 (5)
C13—C14	1.540 (6)	C5—N1	1.463 (5)
C15—C16	1.479 (6)	C—Si (average)	1.872 (11)
C17—C18	1.504 (6)	C7—Si1	1.861 (3)
N—O (average)	1.454 (3)	C9—Si1	1.864 (4)
N1—O3	1.457 (4)	C11—Si1	1.902 (4)
Si—O (average)	1.662 (8)	C13—Si2	1.857 (4)
Si1—O4	1.667 (3)	C15—Si2	1.870 (4)
Si2—O6	1.672 (3)	C17—Si2	1.867 (4)
C—O (average)	1.41 (3)		
O4—Si1—C7	110.23 (15)	C1—O1—C1*A*	110.2 (3)
O4—Si1—C9	102.33 (16)	O4—C2—C1	108.8 (3)
C7—Si1—C9	113.03 (17)	O4—C2—C2*A*	110.2 (3)
O4—Si1—C11	107.48 (16)	C1—C2—C2*A*	102.5 (3)
C7—Si1—C11	109.17 (16)	O2—C2*A*—C2	110.8 (3)
C9—Si1—C11	114.24 (19)	O2—C2*A*—C1*A*	107.4 (3)
O6—Si2—C13	109.42 (18)	C2—C2*A*—C1*A*	104.3 (3)
O6—Si2—C17	109.54 (16)	C3—O2—C2*A*	112.5 (3)
C13—Si2—C17	112.3 (2)	O5—C3—O2	121.8 (4)
O6—Si2—C15	104.44 (18)	O5—C3—C4	129.1 (4)
C13—Si2—C15	110.0 (2)	O2—C3—C4	108.9 (3)
C17—Si2—C15	110.9 (2)	C6—O3—N1	108.8 (3)
O1—C1—N1	108.9 (3)	O6—C4—C1*A*	114.9 (3)
O1—C1—C2	106.3 (3)	O6—C4—C3	111.0 (3)
N1—C1—C2	109.3 (3)	C1*A*—C4—C3	105.3 (3)
O1—C1*A*—C4	113.7 (3)	C2—O4—Si1	122.6 (3)
O1—C1*A*—C2*A*	106.6 (3)	C4—O6—Si2	125.3 (2)
C4—C1*A*—C2*A*	102.9 (3)	C8—C7—Si1	114.3 (3)
O3—N1—C5	105.1 (3)	C12—C11—Si1	116.6 (3)
O3—N1—C1	102.8 (3)	C14—C13—Si2	115.2 (3)
C5—N1—C1	112.0 (3)		

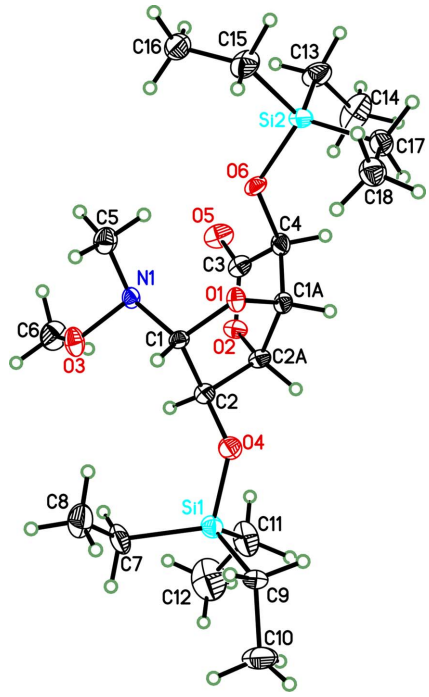

Figure 1
The molecular structure of one of the four symmetry-independent molecules of compound (I), showing the atom-labeling scheme. The atom numbers of the other molecules are derived from the numbering here by adding 20, 40 and 60. Displacement ellipsoids are drawn at the 30% probability level.

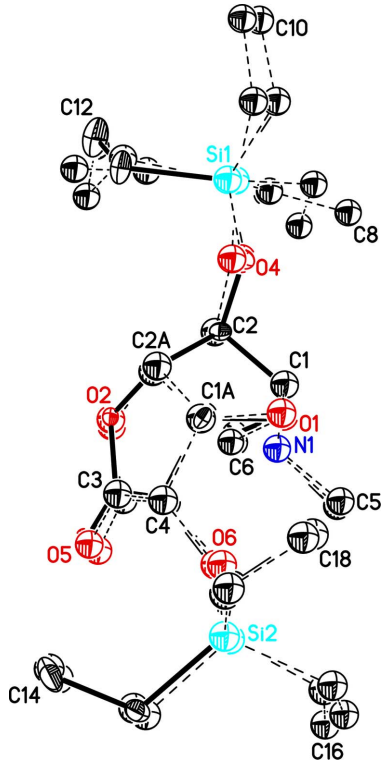

Figure 2
The asymmetric unit of (I) has four different molecules which differ in the conformations of the TES protecting groups, with one terminal methyl group in one molecule also being disordered. All four molecules are shown overlaid upon one another highlighting the differences in the terminal ethyl groups. Displacement ellipsoids are drawn at the 30% probability level.

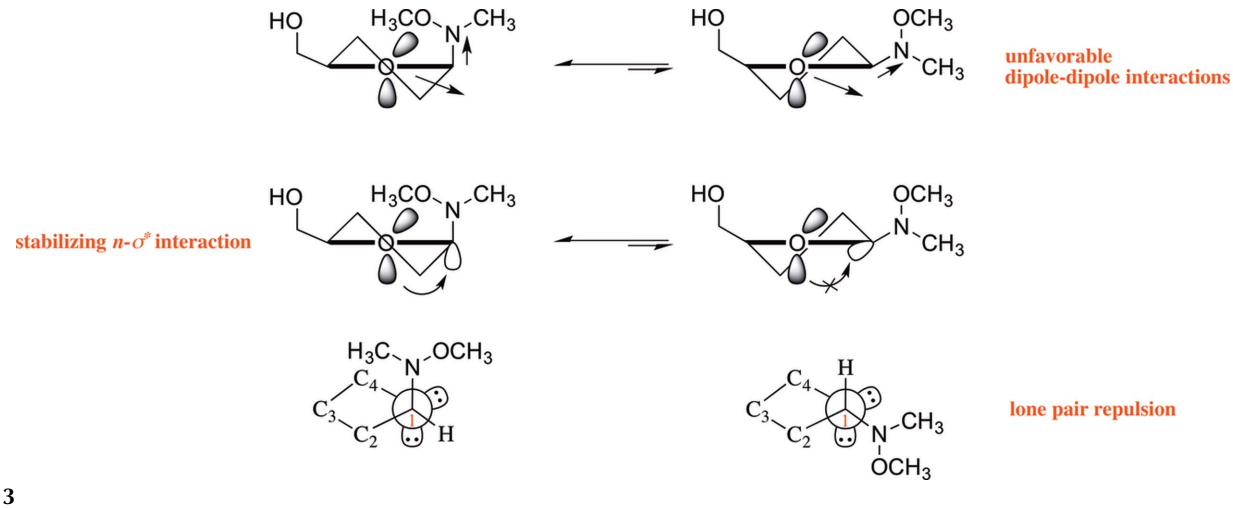

Figure 3
The anomeric effect in furanosides.

lizing hyperconjugative effects of molecular orbital delocalization or electrostatic repulsion.

Worthy of note is that the amine is on the top (*endo*) face of the ring system. The 1,2-*trans* configuration is favored for furanosides owing to the conformational flexibility of the five-membered ring system and the anomeric effect (Satoh & Manabe, 2013). Indeed, a similar trend is observed for nucleosides. Dipole–dipole interactions, stabilizing hyperconjugative effects and electrostatic repulsion also favor the formation of 1,2-transglycofuranosides, as observed for (I) albeit to a lesser degree than is observed for pyranosides (Demchenko, 2008; Thibaudeau *et al.*, 1996) (Fig. 3). The pseudo-axial (*endo*) orientation of the anomeric *N*-methoxymethylamino group contains an O1—C1—N1 angle of 108.9 (3)°, which is similar to those observed for the O—C—N angles of nucleosides (Koole *et al.*, 1988).

The electronic delocalization about the anomeric C atom can also be observed *via* nuclear magnetic resonance (NMR) as the anomeric proton is considerably shielded. Hyperconjugation between the O1 lone-pair electrons and the C1—N1 σ* orbital is similar to that shown in Fig. 3 and is partially responsible for this shift. This interaction is compounded by the electron-rich methoxymethylamine N1 atom and is manifested as a significant upfield shift for H1 to 4.30 p.p.m. in the ^{1}H NMR specrum.

The uniqueness of the neoglycoside species disclosed here becomes apparent when comparing the observed ^{1}H NMR values for (I) with those of other *N*-linked glycosides, as well as other neoglycosides. For example, the chemical shift for the anomeric H atom of nucleosides is typically in the range 5.72–6.28 p.p.m. (Wijmenga & van Buuren, 1998) and the anomeric H atom of similar bicyclic neoglycosides is found at approximately 4.41–4.88 p.p.m. (Goff & Thorson, 2009, 2010). Significant NMR shift differences were also observed in the ^{13}C NMR spectrum. The ^{13}C chemical shift of 104.1 p.p.m. for atom C1 is further downfield than would be typically observed for the anomeric C atom of nucleosides (Ebrahimi *et al.*, 2001; Nair & Young, 1987). To the best of our knowledge, there are no ^{13}C values reported for a similar neoglycoside in the literature.

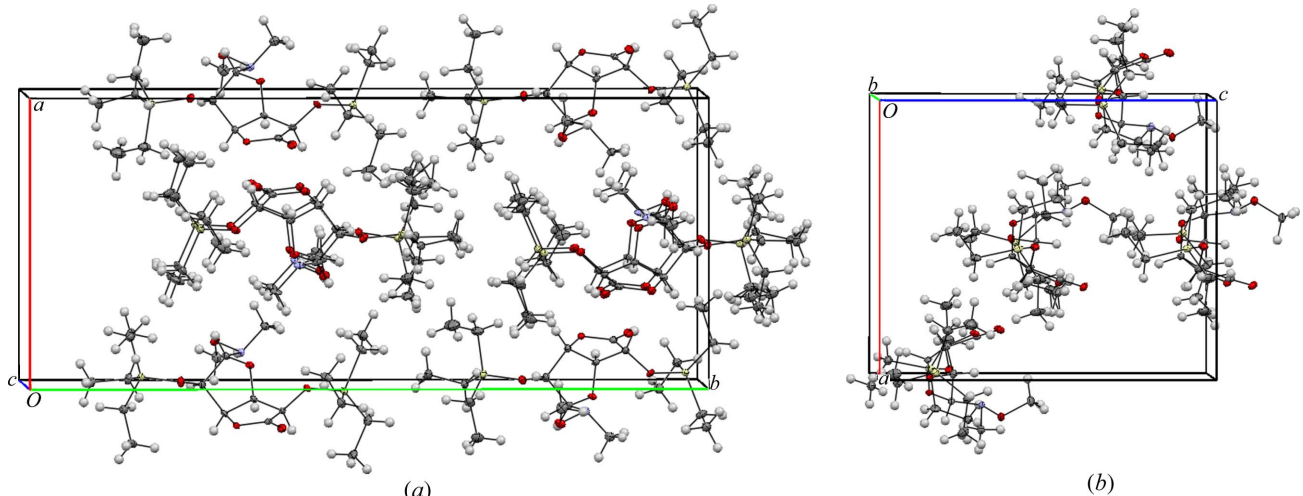

Figure 4
The molecular packing of (I), viewed (*a*) along the crystallographic *c* axis and (*b*) down the crystallographic *b* axis. Displacement ellipsoids are drawn at the 30% probability level.

Furanurono-6,3-lactones are not as flexible as free furanosides due to their rigid bicyclic structures. Since the two five-membered rings are fused at C1*A* and C2*A*, the atom with the largest conformational freedom is the anomeric atom C1. While γ-lactone systems are typically close to planarity, the lactone ring of (I) prefers the E_4 conformation. In agreement with the literature for other β-D-furanurono-6,3-lactones and their derivatives, the furanose ring was found to occupy a $^1T_2$ conformation. The puckering of each ring occurs at positions containing bulky TES ethers. Thus, it can be inferred that the preferred conformation of each ring system is greatly influenced by the steric nature and position of its substituents. Indeed, β-D-mannofuranurono-6,3-lactone (Shalaby *et al.*, 1994), β-D-glucofuranurono-6,3-lactone (Kim *et al.*, 1967) and 5-*O*-pivaloyl-β-D-glucofuranurono-6,3-lactone (Ružić-Toroš *et al.*, 1986) all adopt similar $^1T_2$ conformations in the crystalline form (Liberek *et al.*, 2006). While the $^1T_2$ conformation observed for (I) is slightly twisted about the C1—C2 bond, it still allows the anomeric *N*-methoxymethylamino group to assume a pseudo-axial orientation. This pseudo-axial preference is believed to arise from the *endo*-anomeric effect (Liberek *et al.*, 2006). The results reported herein are in agreement with the literature, as well as with theory, and provide further evidence that the anomeric effect is a main factor in determining the furanoid conformation.

The molecules appear to be held in the crystal by van der Waals forces alone, as there are no hydrogen-bond donors present in the molecule (Fig. 4). In addition, the packing ratio is 21.3 Å^3. While this value is slightly higher than typically observed for the packing of sugars without hydrogen bonds (17.5–19.2 Å^3), it is likely due to the fact that nearly 60% of the atoms in (I) are H atoms (Foces-Foces *et al.*, 1980; Lamba *et al.*, 1986).

4. Conclusions

The one-pot synthesis of a novel next-generation aminoglycoside (neoglycoside) is described. The title compound, (I), was characterized using a variety of techniques, including NMR, IR and single-crystal X-ray analysis. To the best of our knowledge, this is the first published crystal structure of a bicyclic neoglycoside and is the first neoglycoside to be completely and unambiguously characterized. We have demonstrated that a $^1T_2$-like conformation is preferred for the furanoid ring, while the γ-lactone system of (I) adopts an E_4 conformation both in the crystal and in solution. This preference is most likely due to restrictions imposed by the rigid bicyclic framework, the bulky TES ether substituents and the anomeric effect. As such, the structure described here provides further evidence as to the influence of the anomeric effect in furanosides. Finally, the results of this study can be employed to gain insight into the mechanisms by which alkoxyamine-appended natural products are derivatized to enhance biological activity.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: EB5024). Services for accessing these data are described at the back of the journal.

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supplementary materials

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A fused [3.3.0]-neoglycoside lactone derived from glucuronic acid

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Computing details

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT* (Bruker, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(3*S*,3a*S*,5*R*,6*R*,6a*S*)-5-[methoxy(methyl)amino]-3,6-bis[(triethylsilyl)oxy]-hexahydrofuro[3,2-*b*]furan-2-one

Crystal data

$C_{20}H_{41}NO_6Si_2$
M_r = 447.72
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 12.0523 (8) Å
b = 28.1562 (19) Å
c = 14.5558 (10) Å
β = 90.022 (3)°
V = 4939.5 (6) Å^3
Z = 8
$F(000)$ = 1952
D_x = 1.204 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9867 reflections
θ = 3.1–68.4°
μ = 1.58 mm^{-1}
T = 90 K
Plate, colorless
0.60 × 0.28 × 0.10 mm

Data collection

Bruker DUO APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008)
T_{min} = 0.451, T_{max} = 0.853
27457 measured reflections
16785 independent reflections
16549 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 68.3°, θ_{min} = 1.6°
h = −14→14
k = −33→33
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.116
S = 1.04
16785 reflections
1085 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 0.0985P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.03 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack (1983), 7580 Friedel pairs
Absolute structure parameter: 0.004 (16)

Special details

Experimental. A colorless plate with approximate orthogonal dimensions 0.60 *x* 0.28 *x* 0.10 mm3 was placed and optically centered on the Bruker Duo APEXII(1) CCD diffractometer at −183 C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.5deg wide omega-scans, 5 s per frame, and 30 frames per series that were well distributed in reciprocal space. Twenty-one omega and phi-scan data frame series were collected [CuKa] with 0.5deg wide scans and variable frame times based upon diffraction angle. The crystal to detector distance was 4.96 cm, thus providing a nearly complete sphere of data with processing to 2thetamax=136.52deg.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. The following two alerts, #112, indicate possible pseudo symmetry but this is not the case when one overlays the various 4 molecules. 112_ALERT_2_B ADDSYM Detects Additional (Pseudo) Symm. Elem··· 2 112_ALERT_2_B ADDSYM Detects Additional (Pseudo) Symm. Elem··· 2 230_ALERT_2_B Hirshfeld Test Diff for C11 – C12.. 7.7 su

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

One such crystal was then cut to the dimensions 0.60 × 0.28 × 0.10 mm^3 and placed on a Bruker DUO APEXII CCD diffractometer at 90 K (Bruker, 2002). The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.5° wide ω scans (5 s per frame and 30 frames per series) that were well distributed in reciprocal space. 21 ω and φ-scan data-frame series were collected (Cu <it>K</it>α) with 0.5° wide scans and variable frame times based upon diffraction angle. The crystal-to-detector distance was 4.96 cm, thus providing a nearly complete sphere of data with processing to $2\theta_{max}$ = 136.52°. A total of 53215 reflections were collected and corrected for Lorentz and polarization effects in *SAINT* (Bruker, 2009) and absorption using the method of Blessing (1995), as incorporated in *SADABS* (Sheldrick, 2008*b*), with 16851 unique reflections for point group 2 since the molecules were believed to be chiral.

The *SHELXTL* program package (Sheldrick, 2002) was used to determine the probable space group and set up the initial files. System symmetry, systematic absences and intensity statistics indicated the noncentrosymmetric monoclinic space group $P2_1$ (No. 4). The structure was determined by direct methods with the successful location of a majority of the non-H atoms comprising the four unique molecules using the program *SHELXS97* (Sheldrick, 2008*a*). The structure was refined with *SHELXL97* (Fig. 1).

The 53215 data collected were merged, based upon identical indices, to 27457 data (R_{int} = 0.0579) and during least-squares refinement to 16785 unique data (R_{int} = 0.0369). A series of least-squares difference-Fourier cycles were required to locate the remaining non-H atoms comprising the molecules and optimize the disordered ethyl group C atom C32/C32*B*. The central cores of all four molecules are essentially identical, while the terminal ethyl groups have varied positions (Fig. 2). All non-H atoms were refined anisotropically. H atoms were idealized throughout the final convergence stages.

The final structure was refined to convergence with $R(F)$ = 4.70%, $wR(F^2)$ = 11.64%, GOF = 1.043 for all 16785 unique reflections [$R(F)$ = 4.65%, $wR(F^2)$ = 11.58% for those 16549 data with $F_o > 4\sigma(F_o)$].

An empirical correction for extinction was also attempted, but it was ultimately found to be negative and therefore not applied.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Si1	0.97726 (10)	0.18133 (4)	0.17333 (6)	0.02055 (19)	
Si2	0.97092 (8)	0.47909 (3)	0.16411 (8)	0.0192 (2)	
C1	1.0827 (3)	0.30742 (12)	0.2229 (3)	0.0154 (7)	
H1	1.1520	0.2905	0.2039	0.018*	
C1A	0.9360 (3)	0.34772 (13)	0.1543 (3)	0.0174 (7)	
H1A	0.9090	0.3480	0.0893	0.021*	

N1	1.0973 (2)	0.33021 (11)	0.3133 (2)	0.0158 (6)
O1	1.05354 (18)	0.34238 (9)	0.1579 (2)	0.0170 (5)
C2	0.9850 (3)	0.27301 (13)	0.2273 (3)	0.0203 (7)
H2	0.9793	0.2575	0.2889	0.024*
C2A	0.8861 (3)	0.30540 (13)	0.2084 (3)	0.0183 (7)
H2A	0.8278	0.2887	0.1717	0.022*
O2	0.84155 (19)	0.32387 (9)	0.2924 (2)	0.0193 (6)
C3	0.8537 (3)	0.37098 (14)	0.2993 (3)	0.0204 (8)
O3	1.1386 (2)	0.29142 (11)	0.37008 (19)	0.0247 (6)
C4	0.8935 (3)	0.39050 (14)	0.2067 (3)	0.0197 (7)
H4	0.8280	0.4038	0.1731	0.024*
O4	0.9962 (2)	0.23933 (9)	0.1564 (2)	0.0217 (6)
C5	1.1865 (3)	0.36541 (15)	0.3124 (3)	0.0250 (8)
H5A	1.1865	0.3830	0.3705	0.038*
H5B	1.1750	0.3876	0.2614	0.038*
H5C	1.2580	0.3493	0.3047	0.038*
O5	0.8285 (2)	0.39208 (11)	0.3672 (2)	0.0294 (6)
C6	1.0823 (4)	0.29260 (17)	0.4575 (3)	0.0327 (10)
H6A	1.0946	0.3235	0.4868	0.049*
H6B	1.1113	0.2673	0.4970	0.049*
H6C	1.0026	0.2878	0.4479	0.049*
O6	0.9713 (2)	0.42700 (9)	0.2193 (2)	0.0195 (5)
C7	1.0688 (3)	0.15987 (13)	0.2676 (2)	0.0306 (8)
H7A	1.0436	0.1741	0.3261	0.037*
H7B	1.0605	0.1250	0.2730	0.037*
C8	1.1910 (3)	0.17141 (16)	0.2543 (3)	0.0392 (9)
H8A	1.2175	0.1567	0.1973	0.059*
H8B	1.2336	0.1590	0.3064	0.059*
H8C	1.2006	0.2059	0.2506	0.059*
C9	1.0139 (3)	0.15578 (14)	0.0591 (3)	0.0233 (8)
H9A	0.9769	0.1747	0.0107	0.028*
H9B	1.0950	0.1588	0.0500	0.028*
C10	0.9806 (4)	0.10252 (15)	0.0470 (4)	0.0400 (11)
H10A	1.0250	0.0828	0.0886	0.060*
H10B	0.9942	0.0927	−0.0166	0.060*
H10C	0.9017	0.0986	0.0614	0.060*
C11	0.8265 (3)	0.17233 (16)	0.2078 (3)	0.0373 (9)
H11A	0.7986	0.2026	0.2337	0.045*
H11B	0.7831	0.1657	0.1515	0.045*
C12	0.8039 (4)	0.1341 (2)	0.2749 (4)	0.0616 (13)
H12A	0.8317	0.1038	0.2508	0.092*
H12B	0.7238	0.1317	0.2853	0.092*
H12C	0.8413	0.1414	0.3330	0.092*
C13	0.8682 (3)	0.51938 (15)	0.2186 (3)	0.0329 (9)
H13A	0.8884	0.5234	0.2841	0.039*
H13B	0.8737	0.5509	0.1889	0.039*
C14	0.7467 (3)	0.5027 (2)	0.2133 (4)	0.0489 (13)
H14A	0.7246	0.4995	0.1488	0.073*
H14B	0.6987	0.5260	0.2437	0.073*

H14C	0.7393	0.4719	0.2442	0.073*
C15	1.1143 (3)	0.50289 (18)	0.1809 (3)	0.0312 (10)
H15A	1.1665	0.4816	0.1483	0.037*
H15B	1.1184	0.5343	0.1504	0.037*
C16	1.1543 (3)	0.50857 (15)	0.2763 (3)	0.0297 (9)
H16A	1.1004	0.5272	0.3115	0.045*
H16B	1.2260	0.5250	0.2759	0.045*
H16C	1.1631	0.4772	0.3046	0.045*
C17	0.9413 (3)	0.46913 (14)	0.0397 (3)	0.0261 (8)
H17A	0.8716	0.4508	0.0340	0.031*
H17B	0.9294	0.5003	0.0097	0.031*
C18	1.0319 (3)	0.44316 (16)	−0.0106 (3)	0.0294 (9)
H18A	1.0975	0.4636	−0.0153	0.044*
H18B	1.0061	0.4348	−0.0724	0.044*
H18C	1.0511	0.4142	0.0230	0.044*
Si21	0.51986 (9)	0.55631 (4)	0.41834 (7)	0.0219 (2)
Si22	0.53713 (9)	0.25822 (4)	0.41894 (8)	0.0240 (2)
C21	0.4199 (3)	0.43104 (16)	0.4760 (3)	0.0228 (8)
H21	0.3499	0.4479	0.4588	0.027*
C21A	0.5664 (3)	0.39129 (14)	0.4049 (3)	0.0192 (7)
H21A	0.5929	0.3910	0.3398	0.023*
N21	0.4077 (3)	0.40656 (15)	0.5636 (3)	0.0261 (8)
O21	0.4492 (2)	0.39646 (11)	0.4081 (2)	0.0205 (6)
C22	0.5175 (3)	0.46517 (14)	0.4785 (3)	0.0202 (7)
H22	0.5249	0.4812	0.5395	0.024*
C22A	0.6168 (3)	0.43340 (15)	0.4571 (3)	0.0218 (8)
H22A	0.6744	0.4503	0.4200	0.026*
O22	0.6619 (2)	0.41455 (10)	0.5432 (2)	0.0217 (6)
C23	0.6505 (3)	0.36635 (16)	0.5485 (3)	0.0225 (8)
O23	0.3697 (2)	0.44416 (12)	0.6241 (2)	0.0261 (6)
C24	0.6094 (3)	0.34659 (14)	0.4573 (3)	0.0220 (8)
H24	0.6741	0.3332	0.4230	0.026*
O24	0.5060 (2)	0.49852 (10)	0.4053 (2)	0.0217 (6)
C25	0.3173 (3)	0.37146 (18)	0.5598 (3)	0.0287 (9)
H25A	0.2514	0.3861	0.5322	0.043*
H25B	0.2997	0.3607	0.6221	0.043*
H25C	0.3406	0.3443	0.5225	0.043*
O25	0.6752 (2)	0.34488 (11)	0.6161 (2)	0.0274 (6)
C26	0.4300 (4)	0.44054 (17)	0.7064 (3)	0.0323 (10)
H26A	0.4088	0.4113	0.7386	0.048*
H26B	0.4138	0.4680	0.7455	0.048*
H26C	0.5096	0.4397	0.6926	0.048*
O26	0.5319 (2)	0.31071 (10)	0.4702 (2)	0.0245 (6)
C27	0.4242 (3)	0.57774 (14)	0.5113 (3)	0.0337 (8)
H27A	0.4503	0.5648	0.5708	0.040*
H27B	0.4295	0.6128	0.5147	0.040*
C28	0.3050 (3)	0.56437 (16)	0.4988 (3)	0.0395 (9)
H28A	0.2786	0.5762	0.4394	0.059*
H28B	0.2607	0.5784	0.5482	0.059*

H28C	0.2978	0.5297	0.5006	0.059*	
C29	0.4835 (3)	0.58091 (16)	0.3025 (3)	0.0254 (8)	
H29A	0.5230	0.5621	0.2553	0.031*	
H29B	0.4030	0.5763	0.2924	0.031*	
C30	0.5101 (4)	0.63245 (17)	0.2874 (4)	0.0388 (11)	
H30A	0.5904	0.6372	0.2919	0.058*	
H30B	0.4726	0.6517	0.3342	0.058*	
H30C	0.4845	0.6421	0.2262	0.058*	
C31	0.6679 (3)	0.5726 (2)	0.4501 (3)	0.0478 (12)	
H31A	0.7183	0.5517	0.4146	0.057*	0.69
H31B	0.6815	0.6055	0.4292	0.057*	0.69
H31C	0.7085	0.5424	0.4595	0.057*	0.31
H31D	0.7012	0.5880	0.3955	0.057*	0.31
C32	0.6995 (5)	0.5696 (2)	0.5456 (4)	0.0390 (12)	0.69
H32A	0.6490	0.5890	0.5826	0.058*	0.69
H32B	0.7755	0.5812	0.5531	0.058*	0.69
H32C	0.6953	0.5365	0.5660	0.058*	0.69
C32B	0.6909 (10)	0.6009 (5)	0.5239 (8)	0.0375 (19)	0.31
H32D	0.6565	0.6321	0.5149	0.056*	0.31
H32E	0.7714	0.6046	0.5298	0.056*	0.31
H32F	0.6612	0.5862	0.5798	0.056*	0.31
C33	0.6464 (4)	0.22155 (19)	0.4778 (5)	0.0517 (15)	
H33A	0.6239	0.2169	0.5426	0.062*	
H33B	0.6484	0.1899	0.4483	0.062*	
C34	0.7625 (3)	0.24199 (19)	0.4766 (5)	0.0494 (13)	
H34A	0.7884	0.2445	0.4129	0.074*	
H34B	0.8124	0.2211	0.5112	0.074*	
H34C	0.7618	0.2736	0.5048	0.074*	
C35	0.3955 (3)	0.23170 (16)	0.4324 (4)	0.0316 (9)	
H35A	0.3412	0.2521	0.4001	0.038*	
H35B	0.3946	0.2002	0.4022	0.038*	
C36	0.3574 (4)	0.2256 (2)	0.5340 (4)	0.0491 (14)	
H36A	0.4067	0.2031	0.5653	0.074*	
H36B	0.2812	0.2135	0.5353	0.074*	
H36C	0.3603	0.2564	0.5653	0.074*	
C37	0.5699 (3)	0.26545 (15)	0.2938 (3)	0.0283 (9)	
H37A	0.6385	0.2845	0.2876	0.034*	
H37B	0.5845	0.2338	0.2669	0.034*	
C38	0.4754 (4)	0.2899 (2)	0.2385 (3)	0.0399 (11)	
H38A	0.4074	0.2710	0.2437	0.060*	
H38B	0.4970	0.2923	0.1738	0.060*	
H38C	0.4623	0.3218	0.2632	0.060*	
Si41	0.02114 (10)	0.17460 (3)	0.66874 (6)	0.01894 (18)	
Si42	−0.02791 (9)	0.47383 (3)	0.67269 (7)	0.0195 (2)	
C41	0.0909 (3)	0.30075 (13)	0.7260 (3)	0.0178 (7)	
H41	0.1615	0.2842	0.7093	0.021*	
C41A	−0.0544 (3)	0.34022 (13)	0.6556 (3)	0.0175 (7)	
H41A	−0.0814	0.3406	0.5907	0.021*	
N41	0.1010 (2)	0.32475 (10)	0.8160 (2)	0.0141 (6)	

O41	0.06231 (19)	0.33446 (9)	0.6594 (2)	0.0180 (5)
C42	−0.0068 (3)	0.26565 (12)	0.7291 (3)	0.0191 (8)
H42	−0.0130	0.2494	0.7900	0.023*
C42A	−0.1058 (3)	0.29824 (13)	0.7099 (3)	0.0188 (7)
H42A	−0.1641	0.2816	0.6730	0.023*
O42	−0.15062 (19)	0.31694 (9)	0.7937 (2)	0.0196 (6)
C43	−0.1365 (3)	0.36420 (14)	0.8013 (3)	0.0187 (7)
O43	0.1381 (2)	0.28680 (11)	0.87659 (18)	0.0213 (6)
C44	−0.0967 (3)	0.38341 (13)	0.7086 (3)	0.0189 (7)
H44	−0.1623	0.3966	0.6752	0.023*
O44	0.0044 (2)	0.23309 (9)	0.6562 (2)	0.0234 (6)
C45	0.1911 (3)	0.35979 (14)	0.8148 (3)	0.0231 (8)
H45A	0.1895	0.3783	0.8717	0.035*
H45B	0.1815	0.3811	0.7621	0.035*
H45C	0.2625	0.3433	0.8097	0.035*
O45	−0.1617 (2)	0.38519 (10)	0.8692 (2)	0.0287 (6)
C46	0.0791 (4)	0.29041 (15)	0.9618 (3)	0.0276 (9)
H46A	0.0926	0.3217	0.9892	0.041*
H46B	0.1049	0.2656	1.0039	0.041*
H46C	−0.0005	0.2864	0.9506	0.041*
O46	−0.0190 (2)	0.42026 (9)	0.7205 (2)	0.0191 (5)
C47	0.1577 (3)	0.15947 (14)	0.7225 (3)	0.0298 (8)
H47A	0.1780	0.1269	0.7033	0.036*
H47B	0.2144	0.1812	0.6971	0.036*
C48	0.1647 (3)	0.16180 (12)	0.8268 (2)	0.0332 (8)
H48A	0.1486	0.1942	0.8473	0.050*
H48B	0.2395	0.1528	0.8466	0.050*
H48C	0.1105	0.1399	0.8536	0.050*
C49	0.0188 (3)	0.15374 (15)	0.5476 (3)	0.0267 (8)
H49A	−0.0440	0.1691	0.5155	0.032*
H49B	0.0880	0.1641	0.5170	0.032*
C50	0.0076 (3)	0.09882 (15)	0.5368 (3)	0.0296 (9)
H50A	0.0702	0.0832	0.5674	0.044*
H50B	0.0078	0.0906	0.4714	0.044*
H50C	−0.0620	0.0882	0.5648	0.044*
C51	−0.0930 (3)	0.14985 (12)	0.7423 (3)	0.0279 (7)
H51A	−0.0842	0.1149	0.7462	0.033*
H51B	−0.0853	0.1628	0.8052	0.033*
C52	−0.2091 (3)	0.16097 (17)	0.7070 (4)	0.0451 (11)
H52A	−0.2238	0.1950	0.7137	0.068*
H52B	−0.2638	0.1429	0.7426	0.068*
H52C	−0.2145	0.1521	0.6421	0.068*
C53	−0.1323 (3)	0.50961 (14)	0.7362 (3)	0.0274 (8)
H53A	−0.1067	0.5136	0.8004	0.033*
H53B	−0.1364	0.5416	0.7080	0.033*
C54	−0.2494 (3)	0.48785 (14)	0.7374 (3)	0.0339 (9)
H54A	−0.2759	0.4838	0.6742	0.051*
H54B	−0.2999	0.5090	0.7708	0.051*
H54C	−0.2471	0.4569	0.7680	0.051*

C55	0.1143 (3)	0.49937 (17)	0.6860 (3)	0.0285 (9)
H55A	0.1661	0.4799	0.6491	0.034*
H55B	0.1140	0.5317	0.6590	0.034*
C56	0.1598 (4)	0.50284 (15)	0.7817 (3)	0.0301 (9)
H56A	0.1084	0.5211	0.8202	0.045*
H56B	0.2320	0.5188	0.7802	0.045*
H56C	0.1688	0.4709	0.8073	0.045*
C57	−0.0644 (3)	0.46786 (15)	0.5483 (3)	0.0256 (8)
H57A	−0.1328	0.4486	0.5428	0.031*
H57B	−0.0805	0.4998	0.5231	0.031*
C58	0.0260 (4)	0.44506 (19)	0.4909 (3)	0.0335 (10)
H58A	0.0912	0.4658	0.4898	0.050*
H58B	−0.0011	0.4402	0.4281	0.050*
H58C	0.0463	0.4143	0.5178	0.050*
Si61	0.48921 (9)	0.55758 (4)	0.91578 (7)	0.0221 (2)
Si62	0.53586 (8)	0.25881 (3)	0.92032 (8)	0.0207 (2)
C61	0.4211 (3)	0.43303 (16)	0.9769 (3)	0.0218 (8)
H61	0.3512	0.4501	0.9600	0.026*
C61A	0.5664 (3)	0.39218 (14)	0.9057 (3)	0.0197 (8)
H61A	0.5931	0.3916	0.8407	0.024*
N61	0.4086 (3)	0.40830 (13)	1.0648 (3)	0.0218 (7)
O61	0.4491 (2)	0.39810 (10)	0.9089 (2)	0.0203 (6)
C62	0.5185 (3)	0.46671 (13)	0.9786 (2)	0.0184 (7)
H62	0.5262	0.4830	1.0393	0.022*
C62A	0.6180 (3)	0.43418 (14)	0.9580 (3)	0.0179 (7)
H62A	0.6765	0.4507	0.9212	0.021*
O62	0.6614 (2)	0.41511 (10)	1.0443 (2)	0.0220 (6)
C63	0.6473 (3)	0.36728 (16)	1.0500 (4)	0.0238 (9)
O63	0.3700 (2)	0.44583 (11)	1.1252 (2)	0.0226 (6)
C64	0.6074 (3)	0.34761 (14)	0.9583 (3)	0.0216 (8)
H64	0.6727	0.3342	0.9248	0.026*
O64	0.5095 (2)	0.49961 (9)	0.90513 (19)	0.0201 (5)
C65	0.3187 (3)	0.37311 (16)	1.0609 (3)	0.0239 (8)
H65A	0.3008	0.3625	1.1233	0.036*
H65B	0.3424	0.3459	1.0240	0.036*
H65C	0.2528	0.3875	1.0329	0.036*
O65	0.6699 (2)	0.34548 (11)	1.1179 (2)	0.0274 (6)
C66	0.4304 (4)	0.44263 (17)	1.2069 (3)	0.0324 (10)
H66A	0.4154	0.4120	1.2364	0.049*
H66B	0.4084	0.4684	1.2482	0.049*
H66C	0.5098	0.4452	1.1934	0.049*
O66	0.5294 (2)	0.31180 (10)	0.9697 (2)	0.0245 (6)
C67	0.3514 (3)	0.57240 (15)	0.9667 (3)	0.0331 (8)
H67A	0.3297	0.6043	0.9444	0.040*
H67B	0.2965	0.5495	0.9424	0.040*
C68	0.3424 (3)	0.57237 (15)	1.0694 (3)	0.0405 (9)
H68A	0.3468	0.5397	1.0921	0.061*
H68B	0.2712	0.5864	1.0876	0.061*
H68C	0.4032	0.5911	1.0956	0.061*

C69	0.4964 (3)	0.58018 (16)	0.7945 (3)	0.0290 (9)
H69A	0.5636	0.5669	0.7650	0.035*
H69B	0.4313	0.5680	0.7604	0.035*
C70	0.4994 (3)	0.63377 (16)	0.7850 (4)	0.0346 (10)
H70A	0.4388	0.6477	0.8209	0.052*
H70B	0.4908	0.6424	0.7202	0.052*
H70C	0.5706	0.6458	0.8079	0.052*
C71	0.6017 (3)	0.58286 (12)	0.9905 (3)	0.0310 (8)
H71A	0.5908	0.6176	0.9948	0.037*
H71B	0.5932	0.5696	1.0531	0.037*
C72	0.7184 (3)	0.57353 (15)	0.9585 (3)	0.0366 (9)
H72A	0.7709	0.5851	1.0047	0.055*
H72B	0.7313	0.5901	0.9003	0.055*
H72C	0.7289	0.5393	0.9495	0.055*
C73	0.6414 (4)	0.2215 (2)	0.9820 (5)	0.0516 (15)
H73A	0.6235	0.2217	1.0483	0.062*
H73B	0.6343	0.1884	0.9601	0.062*
C74	0.7610 (3)	0.2368 (2)	0.9704 (4)	0.0501 (13)
H74A	0.7794	0.2380	0.9049	0.075*
H74B	0.8098	0.2139	1.0013	0.075*
H74C	0.7714	0.2683	0.9976	0.075*
C75	0.3950 (3)	0.23227 (15)	0.9314 (3)	0.0276 (9)
H75A	0.3415	0.2527	0.8982	0.033*
H75B	0.3951	0.2009	0.9008	0.033*
C76	0.3528 (3)	0.22566 (18)	1.0332 (4)	0.0368 (11)
H76A	0.4013	0.2033	1.0655	0.055*
H76B	0.2768	0.2132	1.0324	0.055*
H76C	0.3539	0.2564	1.0649	0.055*
C77	0.5727 (3)	0.26512 (15)	0.7963 (3)	0.0267 (8)
H77A	0.6417	0.2841	0.7912	0.032*
H77B	0.5879	0.2332	0.7706	0.032*
C78	0.4804 (4)	0.2893 (2)	0.7382 (3)	0.0410 (12)
H78A	0.4102	0.2724	0.7470	0.062*
H78B	0.5010	0.2884	0.6731	0.062*
H78C	0.4718	0.3224	0.7578	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0202 (4)	0.0191 (4)	0.0223 (4)	−0.0026 (4)	−0.0009 (4)	0.0039 (3)
Si2	0.0169 (4)	0.0154 (4)	0.0252 (5)	−0.0006 (4)	0.0011 (4)	−0.0018 (4)
C1	0.0187 (15)	0.0135 (15)	0.0139 (16)	−0.0035 (12)	−0.0001 (13)	−0.0001 (12)
C1A	0.0133 (15)	0.0197 (15)	0.0191 (17)	−0.0049 (13)	−0.0026 (14)	0.0037 (14)
N1	0.0190 (13)	0.0177 (13)	0.0107 (14)	0.0037 (11)	−0.0011 (11)	−0.0012 (12)
O1	0.0122 (11)	0.0230 (12)	0.0157 (12)	0.0028 (9)	0.0009 (10)	0.0013 (10)
C2	0.0245 (16)	0.0179 (16)	0.0186 (16)	0.0002 (14)	−0.0032 (15)	−0.0012 (13)
C2A	0.0185 (16)	0.0161 (15)	0.0203 (19)	−0.0008 (13)	0.0008 (14)	−0.0042 (13)
O2	0.0138 (10)	0.0156 (12)	0.0284 (15)	−0.0004 (9)	0.0039 (10)	−0.0007 (10)
C3	0.0137 (15)	0.0233 (18)	0.0244 (19)	−0.0008 (13)	0.0026 (14)	−0.0051 (15)
O3	0.0254 (12)	0.0343 (15)	0.0143 (13)	0.0066 (11)	−0.0005 (10)	0.0035 (11)

C4	0.0138 (14)	0.0216 (17)	0.0237 (19)	−0.0007 (13)	−0.0039 (13)	0.0034 (15)
O4	0.0247 (14)	0.0212 (13)	0.0191 (11)	0.0026 (9)	0.0010 (10)	−0.0016 (11)
C5	0.0267 (18)	0.0292 (18)	0.0192 (18)	0.0011 (15)	−0.0007 (15)	−0.0021 (16)
O5	0.0227 (12)	0.0297 (15)	0.0357 (16)	0.0008 (11)	0.0091 (11)	−0.0094 (13)
C6	0.040 (2)	0.040 (2)	0.019 (2)	−0.0049 (18)	0.0027 (17)	0.0034 (18)
O6	0.0168 (10)	0.0186 (11)	0.0230 (12)	−0.0071 (10)	−0.0027 (10)	−0.0020 (10)
C7	0.0286 (18)	0.0317 (17)	0.0316 (15)	0.0030 (15)	−0.0079 (14)	0.0135 (13)
C8	0.0313 (19)	0.046 (2)	0.040 (2)	0.0004 (18)	−0.0117 (17)	0.0011 (18)
C9	0.0283 (17)	0.0145 (16)	0.0270 (17)	0.0021 (14)	−0.0029 (14)	−0.0034 (14)
C10	0.0312 (18)	0.0228 (18)	0.066 (3)	0.0016 (17)	−0.006 (2)	−0.0144 (19)
C11	0.0230 (17)	0.051 (2)	0.0377 (19)	−0.0023 (18)	0.0016 (16)	0.0157 (18)
C12	0.038 (2)	0.078 (3)	0.069 (3)	−0.008 (2)	0.005 (2)	0.019 (3)
C13	0.0265 (18)	0.0273 (19)	0.045 (2)	0.0037 (15)	−0.0024 (17)	−0.0135 (17)
C14	0.0263 (19)	0.068 (3)	0.052 (3)	0.016 (2)	−0.0039 (19)	−0.030 (3)
C15	0.0214 (18)	0.037 (2)	0.035 (2)	−0.0119 (16)	0.0011 (17)	0.0023 (19)
C16	0.0290 (19)	0.0286 (18)	0.031 (2)	0.0002 (16)	−0.0040 (17)	−0.0043 (16)
C17	0.0260 (17)	0.0272 (18)	0.0251 (18)	−0.0012 (15)	−0.0023 (15)	0.0047 (16)
C18	0.0312 (18)	0.030 (2)	0.027 (2)	−0.0002 (17)	0.0004 (18)	0.0030 (17)
Si21	0.0168 (4)	0.0264 (4)	0.0225 (4)	−0.0026 (5)	0.0000 (4)	−0.0049 (3)
Si22	0.0198 (4)	0.0207 (5)	0.0315 (6)	−0.0029 (4)	−0.0042 (5)	0.0068 (4)
C21	0.0153 (16)	0.036 (2)	0.0167 (18)	0.0060 (15)	−0.0039 (14)	0.0010 (16)
C21A	0.0129 (15)	0.0248 (17)	0.0198 (17)	0.0051 (14)	0.0011 (14)	0.0066 (15)
N21	0.0168 (13)	0.044 (2)	0.0175 (16)	0.0003 (13)	0.0016 (12)	−0.0013 (15)
O21	0.0144 (12)	0.0292 (12)	0.0179 (13)	0.0012 (11)	−0.0008 (10)	−0.0003 (10)
C22	0.0203 (15)	0.0271 (19)	0.0132 (15)	0.0005 (15)	−0.0021 (14)	0.0051 (14)
C22A	0.0194 (17)	0.0279 (19)	0.0181 (19)	0.0010 (14)	−0.0017 (14)	0.0090 (15)
O22	0.0197 (11)	0.0269 (13)	0.0184 (13)	−0.0014 (10)	−0.0029 (10)	0.0044 (11)
C23	0.0121 (15)	0.0263 (18)	0.029 (2)	0.0015 (14)	0.0009 (15)	0.0033 (16)
O23	0.0253 (12)	0.0312 (15)	0.0219 (14)	0.0045 (11)	0.0011 (12)	0.0013 (12)
C24	0.0200 (15)	0.0222 (17)	0.0236 (19)	−0.0024 (14)	−0.0065 (14)	0.0110 (15)
O24	0.0245 (14)	0.0230 (13)	0.0175 (11)	0.0066 (10)	0.0012 (10)	0.0052 (11)
C25	0.0149 (16)	0.048 (2)	0.023 (2)	−0.0105 (17)	0.0057 (15)	0.0051 (18)
O25	0.0246 (12)	0.0344 (15)	0.0232 (13)	−0.0057 (12)	−0.0096 (10)	0.0085 (12)
C26	0.0327 (19)	0.048 (3)	0.0161 (19)	−0.0066 (19)	0.0017 (16)	−0.0005 (18)
O26	0.0187 (11)	0.0282 (14)	0.0266 (13)	−0.0052 (11)	−0.0011 (11)	0.0009 (11)
C27	0.0282 (19)	0.0365 (19)	0.0365 (17)	0.0025 (15)	0.0065 (15)	−0.0119 (15)
C28	0.0227 (17)	0.049 (2)	0.047 (2)	0.0082 (17)	0.0109 (17)	−0.0104 (19)
C29	0.0214 (15)	0.0242 (18)	0.0306 (19)	0.0031 (15)	0.0032 (16)	0.0050 (15)
C30	0.0256 (19)	0.030 (2)	0.061 (3)	−0.0114 (16)	−0.005 (2)	0.009 (2)
C31	0.0240 (19)	0.079 (3)	0.040 (2)	−0.006 (2)	0.0006 (17)	−0.019 (2)
C32	0.029 (2)	0.051 (3)	0.037 (3)	−0.010 (3)	−0.003 (2)	0.009 (2)
C32B	0.024 (4)	0.047 (4)	0.042 (4)	−0.010 (4)	−0.002 (3)	0.002 (4)
C33	0.026 (2)	0.040 (2)	0.089 (4)	0.0031 (18)	−0.006 (2)	0.036 (3)
C34	0.0220 (18)	0.049 (3)	0.078 (4)	0.0028 (18)	−0.011 (2)	0.025 (2)
C35	0.029 (2)	0.025 (2)	0.040 (2)	−0.0095 (16)	−0.0028 (19)	0.0052 (18)
C36	0.027 (2)	0.069 (3)	0.051 (3)	−0.023 (2)	−0.005 (2)	0.024 (3)
C37	0.0205 (16)	0.0269 (19)	0.037 (2)	−0.0053 (15)	0.0003 (16)	−0.0028 (18)
C38	0.0329 (19)	0.060 (3)	0.027 (2)	−0.013 (2)	−0.005 (2)	0.005 (2)
Si41	0.0197 (4)	0.0156 (4)	0.0215 (4)	0.0016 (4)	−0.0011 (4)	−0.0006 (3)

Si42	0.0199 (4)	0.0159 (4)	0.0227 (5)	−0.0041 (4)	0.0021 (5)	−0.0014 (4)
C41	0.0181 (16)	0.0182 (15)	0.0171 (17)	−0.0016 (13)	−0.0029 (13)	−0.0005 (14)
C41A	0.0165 (15)	0.0165 (16)	0.0194 (17)	−0.0054 (13)	−0.0017 (14)	0.0047 (14)
N41	0.0187 (12)	0.0132 (14)	0.0103 (14)	−0.0012 (10)	−0.0011 (11)	−0.0010 (11)
O41	0.0159 (11)	0.0241 (12)	0.0138 (12)	−0.0012 (10)	0.0010 (10)	0.0011 (10)
C42	0.0219 (17)	0.0136 (15)	0.0218 (17)	0.0007 (12)	−0.0016 (13)	0.0019 (14)
C42A	0.0170 (15)	0.0176 (16)	0.0217 (18)	−0.0083 (13)	−0.0004 (14)	0.0044 (14)
O42	0.0144 (11)	0.0142 (11)	0.0301 (15)	−0.0023 (9)	0.0066 (10)	0.0007 (10)
C43	0.0127 (14)	0.0183 (16)	0.0252 (19)	0.0018 (13)	0.0025 (13)	−0.0028 (14)
O43	0.0263 (12)	0.0249 (13)	0.0128 (12)	0.0066 (10)	−0.0012 (10)	0.0032 (10)
C44	0.0130 (13)	0.0155 (16)	0.0281 (19)	−0.0003 (12)	−0.0021 (14)	0.0049 (14)
O44	0.0327 (15)	0.0179 (12)	0.0195 (11)	−0.0002 (10)	−0.0023 (11)	−0.0012 (10)
C45	0.0225 (17)	0.0249 (18)	0.0220 (19)	−0.0018 (14)	0.0007 (15)	0.0015 (15)
O45	0.0241 (12)	0.0261 (14)	0.0358 (16)	0.0017 (11)	0.0072 (11)	−0.0061 (12)
C46	0.0349 (19)	0.030 (2)	0.0180 (19)	−0.0013 (16)	0.0044 (16)	0.0033 (16)
O46	0.0169 (10)	0.0165 (11)	0.0240 (12)	−0.0054 (9)	−0.0004 (10)	−0.0027 (10)
C47	0.0248 (19)	0.0303 (17)	0.0342 (19)	0.0021 (15)	−0.0006 (15)	0.0075 (15)
C48	0.0386 (19)	0.0260 (15)	0.0349 (17)	0.0048 (14)	−0.0186 (16)	0.0043 (14)
C49	0.0381 (19)	0.0200 (17)	0.0219 (17)	0.0053 (16)	−0.0010 (16)	0.0017 (14)
C50	0.032 (2)	0.0240 (18)	0.0323 (18)	0.0053 (15)	−0.0029 (16)	−0.0102 (15)
C51	0.0222 (17)	0.0219 (15)	0.0396 (18)	−0.0015 (14)	0.0006 (14)	0.0008 (13)
C52	0.0200 (18)	0.041 (2)	0.074 (3)	−0.0027 (16)	−0.003 (2)	−0.011 (2)
C53	0.0299 (18)	0.0243 (17)	0.0279 (18)	0.0019 (15)	0.0022 (15)	−0.0099 (15)
C54	0.0308 (19)	0.0250 (18)	0.046 (2)	0.0056 (15)	0.0122 (17)	−0.0066 (16)
C55	0.0253 (18)	0.035 (2)	0.025 (2)	−0.0126 (16)	0.0005 (16)	0.0016 (17)
C56	0.0326 (19)	0.0319 (19)	0.0259 (19)	−0.0048 (17)	−0.0023 (17)	−0.0096 (16)
C57	0.0298 (18)	0.0250 (18)	0.0222 (18)	−0.0039 (16)	−0.0026 (15)	0.0054 (15)
C58	0.0261 (17)	0.048 (3)	0.027 (2)	−0.011 (2)	0.0060 (18)	−0.008 (2)
Si61	0.0222 (5)	0.0215 (4)	0.0226 (4)	0.0008 (4)	−0.0043 (4)	−0.0018 (3)
Si62	0.0168 (4)	0.0179 (4)	0.0274 (5)	−0.0007 (4)	−0.0023 (5)	0.0025 (4)
C61	0.0190 (16)	0.0283 (18)	0.0180 (18)	0.0030 (15)	−0.0033 (14)	0.0007 (16)
C61A	0.0161 (16)	0.0233 (17)	0.0196 (17)	0.0023 (14)	0.0003 (14)	0.0049 (15)
N61	0.0177 (13)	0.0295 (18)	0.0183 (16)	−0.0026 (12)	0.0026 (12)	−0.0009 (13)
O61	0.0191 (13)	0.0241 (12)	0.0177 (13)	−0.0041 (11)	−0.0015 (10)	−0.0025 (10)
C62	0.0206 (14)	0.0206 (17)	0.0140 (15)	−0.0024 (14)	−0.0036 (14)	0.0023 (14)
C62A	0.0163 (16)	0.0213 (16)	0.0161 (18)	0.0001 (13)	−0.0007 (13)	0.0048 (14)
O62	0.0229 (12)	0.0235 (13)	0.0195 (13)	−0.0036 (10)	−0.0033 (11)	0.0034 (11)
C63	0.0113 (16)	0.0259 (18)	0.034 (2)	−0.0015 (14)	−0.0009 (15)	0.0015 (17)
O63	0.0254 (12)	0.0238 (13)	0.0185 (13)	0.0005 (10)	−0.0005 (11)	0.0002 (11)
C64	0.0185 (15)	0.0199 (16)	0.0264 (19)	−0.0050 (14)	−0.0033 (14)	0.0090 (15)
O64	0.0229 (13)	0.0185 (12)	0.0189 (11)	−0.0018 (9)	0.0003 (11)	0.0044 (10)
C65	0.0128 (15)	0.035 (2)	0.024 (2)	−0.0071 (15)	0.0052 (14)	0.0082 (17)
O65	0.0254 (12)	0.0298 (14)	0.0269 (14)	−0.0066 (12)	−0.0100 (11)	0.0096 (12)
C66	0.0333 (19)	0.046 (3)	0.018 (2)	−0.0082 (18)	−0.0016 (17)	−0.0010 (19)
O66	0.0206 (11)	0.0239 (13)	0.0291 (14)	−0.0032 (11)	0.0022 (12)	−0.0026 (11)
C67	0.0263 (18)	0.040 (2)	0.0334 (19)	0.0049 (16)	−0.0014 (16)	−0.0072 (16)
C68	0.033 (2)	0.041 (2)	0.047 (2)	−0.0025 (17)	0.0096 (18)	0.0023 (17)
C69	0.036 (2)	0.0245 (19)	0.0269 (18)	0.0049 (15)	0.0016 (15)	0.0094 (15)
C70	0.035 (2)	0.0245 (18)	0.044 (2)	−0.0066 (15)	−0.0028 (17)	0.0051 (17)

C71	0.0336 (19)	0.0215 (15)	0.0378 (18)	−0.0058 (15)	−0.0129 (15)	−0.0008 (14)
C72	0.0225 (17)	0.032 (2)	0.055 (2)	−0.0053 (15)	−0.0028 (18)	−0.0055 (18)
C73	0.030 (2)	0.053 (3)	0.072 (4)	0.010 (2)	−0.003 (2)	0.035 (3)
C74	0.0217 (19)	0.062 (3)	0.066 (3)	0.015 (2)	−0.001 (2)	0.025 (3)
C75	0.0256 (18)	0.0143 (17)	0.043 (2)	−0.0040 (14)	−0.0026 (18)	−0.0001 (17)
C76	0.0241 (18)	0.040 (2)	0.047 (3)	−0.0102 (17)	−0.0004 (19)	0.008 (2)
C77	0.0186 (15)	0.0292 (19)	0.032 (2)	−0.0036 (15)	0.0023 (15)	−0.0054 (17)
C78	0.036 (2)	0.063 (3)	0.025 (2)	−0.008 (2)	0.0015 (19)	0.005 (2)

Geometric parameters (Å, º)

C—C	1.52 (3)	C37—C38	1.555 (6)
C1A—C2A	1.550 (5)	C37—H37A	0.9900
C1A—C4	1.516 (5)	C37—H37B	0.9900
C1—C2	1.526 (5)	C38—H38A	0.9800
C2—C2A	1.526 (5)	C38—H38B	0.9800
C3—C4	1.531 (6)	C38—H38C	0.9800
C7—C8	1.520 (5)	Si41—O44	1.669 (3)
C9—C10	1.562 (5)	Si41—C49	1.858 (4)
C11—C12	1.479 (6)	Si41—C47	1.872 (4)
C13—C14	1.540 (6)	Si41—C51	1.878 (4)
C15—C16	1.479 (6)	Si42—O46	1.664 (3)
C17—C18	1.504 (6)	Si42—C53	1.859 (4)
N—O	1.454 (3)	Si42—C55	1.868 (4)
N1—O3	1.457 (4)	Si42—C57	1.871 (4)
Si—O	1.662 (8)	C41—O41	1.400 (5)
Si1—O4	1.667 (3)	C41—N41	1.479 (5)
Si2—O6	1.672 (3)	C41—C42	1.538 (5)
C—O	1.41 (3)	C41—H41	1.0000
C1A—O1	1.425 (4)	C41A—O41	1.418 (4)
C1—O1	1.409 (4)	C41A—C44	1.528 (5)
C2A—O2	1.433 (5)	C41A—C42A	1.551 (5)
C2—O4	1.408 (5)	C41A—H41A	1.0000
C4—O6	1.403 (4)	N41—O43	1.456 (4)
C3—O2	1.338 (5)	N41—C45	1.468 (5)
C3—O5	1.193 (5)	C42—O44	1.408 (5)
C—N	1.47 (1)	C42—C42A	1.532 (5)
C1—N1	1.475 (5)	C42—H42	1.0000
C5—N1	1.463 (5)	C42A—O42	1.434 (5)
C—Si	1.872 (11)	C42A—H42A	1.0000
C7—Si1	1.861 (3)	O42—C43	1.346 (5)
C9—Si1	1.864 (4)	C43—O45	1.191 (5)
C11—Si1	1.902 (4)	C43—C44	1.531 (6)
C13—Si2	1.857 (4)	O43—C46	1.433 (5)
C15—Si2	1.870 (4)	C44—O46	1.408 (4)
C17—Si2	1.867 (4)	C44—H44	1.0000
C1—H1	1.0000	C45—H45A	0.9800
C1A—H1A	1.0000	C45—H45B	0.9800
C2—H2	1.0000	C45—H45C	0.9800
C2A—H2A	1.0000	C46—H46A	0.9800

O2—C3	1.338 (5)	C46—H46B	0.9800
O3—C6	1.443 (5)	C46—H46C	0.9800
C4—H4	1.0000	C47—C48	1.521 (5)
C5—H5A	0.9800	C47—H47A	0.9900
C5—H5B	0.9800	C47—H47B	0.9900
C5—H5C	0.9800	C48—H48A	0.9800
C6—H6A	0.9800	C48—H48B	0.9800
C6—H6B	0.9800	C48—H48C	0.9800
C6—H6C	0.9800	C49—C50	1.560 (6)
C7—H7A	0.9900	C49—H49A	0.9900
C7—H7B	0.9900	C49—H49B	0.9900
C8—H8A	0.9800	C50—H50A	0.9800
C8—H8B	0.9800	C50—H50B	0.9800
C8—H8C	0.9800	C50—H50C	0.9800
C9—H9A	0.9900	C51—C52	1.523 (5)
C9—H9B	0.9900	C51—H51A	0.9900
C10—H10A	0.9800	C51—H51B	0.9900
C10—H10B	0.9800	C52—H52A	0.9800
C10—H10C	0.9800	C52—H52B	0.9800
C11—H11A	0.9900	C52—H52C	0.9800
C11—H11B	0.9900	C53—C54	1.539 (5)
C12—H12A	0.9800	C53—H53A	0.9900
C12—H12B	0.9800	C53—H53B	0.9900
C12—H12C	0.9800	C54—H54A	0.9800
C13—H13A	0.9900	C54—H54B	0.9800
C13—H13B	0.9900	C54—H54C	0.9800
C14—H14A	0.9800	C55—C56	1.501 (6)
C14—H14B	0.9800	C55—H55A	0.9900
C14—H14C	0.9800	C55—H55B	0.9900
C15—H15A	0.9900	C56—H56A	0.9800
C15—H15B	0.9900	C56—H56B	0.9800
C16—H16A	0.9800	C56—H56C	0.9800
C16—H16B	0.9800	C57—C58	1.515 (6)
C16—H16C	0.9800	C57—H57A	0.9900
C17—H17A	0.9900	C57—H57B	0.9900
C17—H17B	0.9900	C58—H58A	0.9800
C18—H18A	0.9800	C58—H58B	0.9800
C18—H18B	0.9800	C58—H58C	0.9800
C18—H18C	0.9800	Si61—O64	1.658 (3)
Si21—O24	1.647 (3)	Si61—C67	1.866 (4)
Si21—C29	1.875 (4)	Si61—C71	1.877 (4)
Si21—C27	1.877 (4)	Si61—C69	1.878 (4)
Si21—C31	1.899 (4)	Si62—O66	1.658 (3)
Si22—O26	1.657 (3)	Si62—C75	1.862 (4)
Si22—C35	1.874 (4)	Si62—C77	1.867 (5)
Si22—C37	1.875 (5)	Si62—C73	1.878 (5)
Si22—C33	1.880 (5)	C61—O61	1.436 (5)
C21—O21	1.432 (5)	C61—N61	1.464 (5)
C21—N21	1.457 (6)	C61—C62	1.510 (5)

C21—C22	1.519 (5)	C61—H61	1.0000
C21—H21	1.0000	C61A—O61	1.424 (5)
C21A—O21	1.420 (4)	C61A—C62A	1.537 (6)
C21A—C22A	1.533 (6)	C61A—C64	1.551 (5)
C21A—C24	1.560 (5)	C61A—H61A	1.0000
C21A—H21A	1.0000	N61—O63	1.452 (5)
N21—O23	1.451 (5)	N61—C65	1.470 (5)
N21—C25	1.472 (5)	C62—O64	1.419 (4)
C22—O24	1.427 (4)	C62—C62A	1.538 (5)
C22—C22A	1.526 (5)	C62—H62	1.0000
C22—H22	1.0000	C62A—O62	1.463 (5)
C22A—O22	1.465 (5)	C62A—H62A	1.0000
C22A—H22A	1.0000	O62—C63	1.360 (5)
O22—C23	1.366 (5)	C63—O65	1.195 (6)
C23—O25	1.193 (5)	C63—C64	1.523 (6)
C23—C24	1.523 (6)	O63—C66	1.397 (5)
O23—C26	1.406 (5)	C64—O66	1.388 (4)
C24—O26	1.389 (4)	C64—H64	1.0000
C24—H24	1.0000	C65—H65A	0.9800
C25—H25A	0.9800	C65—H65B	0.9800
C25—H25B	0.9800	C65—H65C	0.9800
C25—H25C	0.9800	C66—H66A	0.9800
C26—H26A	0.9800	C66—H66B	0.9800
C26—H26B	0.9800	C66—H66C	0.9800
C26—H26C	0.9800	C67—C68	1.498 (5)
C27—C28	1.496 (5)	C67—H67A	0.9900
C27—H27A	0.9900	C67—H67B	0.9900
C27—H27B	0.9900	C68—H68A	0.9800
C28—H28A	0.9800	C68—H68B	0.9800
C28—H28B	0.9800	C68—H68C	0.9800
C28—H28C	0.9800	C69—C70	1.515 (6)
C29—C30	1.502 (6)	C69—H69A	0.9900
C29—H29A	0.9900	C69—H69B	0.9900
C29—H29B	0.9900	C70—H70A	0.9800
C30—H30A	0.9800	C70—H70B	0.9800
C30—H30B	0.9800	C70—H70C	0.9800
C30—H30C	0.9800	C71—C72	1.505 (5)
C31—C32B	1.366 (13)	C71—H71A	0.9900
C31—C32	1.444 (7)	C71—H71B	0.9900
C31—H31A	0.9900	C72—H72A	0.9800
C31—H31B	0.9900	C72—H72B	0.9800
C31—H31C	0.9900	C72—H72C	0.9800
C31—H31D	0.9900	C73—C74	1.513 (6)
C32—H32A	0.9800	C73—H73A	0.9900
C32—H32B	0.9800	C73—H73B	0.9900
C32—H32C	0.9800	C74—H74A	0.9800
C32B—H32D	0.9800	C74—H74B	0.9800
C32B—H32E	0.9800	C74—H74C	0.9800
C32B—H32F	0.9800	C75—C76	1.577 (7)

C33—C34	1.513 (6)	C75—H75A	0.9900
C33—H33A	0.9900	C75—H75B	0.9900
C33—H33B	0.9900	C76—H76A	0.9800
C34—H34A	0.9800	C76—H76B	0.9800
C34—H34B	0.9800	C76—H76C	0.9800
C34—H34C	0.9800	C77—C78	1.555 (7)
C35—C36	1.558 (7)	C77—H77A	0.9900
C35—H35A	0.9900	C77—H77B	0.9900
C35—H35B	0.9900	C78—H78A	0.9800
C36—H36A	0.9800	C78—H78B	0.9800
C36—H36B	0.9800	C78—H78C	0.9800
C36—H36C	0.9800		

O4—Si1—C7	110.23 (15)	C37—C38—H38A	109.5
O4—Si1—C9	102.33 (16)	C37—C38—H38B	109.5
C7—Si1—C9	113.03 (17)	H38A—C38—H38B	109.5
O4—Si1—C11	107.48 (16)	C37—C38—H38C	109.5
C7—Si1—C11	109.17 (16)	H38A—C38—H38C	109.5
C9—Si1—C11	114.24 (19)	H38B—C38—H38C	109.5
O6—Si2—C13	109.42 (18)	O44—Si41—C49	101.91 (17)
O6—Si2—C17	109.54 (16)	O44—Si41—C47	112.11 (16)
C13—Si2—C17	112.3 (2)	C49—Si41—C47	109.75 (18)
O6—Si2—C15	104.44 (18)	O44—Si41—C51	109.86 (15)
C13—Si2—C15	110.0 (2)	C49—Si41—C51	114.41 (18)
C17—Si2—C15	110.9 (2)	C47—Si41—C51	108.74 (16)
O1—C1—N1	108.9 (3)	O46—Si42—C53	109.09 (17)
O1—C1—C2	106.3 (3)	O46—Si42—C55	104.27 (18)
N1—C1—C2	109.3 (3)	C53—Si42—C55	111.2 (2)
O1—C1—H1	110.8	O46—Si42—C57	109.77 (16)
N1—C1—H1	110.8	C53—Si42—C57	111.80 (19)
C2—C1—H1	110.8	C55—Si42—C57	110.48 (19)
O1—C1A—C4	113.7 (3)	O41—C41—N41	108.9 (3)
O1—C1A—C2A	106.6 (3)	O41—C41—C42	105.5 (3)
C4—C1A—C2A	102.9 (3)	N41—C41—C42	109.3 (3)
O1—C1A—H1A	111.1	O41—C41—H41	111.0
C4—C1A—H1A	111.1	N41—C41—H41	111.0
C2A—C1A—H1A	111.1	C42—C41—H41	111.0
O3—N1—C5	105.1 (3)	O41—C41A—C44	113.7 (3)
O3—N1—C1	102.8 (3)	O41—C41A—C42A	106.9 (3)
C5—N1—C1	112.0 (3)	C44—C41A—C42A	102.5 (3)
C1—O1—C1A	110.2 (3)	O41—C41A—H41A	111.1
O4—C2—C1	108.8 (3)	C44—C41A—H41A	111.1
O4—C2—C2A	110.2 (3)	C42A—C41A—H41A	111.1
C1—C2—C2A	102.5 (3)	O43—N41—C45	105.9 (3)
O4—C2—H2	111.7	O43—N41—C41	103.1 (2)
C1—C2—H2	111.7	C45—N41—C41	110.9 (3)
C2A—C2—H2	111.7	C41—O41—C41A	110.4 (3)
O2—C2A—C2	110.8 (3)	O44—C42—C42A	109.1 (3)
O2—C2A—C1A	107.4 (3)	O44—C42—C41	108.8 (3)

C2—C2A—C1A	104.3 (3)	C42A—C42—C41	101.9 (3)
O2—C2A—H2A	111.3	O44—C42—H42	112.1
C2—C2A—H2A	111.3	C42A—C42—H42	112.1
C1A—C2A—H2A	111.3	C41—C42—H42	112.1
C3—O2—C2A	112.5 (3)	O42—C42A—C42	111.0 (3)
O5—C3—O2	121.8 (4)	O42—C42A—C41A	107.7 (3)
O5—C3—C4	129.1 (4)	C42—C42A—C41A	103.8 (3)
O2—C3—C4	108.9 (3)	O42—C42A—H42A	111.4
C6—O3—N1	108.8 (3)	C42—C42A—H42A	111.4
O6—C4—C1A	114.9 (3)	C41A—C42A—H42A	111.4
O6—C4—C3	111.0 (3)	C43—O42—C42A	112.7 (3)
C1A—C4—C3	105.3 (3)	O45—C43—O42	121.8 (4)
O6—C4—H4	108.5	O45—C43—C44	129.5 (4)
C1A—C4—H4	108.5	O42—C43—C44	108.4 (3)
C3—C4—H4	108.5	C46—O43—N41	108.7 (3)
C2—O4—Si1	122.6 (3)	O46—C44—C41A	115.2 (3)
N1—C5—H5A	109.5	O46—C44—C43	111.2 (3)
N1—C5—H5B	109.5	C41A—C44—C43	105.6 (3)
H5A—C5—H5B	109.5	O46—C44—H44	108.2
N1—C5—H5C	109.5	C41A—C44—H44	108.2
H5A—C5—H5C	109.5	C43—C44—H44	108.2
H5B—C5—H5C	109.5	C42—O44—Si41	124.9 (2)
O3—C6—H6A	109.5	N41—C45—H45A	109.5
O3—C6—H6B	109.5	N41—C45—H45B	109.5
H6A—C6—H6B	109.5	H45A—C45—H45B	109.5
O3—C6—H6C	109.5	N41—C45—H45C	109.5
H6A—C6—H6C	109.5	H45A—C45—H45C	109.5
H6B—C6—H6C	109.5	H45B—C45—H45C	109.5
C4—O6—Si2	125.3 (2)	O43—C46—H46A	109.5
C8—C7—Si1	114.3 (3)	O43—C46—H46B	109.5
C8—C7—H7A	108.7	H46A—C46—H46B	109.5
Si1—C7—H7A	108.7	O43—C46—H46C	109.5
C8—C7—H7B	108.7	H46A—C46—H46C	109.5
Si1—C7—H7B	108.7	H46B—C46—H46C	109.5
H7A—C7—H7B	107.6	C44—O46—Si42	125.0 (2)
C7—C8—H8A	109.5	C48—C47—Si41	117.1 (3)
C7—C8—H8B	109.5	C48—C47—H47A	108.0
H8A—C8—H8B	109.5	Si41—C47—H47A	108.0
C7—C8—H8C	109.5	C48—C47—H47B	108.0
H8A—C8—H8C	109.5	Si41—C47—H47B	108.0
H8B—C8—H8C	109.5	H47A—C47—H47B	107.3
C10—C9—Si1	114.2 (3)	C47—C48—H48A	109.5
C10—C9—H9A	108.7	C47—C48—H48B	109.5
Si1—C9—H9A	108.7	H48A—C48—H48B	109.5
C10—C9—H9B	108.7	C47—C48—H48C	109.5
Si1—C9—H9B	108.7	H48A—C48—H48C	109.5
H9A—C9—H9B	107.6	H48B—C48—H48C	109.5
C9—C10—H10A	109.5	C50—C49—Si41	114.2 (3)
C9—C10—H10B	109.5	C50—C49—H49A	108.7

H10A—C10—H10B	109.5	Si41—C49—H49A	108.7
C9—C10—H10C	109.5	C50—C49—H49B	108.7
H10A—C10—H10C	109.5	Si41—C49—H49B	108.7
H10B—C10—H10C	109.5	H49A—C49—H49B	107.6
C12—C11—Si1	116.6 (3)	C49—C50—H50A	109.5
C12—C11—H11A	108.1	C49—C50—H50B	109.5
Si1—C11—H11A	108.1	H50A—C50—H50B	109.5
C12—C11—H11B	108.1	C49—C50—H50C	109.5
Si1—C11—H11B	108.1	H50A—C50—H50C	109.5
H11A—C11—H11B	107.3	H50B—C50—H50C	109.5
C11—C12—H12A	109.5	C52—C51—Si41	113.9 (3)
C11—C12—H12B	109.5	C52—C51—H51A	108.8
H12A—C12—H12B	109.5	Si41—C51—H51A	108.8
C11—C12—H12C	109.5	C52—C51—H51B	108.8
H12A—C12—H12C	109.5	Si41—C51—H51B	108.8
H12B—C12—H12C	109.5	H51A—C51—H51B	107.7
C14—C13—Si2	115.2 (3)	C51—C52—H52A	109.5
C14—C13—H13A	108.5	C51—C52—H52B	109.5
Si2—C13—H13A	108.5	H52A—C52—H52B	109.5
C14—C13—H13B	108.5	C51—C52—H52C	109.5
Si2—C13—H13B	108.5	H52A—C52—H52C	109.5
H13A—C13—H13B	107.5	H52B—C52—H52C	109.5
C13—C14—H14A	109.5	C54—C53—Si42	114.3 (3)
C13—C14—H14B	109.5	C54—C53—H53A	108.7
H14A—C14—H14B	109.5	Si42—C53—H53A	108.7
C13—C14—H14C	109.5	C54—C53—H53B	108.7
H14A—C14—H14C	109.5	Si42—C53—H53B	108.7
H14B—C14—H14C	109.5	H53A—C53—H53B	107.6
C16—C15—Si2	117.5 (3)	C53—C54—H54A	109.5
C16—C15—H15A	107.9	C53—C54—H54B	109.5
Si2—C15—H15A	107.9	H54A—C54—H54B	109.5
C16—C15—H15B	107.9	C53—C54—H54C	109.5
Si2—C15—H15B	107.9	H54A—C54—H54C	109.5
H15A—C15—H15B	107.2	H54B—C54—H54C	109.5
C15—C16—H16A	109.5	C56—C55—Si42	117.1 (3)
C15—C16—H16B	109.5	C56—C55—H55A	108.0
H16A—C16—H16B	109.5	Si42—C55—H55A	108.0
C15—C16—H16C	109.5	C56—C55—H55B	108.0
H16A—C16—H16C	109.5	Si42—C55—H55B	108.0
H16B—C16—H16C	109.5	H55A—C55—H55B	107.3
C18—C17—Si2	114.0 (3)	C55—C56—H56A	109.5
C18—C17—H17A	108.8	C55—C56—H56B	109.5
Si2—C17—H17A	108.8	H56A—C56—H56B	109.5
C18—C17—H17B	108.8	C55—C56—H56C	109.5
Si2—C17—H17B	108.8	H56A—C56—H56C	109.5
H17A—C17—H17B	107.6	H56B—C56—H56C	109.5
C17—C18—H18A	109.5	C58—C57—Si42	113.7 (3)
C17—C18—H18B	109.5	C58—C57—H57A	108.8
H18A—C18—H18B	109.5	Si42—C57—H57A	108.8

C17—C18—H18C	109.5	C58—C57—H57B	108.8
H18A—C18—H18C	109.5	Si42—C57—H57B	108.8
H18B—C18—H18C	109.5	H57A—C57—H57B	107.7
O24—Si21—C29	103.75 (17)	C57—C58—H58A	109.5
O24—Si21—C27	109.78 (16)	C57—C58—H58B	109.5
C29—Si21—C27	112.73 (18)	H58A—C58—H58B	109.5
O24—Si21—C31	111.2 (2)	C57—C58—H58C	109.5
C29—Si21—C31	110.4 (2)	H58A—C58—H58C	109.5
C27—Si21—C31	108.93 (18)	H58B—C58—H58C	109.5
O26—Si22—C35	105.86 (19)	O64—Si61—C67	112.89 (17)
O26—Si22—C37	110.40 (17)	O64—Si61—C71	108.71 (15)
C35—Si22—C37	109.7 (2)	C67—Si61—C71	109.10 (17)
O26—Si22—C33	108.2 (2)	O64—Si61—C69	103.81 (18)
C35—Si22—C33	111.8 (2)	C67—Si61—C69	109.84 (18)
C37—Si22—C33	110.8 (3)	C71—Si61—C69	112.47 (19)
O21—C21—N21	107.9 (4)	O66—Si62—C75	106.31 (18)
O21—C21—C22	104.8 (3)	O66—Si62—C77	110.14 (18)
N21—C21—C22	110.9 (3)	C75—Si62—C77	109.83 (19)
O21—C21—H21	111.0	O66—Si62—C73	109.2 (2)
N21—C21—H21	111.0	C75—Si62—C73	110.6 (2)
C22—C21—H21	111.0	C77—Si62—C73	110.7 (2)
O21—C21A—C22A	107.4 (3)	O61—C61—N61	107.5 (3)
O21—C21A—C24	113.4 (3)	O61—C61—C62	104.9 (3)
C22A—C21A—C24	104.5 (3)	N61—C61—C62	111.4 (3)
O21—C21A—H21A	110.4	O61—C61—H61	110.9
C22A—C21A—H21A	110.4	N61—C61—H61	110.9
C24—C21A—H21A	110.4	C62—C61—H61	110.9
O23—N21—C21	102.6 (3)	O61—C61A—C62A	107.2 (3)
O23—N21—C25	106.2 (3)	O61—C61A—C64	113.2 (3)
C21—N21—C25	111.1 (3)	C62A—C61A—C64	104.5 (3)
C21A—O21—C21	109.7 (3)	O61—C61A—H61A	110.6
O24—C22—C21	108.9 (3)	C62A—C61A—H61A	110.6
O24—C22—C22A	108.0 (3)	C64—C61A—H61A	110.6
C21—C22—C22A	103.4 (3)	O63—N61—C61	102.5 (3)
O24—C22—H22	112.0	O63—N61—C65	106.1 (3)
C21—C22—H22	112.0	C61—N61—C65	111.3 (3)
C22A—C22—H22	112.0	C61A—O61—C61	109.7 (3)
O22—C22A—C22	109.2 (3)	O64—C62—C61	109.8 (3)
O22—C22A—C21A	106.9 (3)	O64—C62—C62A	107.5 (3)
C22—C22A—C21A	104.1 (3)	C61—C62—C62A	103.2 (3)
O22—C22A—H22A	112.1	O64—C62—H62	112.0
C22—C22A—H22A	112.1	C61—C62—H62	112.0
C21A—C22A—H22A	112.1	C62A—C62—H62	112.0
C23—O22—C22A	111.8 (3)	O62—C62A—C61A	106.7 (3)
O25—C23—O22	121.7 (4)	O62—C62A—C62	109.2 (3)
O25—C23—C24	128.0 (4)	C61A—C62A—C62	103.9 (3)
O22—C23—C24	110.2 (4)	O62—C62A—H62A	112.2
C26—O23—N21	107.5 (3)	C61A—C62A—H62A	112.2
O26—C24—C23	111.5 (3)	C62—C62A—H62A	112.2

O26—C24—C21A	115.4 (3)	C63—O62—C62A	111.8 (3)
C23—C24—C21A	103.8 (3)	O65—C63—O62	122.1 (4)
O26—C24—H24	108.6	O65—C63—C64	127.6 (4)
C23—C24—H24	108.6	O62—C63—C64	110.3 (4)
C21A—C24—H24	108.6	C66—O63—N61	107.6 (3)
C22—O24—Si21	123.7 (3)	O66—C64—C63	111.9 (3)
N21—C25—H25A	109.5	O66—C64—C61A	115.6 (3)
N21—C25—H25B	109.5	C63—C64—C61A	103.8 (3)
H25A—C25—H25B	109.5	O66—C64—H64	108.4
N21—C25—H25C	109.5	C63—C64—H64	108.4
H25A—C25—H25C	109.5	C61A—C64—H64	108.4
H25B—C25—H25C	109.5	C62—O64—Si61	125.7 (2)
O23—C26—H26A	109.5	N61—C65—H65A	109.5
O23—C26—H26B	109.5	N61—C65—H65B	109.5
H26A—C26—H26B	109.5	H65A—C65—H65B	109.5
O23—C26—H26C	109.5	N61—C65—H65C	109.5
H26A—C26—H26C	109.5	H65A—C65—H65C	109.5
H26B—C26—H26C	109.5	H65B—C65—H65C	109.5
C24—O26—Si22	124.2 (3)	O63—C66—H66A	109.5
C28—C27—Si21	114.9 (3)	O63—C66—H66B	109.5
C28—C27—H27A	108.5	H66A—C66—H66B	109.5
Si21—C27—H27A	108.5	O63—C66—H66C	109.5
C28—C27—H27B	108.5	H66A—C66—H66C	109.5
Si21—C27—H27B	108.5	H66B—C66—H66C	109.5
H27A—C27—H27B	107.5	C64—O66—Si62	124.8 (3)
C27—C28—H28A	109.5	C68—C67—Si61	117.5 (3)
C27—C28—H28B	109.5	C68—C67—H67A	107.9
H28A—C28—H28B	109.5	Si61—C67—H67A	107.9
C27—C28—H28C	109.5	C68—C67—H67B	107.9
H28A—C28—H28C	109.5	Si61—C67—H67B	107.9
H28B—C28—H28C	109.5	H67A—C67—H67B	107.2
C30—C29—Si21	116.0 (4)	C67—C68—H68A	109.5
C30—C29—H29A	108.3	C67—C68—H68B	109.5
Si21—C29—H29A	108.3	H68A—C68—H68B	109.5
C30—C29—H29B	108.3	C67—C68—H68C	109.5
Si21—C29—H29B	108.3	H68A—C68—H68C	109.5
H29A—C29—H29B	107.4	H68B—C68—H68C	109.5
C29—C30—H30A	109.5	C70—C69—Si61	115.1 (3)
C29—C30—H30B	109.5	C70—C69—H69A	108.5
H30A—C30—H30B	109.5	Si61—C69—H69A	108.5
C29—C30—H30C	109.5	C70—C69—H69B	108.5
H30A—C30—H30C	109.5	Si61—C69—H69B	108.5
H30B—C30—H30C	109.5	H69A—C69—H69B	107.5
C32B—C31—Si21	121.5 (6)	C69—C70—H70A	109.5
C32—C31—Si21	117.9 (4)	C69—C70—H70B	109.5
C32—C31—H31A	107.8	H70A—C70—H70B	109.5
Si21—C31—H31A	107.8	C69—C70—H70C	109.5
C32—C31—H31B	107.8	H70A—C70—H70C	109.5
Si21—C31—H31B	107.8	H70B—C70—H70C	109.5

H31A—C31—H31B	107.2	C72—C71—Si61	115.4 (3)
C32B—C31—H31C	107.0	C72—C71—H71A	108.4
Si21—C31—H31C	107.0	Si61—C71—H71A	108.4
C32B—C31—H31D	107.0	C72—C71—H71B	108.4
Si21—C31—H31D	107.0	Si61—C71—H71B	108.4
H31C—C31—H31D	106.7	H71A—C71—H71B	107.5
C31—C32—H32A	109.5	C71—C72—H72A	109.5
C31—C32—H32B	109.5	C71—C72—H72B	109.5
H32A—C32—H32B	109.5	H72A—C72—H72B	109.5
C31—C32—H32C	109.5	C71—C72—H72C	109.5
H32A—C32—H32C	109.5	H72A—C72—H72C	109.5
H32B—C32—H32C	109.5	H72B—C72—H72C	109.5
C31—C32B—H32D	109.5	C74—C73—Si62	115.7 (4)
C31—C32B—H32E	109.5	C74—C73—H73A	108.4
H32D—C32B—H32E	109.5	Si62—C73—H73A	108.4
C31—C32B—H32F	109.5	C74—C73—H73B	108.4
H32D—C32B—H32F	109.5	Si62—C73—H73B	108.4
H32E—C32B—H32F	109.5	H73A—C73—H73B	107.4
C34—C33—Si22	115.7 (3)	C73—C74—H74A	109.5
C34—C33—H33A	108.4	C73—C74—H74B	109.5
Si22—C33—H33A	108.4	H74A—C74—H74B	109.5
C34—C33—H33B	108.4	C73—C74—H74C	109.5
Si22—C33—H33B	108.4	H74A—C74—H74C	109.5
H33A—C33—H33B	107.4	H74B—C74—H74C	109.5
C33—C34—H34A	109.5	C76—C75—Si62	115.0 (3)
C33—C34—H34B	109.5	C76—C75—H75A	108.5
H34A—C34—H34B	109.5	Si62—C75—H75A	108.5
C33—C34—H34C	109.5	C76—C75—H75B	108.5
H34A—C34—H34C	109.5	Si62—C75—H75B	108.5
H34B—C34—H34C	109.5	H75A—C75—H75B	107.5
C36—C35—Si22	114.3 (3)	C75—C76—H76A	109.5
C36—C35—H35A	108.7	C75—C76—H76B	109.5
Si22—C35—H35A	108.7	H76A—C76—H76B	109.5
C36—C35—H35B	108.7	C75—C76—H76C	109.5
Si22—C35—H35B	108.7	H76A—C76—H76C	109.5
H35A—C35—H35B	107.6	H76B—C76—H76C	109.5
C35—C36—H36A	109.5	C78—C77—Si62	113.4 (3)
C35—C36—H36B	109.5	C78—C77—H77A	108.9
H36A—C36—H36B	109.5	Si62—C77—H77A	108.9
C35—C36—H36C	109.5	C78—C77—H77B	108.9
H36A—C36—H36C	109.5	Si62—C77—H77B	108.9
H36B—C36—H36C	109.5	H77A—C77—H77B	107.7
C38—C37—Si22	113.3 (3)	C77—C78—H78A	109.5
C38—C37—H37A	108.9	C77—C78—H78B	109.5
Si22—C37—H37A	108.9	H78A—C78—H78B	109.5
C38—C37—H37B	108.9	C77—C78—H78C	109.5
Si22—C37—H37B	108.9	H78A—C78—H78C	109.5
H37A—C37—H37B	107.7	H78B—C78—H78C	109.5

O1—C1—N1—O3	−173.9 (3)	C33—Si22—C37—C38	172.6 (3)
C2—C1—N1—O3	70.4 (3)	O41—C41—N41—O43	−176.2 (3)
O1—C1—N1—C5	−61.6 (4)	C42—C41—N41—O43	69.0 (3)
C2—C1—N1—C5	−177.3 (3)	O41—C41—N41—C45	−63.3 (4)
N1—C1—O1—C1A	−89.6 (3)	C42—C41—N41—C45	−178.1 (3)
C2—C1—O1—C1A	28.1 (4)	N41—C41—O41—C41A	−86.8 (4)
C4—C1A—O1—C1	100.9 (4)	C42—C41—O41—C41A	30.4 (4)
C2A—C1A—O1—C1	−11.8 (4)	C44—C41A—O41—C41	98.8 (4)
O1—C1—C2—O4	84.5 (3)	C42A—C41A—O41—C41	−13.5 (4)
N1—C1—C2—O4	−158.1 (3)	O41—C41—C42—O44	81.0 (3)
O1—C1—C2—C2A	−32.2 (4)	N41—C41—C42—O44	−162.1 (3)
N1—C1—C2—C2A	85.2 (3)	O41—C41—C42—C42A	−34.2 (4)
O4—C2—C2A—O2	153.2 (3)	N41—C41—C42—C42A	82.7 (4)
C1—C2—C2A—O2	−91.1 (3)	O44—C42—C42A—O42	154.8 (3)
O4—C2—C2A—C1A	−91.4 (4)	C41—C42—C42A—O42	−90.2 (3)
C1—C2—C2A—C1A	24.2 (4)	O44—C42—C42A—C41A	−89.8 (3)
O1—C1A—C2A—O2	108.7 (3)	C41—C42—C42A—C41A	25.2 (4)
C4—C1A—C2A—O2	−11.2 (3)	O41—C41A—C42A—O42	108.9 (3)
O1—C1A—C2A—C2	−9.0 (4)	C44—C41A—C42A—O42	−11.0 (3)
C4—C1A—C2A—C2	−128.9 (3)	O41—C41A—C42A—C42	−8.9 (4)
C2—C2A—O2—C3	114.4 (3)	C44—C41A—C42A—C42	−128.7 (3)
C1A—C2A—O2—C3	1.0 (4)	C42—C42A—O42—C43	113.3 (3)
C2A—O2—C3—O5	−174.8 (3)	C41A—C42A—O42—C43	0.3 (4)
C2A—O2—C3—C4	9.8 (4)	C42A—O42—C43—O45	−174.8 (3)
C5—N1—O3—C6	106.2 (3)	C42A—O42—C43—C44	10.8 (4)
C1—N1—O3—C6	−136.5 (3)	C45—N41—O43—C46	105.4 (3)
O1—C1A—C4—O6	23.7 (5)	C41—N41—O43—C46	−138.0 (3)
C2A—C1A—C4—O6	138.5 (3)	O41—C41A—C44—O46	24.7 (5)
O1—C1A—C4—C3	−98.8 (3)	C42A—C41A—C44—O46	139.6 (3)
C2A—C1A—C4—C3	16.1 (3)	O41—C41A—C44—C43	−98.4 (3)
O5—C3—C4—O6	43.3 (5)	C42A—C41A—C44—C43	16.5 (3)
O2—C3—C4—O6	−141.7 (3)	O45—C43—C44—O46	42.9 (5)
O5—C3—C4—C1A	168.3 (4)	O42—C43—C44—O46	−143.2 (3)
O2—C3—C4—C1A	−16.7 (3)	O45—C43—C44—C41A	168.6 (4)
C1—C2—O4—Si1	134.6 (3)	O42—C43—C44—C41A	−17.6 (4)
C2A—C2—O4—Si1	−113.8 (3)	C42A—C42—O44—Si41	−131.9 (3)
C7—Si1—O4—C2	−55.3 (3)	C41—C42—O44—Si41	117.6 (3)
C9—Si1—O4—C2	−175.8 (3)	C49—Si41—O44—C42	175.8 (3)
C11—Si1—O4—C2	63.6 (3)	C47—Si41—O44—C42	−66.9 (3)
C1A—C4—O6—Si2	106.7 (3)	C51—Si41—O44—C42	54.1 (3)
C3—C4—O6—Si2	−134.0 (3)	C41A—C44—O46—Si42	112.8 (3)
C13—Si2—O6—C4	79.9 (3)	C43—C44—O46—Si42	−127.1 (3)
C17—Si2—O6—C4	−43.6 (3)	C53—Si42—O46—C44	76.7 (3)
C15—Si2—O6—C4	−162.4 (3)	C55—Si42—O46—C44	−164.5 (3)
O4—Si1—C7—C8	−54.0 (3)	C57—Si42—O46—C44	−46.1 (3)
C9—Si1—C7—C8	59.8 (3)	O44—Si41—C47—C48	83.1 (3)
C11—Si1—C7—C8	−171.8 (3)	C49—Si41—C47—C48	−164.4 (3)
O4—Si1—C9—C10	−167.9 (3)	C51—Si41—C47—C48	−38.5 (3)
C7—Si1—C9—C10	73.6 (3)	O44—Si41—C49—C50	−167.7 (3)

C11—Si1—C9—C10	−52.0 (3)	C47—Si41—C49—C50	73.4 (3)
O4—Si1—C11—C12	−145.3 (4)	C51—Si41—C49—C50	−49.2 (3)
C7—Si1—C11—C12	−25.8 (4)	O44—Si41—C51—C52	55.4 (3)
C9—Si1—C11—C12	101.9 (4)	C49—Si41—C51—C52	−58.5 (3)
O6—Si2—C13—C14	−63.3 (4)	C47—Si41—C51—C52	178.5 (3)
C17—Si2—C13—C14	58.5 (4)	O46—Si42—C53—C54	−59.2 (3)
C15—Si2—C13—C14	−177.5 (4)	C55—Si42—C53—C54	−173.7 (3)
O6—Si2—C15—C16	−56.6 (4)	C57—Si42—C53—C54	62.4 (4)
C13—Si2—C15—C16	60.7 (4)	O46—Si42—C55—C56	−57.4 (4)
C17—Si2—C15—C16	−174.5 (3)	C53—Si42—C55—C56	60.0 (4)
O6—Si2—C17—C18	−67.4 (3)	C57—Si42—C55—C56	−175.2 (3)
C13—Si2—C17—C18	170.8 (3)	O46—Si42—C57—C58	−67.0 (3)
C15—Si2—C17—C18	47.3 (4)	C53—Si42—C57—C58	171.8 (3)
O21—C21—N21—O23	−175.6 (3)	C55—Si42—C57—C58	47.4 (4)
C22—C21—N21—O23	70.2 (4)	O61—C61—N61—O63	−174.6 (3)
O21—C21—N21—C25	−62.5 (4)	C62—C61—N61—O63	71.0 (4)
C22—C21—N21—C25	−176.7 (3)	O61—C61—N61—C65	−61.5 (4)
C22A—C21A—O21—C21	−14.3 (5)	C62—C61—N61—C65	−176.0 (3)
C24—C21A—O21—C21	100.6 (4)	C62A—C61A—O61—C61	−14.3 (5)
N21—C21—O21—C21A	−88.5 (4)	C64—C61A—O61—C61	100.4 (4)
C22—C21—O21—C21A	29.8 (4)	N61—C61—O61—C61A	−88.3 (4)
O21—C21—C22—O24	82.0 (4)	C62—C61—O61—C61A	30.4 (4)
N21—C21—C22—O24	−161.8 (3)	O61—C61—C62—O64	81.0 (4)
O21—C21—C22—C22A	−32.6 (4)	N61—C61—C62—O64	−162.9 (3)
N21—C21—C22—C22A	83.6 (4)	O61—C61—C62—C62A	−33.4 (4)
O24—C22—C22A—O22	154.6 (3)	N61—C61—C62—C62A	82.7 (4)
C21—C22—C22A—O22	−90.1 (4)	O61—C61A—C62A—O62	108.1 (3)
O24—C22—C22A—C21A	−91.6 (4)	C64—C61A—C62A—O62	−12.3 (4)
C21—C22—C22A—C21A	23.7 (4)	O61—C61A—C62A—C62	−7.2 (4)
O21—C21A—C22A—O22	108.6 (3)	C64—C61A—C62A—C62	−127.6 (3)
C24—C21A—C22A—O22	−12.1 (4)	O64—C62—C62A—O62	154.8 (3)
O21—C21A—C22A—C22	−6.8 (4)	C61—C62—C62A—O62	−89.1 (3)
C24—C21A—C22A—C22	−127.6 (3)	O64—C62—C62A—C61A	−91.6 (3)
C22—C22A—O22—C23	114.9 (3)	C61—C62—C62A—C61A	24.4 (4)
C21A—C22A—O22—C23	2.9 (4)	C61A—C62A—O62—C63	2.4 (4)
C22A—O22—C23—O25	−174.7 (3)	C62—C62A—O62—C63	114.1 (3)
C22A—O22—C23—C24	8.2 (4)	C62A—O62—C63—O65	−174.5 (3)
C21—N21—O23—C26	−135.4 (3)	C62A—O62—C63—C64	9.0 (4)
C25—N21—O23—C26	108.0 (3)	C61—N61—O63—C66	−134.7 (3)
O25—C23—C24—O26	42.8 (5)	C65—N61—O63—C66	108.4 (3)
O22—C23—C24—O26	−140.3 (3)	O65—C63—C64—O66	42.2 (5)
O25—C23—C24—C21A	167.8 (4)	O62—C63—C64—O66	−141.6 (3)
O22—C23—C24—C21A	−15.4 (4)	O65—C63—C64—C61A	167.5 (4)
O21—C21A—C24—O26	21.8 (5)	O62—C63—C64—C61A	−16.3 (4)
C22A—C21A—C24—O26	138.5 (4)	O61—C61A—C64—O66	23.4 (5)
O21—C21A—C24—C23	−100.5 (4)	C62A—C61A—C64—O66	139.6 (4)
C22A—C21A—C24—C23	16.1 (4)	O61—C61A—C64—C63	−99.5 (4)
C21—C22—O24—Si21	132.0 (3)	C62A—C61A—C64—C63	16.7 (4)
C22A—C22—O24—Si21	−116.4 (3)	C61—C62—O64—Si61	113.7 (3)

C29—Si21—O24—C22	−175.2 (3)	C62A—C62—O64—Si61	−134.6 (3)
C27—Si21—O24—C22	−54.5 (3)	C67—Si61—O64—C62	−65.3 (3)
C31—Si21—O24—C22	66.1 (3)	C71—Si61—O64—C62	55.9 (3)
C23—C24—O26—Si22	−130.6 (3)	C69—Si61—O64—C62	175.8 (3)
C21A—C24—O26—Si22	111.3 (4)	C63—C64—O66—Si62	−129.0 (3)
C35—Si22—O26—C24	−163.6 (3)	C61A—C64—O66—Si62	112.5 (4)
C37—Si22—O26—C24	−45.0 (3)	C75—Si62—O66—C64	−164.3 (3)
C33—Si22—O26—C24	76.4 (3)	C77—Si62—O66—C64	−45.4 (3)
O24—Si21—C27—C28	−53.9 (4)	C73—Si62—O66—C64	76.4 (4)
C29—Si21—C27—C28	61.2 (4)	O64—Si61—C67—C68	84.7 (3)
C31—Si21—C27—C28	−175.9 (3)	C71—Si61—C67—C68	−36.3 (4)
O24—Si21—C29—C30	−168.4 (3)	C69—Si61—C67—C68	−160.0 (3)
C27—Si21—C29—C30	72.9 (4)	O64—Si61—C69—C70	−170.3 (3)
C31—Si21—C29—C30	−49.2 (4)	C67—Si61—C69—C70	68.7 (3)
O24—Si21—C31—C32B	−128.9 (8)	C71—Si61—C69—C70	−53.0 (3)
C29—Si21—C31—C32B	116.5 (8)	O64—Si61—C71—C72	56.1 (3)
C27—Si21—C31—C32B	−7.8 (9)	C67—Si61—C71—C72	179.6 (3)
O24—Si21—C31—C32	−83.8 (5)	C69—Si61—C71—C72	−58.2 (3)
C29—Si21—C31—C32	161.6 (4)	O66—Si62—C73—C74	−69.0 (5)
C27—Si21—C31—C32	37.3 (5)	C75—Si62—C73—C74	174.3 (5)
O26—Si22—C33—C34	−59.6 (6)	C77—Si62—C73—C74	52.4 (6)
C35—Si22—C33—C34	−175.7 (5)	O66—Si62—C75—C76	−60.0 (3)
C37—Si22—C33—C34	61.5 (6)	C77—Si62—C75—C76	−179.2 (3)
O26—Si22—C35—C36	−58.9 (4)	C73—Si62—C75—C76	58.4 (4)
C37—Si22—C35—C36	−178.0 (4)	O66—Si62—C77—C78	−67.6 (3)
C33—Si22—C35—C36	58.7 (5)	C75—Si62—C77—C78	49.2 (4)
O26—Si22—C37—C38	−67.6 (3)	C73—Si62—C77—C78	171.6 (4)
C35—Si22—C37—C38	48.7 (4)		