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(18-Crown-6-κ^6O){(trimethylsilyl)[6-(trimethylsilylamino)pyridin-2-yl-κN^1]azanido-κN}potassium(I)

Sydney E. Toney,[a] Patrick C. Hillesheim[b] and Gary L. Guillet[a]*

[a]Department of Chemistry and Physics, Armstrong State University, Savannah, GA 31419, USA, and [b]Department of Chemistry, The University of Tennessee, Knoxville, TN, USA. *Correspondence e-mail: gary.guillet@armstrong.edu

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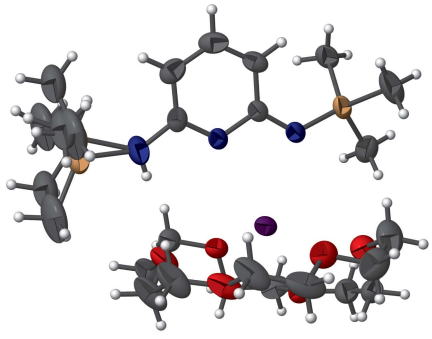
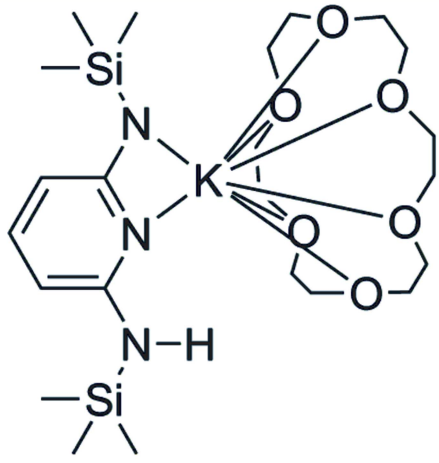
Structural data: full structural data are available from iucrdata.iucr.org

The title complex, $[K(C_{11}H_{22}N_3Si_2)(C_{12}H_{24}O_6)]$, contains a single K^+ cation coordinated by a monoanionic (6-trimethylsilylaminopyridin-2-yl)(trimethylsilyl)azanide (H*L*) ligand and an 18-crown-6 ligand. One $SiMe_3$ group in H*L* is disordered over two positions, with an occupancy ratio of 0.727 (7):0.273 (7). The K^+ cation is eight-coordinated, distributed between the pyridyl and azanide N atoms of H*L*, forming a four-membered ring, and a κ^6 18-crown-6 ligand. The complex is best described as a contact ion pair and, unlike other similar literature examples, this complex does not dimerize. No intermolecular hydrogen bonding is observed in the crystal structure, likely indicating that weak electrostatic interactions are the dominant feature directing the crystal packing.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is a monometallic complex comprised of a K^+ cation that is eight-coordinate bonded to a monoanionic 6-(trimethylsilylamino)pyridin-2-yl-(trimethylsilyl)azanido ligand *via* the pyridyl nitrogen (N2) and the azanido nitrogen (N1), as well as to an 18-crown-6 ligand *via* six O atoms (O1–O6). The complex was synthesized by combining 2,6-bis(trimethylsilylamino)pyridine with excess potassium hydride and 18-crown-6 in tetrahydrofuran (THF) and is best described as a contact ion pair.

There are two prior comparable complexes formed from the similar 2-trimethylsilylaminopyridine ligand and a crown ether derivative, one utilizing 12-crown-4 (Liddle & Clegg, 2001) and the other 15-crown-5 (Liddle & Clegg, 2002). In contrast to the title complex, these structures both form dimers in the crystal structure. The dimer formed with 12-crown-4 bridges the two K^+ cations by the pyridyl and azanido N atoms of the 2-trimethylsilylazanidopyridine, while the dimer formed with 15-crown-5 bridges instead *via* a single O atom in the 15-crown-5 ligand. This difference in the 15-crown-5 complex is

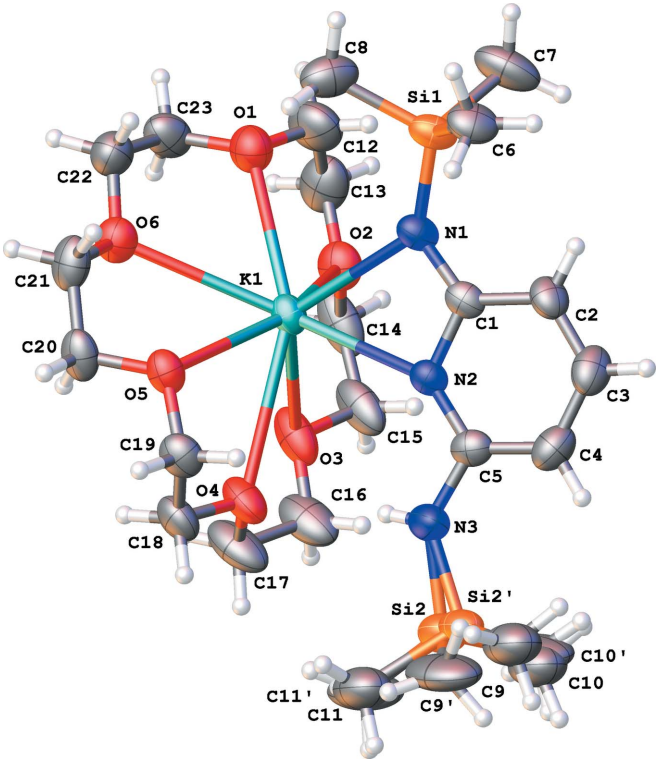


Figure 1
A view of the molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[K(C_{11}H_{22}N_3Si_2)(C_{12}H_{24}O_6)]$
M_r	555.91
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	173
a, *b*, *c* (Å)	20.3301 (10), 17.4558 (6), 20.5691 (11)
β (°)	119.399 (7)
V (Å^3)	6359.5 (6)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.28
Crystal size (mm)	0.56 × 0.36 × 0.29
Data collection	
Diffractometer	Rigaku XtaLAB mini
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.797, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	27816, 5770, 4565
R_{int}	0.046
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.600
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.140, 1.05
No. of reflections	5770
No. of parameters	350
No. of restraints	6
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.64, −0.28

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

attributed by the authors to the increased steric interaction between the bulkier 15-crown-5 and the trimethylsilyl moiety, labelling it a 'slipped' dimer. The K—$N_{pyridyl}$ bond length in the title complex, K1—N2 = 2.858 (2) Å, fits in the range set by these two complexes (2.793–2.858 Å) as does the K—$N_{azanido}$ bond length, K1—N1 = 2.822 (2) Å with the range being 2.800–2.912 Å for the prior complexes. The bonds between the potassium and the crown ether O atoms cover a similar range [2.770 (2)–3.049 (2) Å] to those observed for the 12-crown-4 complex [2.778–3.227 Å] and the 15-crown-5 complex [2.776–2.875 Å]. This is unsurprising since the K^+ cation in all three cases is eight-coordinate.

One stark difference is the distance the K^+ cation sits above the mean plane of the crown ether O atoms. In the title complex the distance is 1.100 (1) Å, while in the 12-crown-4 complex it is 2.535 Å and in the 15-crown-5 complex it is 1.558 Å. It is commonly accepted that K^+ has close to an ideal fit within the 18-crown-6 molecule compared to other crown ether derivatives like 12-crown-4 or 15-crown-5. This is supported by a related complex reported with the less sterically demanding 2-phenylamidopyridine ligand and dibenzo-18-crown-6. This complex exhibits the same coordination environment about the K^+ cation as observed in the title complex with similar K—N bond lengths for the $N_{Pyridyl}$ and $N_{Azanido}$ of 2.852 and 2.794 Å, respectively, and the K^+ cation in this case sits 0.783 Å above the mean plane of O atoms of the dibenzo-18-crown-6 (Clegg & Liddle, 2004). The shorter K^+–crown distance compared to the related distance in the title complex could be attributed to the closer approach of the dibenzo-18-crown-6 to the less sterically bulky 2-phenylaminopyridine ligand compared to the 6-(trimethylsilylamino)pyridin-2-yl-(trimethylsilyl)azanido ligand used in this work.

Synthesis and crystallization

The 2,6-bis(trimethylsilylamino)pyridine used herein was synthesized according to a previous report (Danièle *et al.*, 2001). The synthesis of the title complex was carried out in an air-free environment in an N_2-filled glovebox. 2,6-Bis(trimethylsilylamino)pyridine (125 mg, 0.493 mmol) was dissolved in 5 ml of THF. Approximately two equivalents of potassium hydride (40 mg, 0.998 mmol) and two equivalents of 18-crown-6 (261 mg, 0.987 mmol) were added to the reaction with an additional 5 ml of THF. The slurry was heated to 45°C to form a golden-yellow solution over the course of one h. The solution was cooled to room temperature and the solvent was removed under reduced pressure to yield a powdery white residue. The residue was dissolved in benzene and filtered through celite. Crystals appropriate for X-ray crystallography were grown by a layering diffusion of the benzene solution with hexanes and the complex was isolated as pale-yellow crystals.

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 1. One $SiMe_3$ group is disordered over two positions, Si2/C9/C10/C11 and Si2′/C9′/C10′/C11′, with site occupancies converging to 0.727 (7) and 0.273 (7), respectively.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161338 [doi:10.1107/S2414314616013389]

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Crystal data

$[K(C_{11}H_{22}N_3Si_2)(C_{12}H_{24}O_6)]$
M_r = 555.91
Monoclinic, *I*2/*a*
a = 20.3301 (10) Å
b = 17.4558 (6) Å
c = 20.5691 (11) Å
β = 119.399 (7)°
V = 6359.5 (6) Å^3
Z = 8
F(000) = 2400
D_x = 1.161 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 8021 reflections
θ = 1.6–27.5°
μ = 0.28 mm^{-1}
T = 173 K
Block, pale yellow
0.56 × 0.36 × 0.29 mm

Data collection

Rigaku XtaLAB mini diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.797, T_{max} = 1.000
27816 measured reflections
5770 independent reflections
4565 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.3°, θ_{min} = 2.3°
h = −24→24
k = −20→20
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.140
S = 1.05
5770 reflections
350 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 12.3202P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
K1	0.37601 (3)	0.33574 (4)	0.41659 (3)	0.03787 (17)	

Si1	0.53829 (5)	0.24442 (5)	0.36944 (4)	0.0440 (2)	
Si2	0.22824 (17)	0.56321 (18)	0.16953 (19)	0.0538 (7)	0.727 (7)
O5	0.24060 (11)	0.25955 (12)	0.33205 (11)	0.0432 (5)	
O6	0.33257 (12)	0.20087 (12)	0.47690 (11)	0.0493 (5)	
O2	0.48287 (12)	0.40416 (13)	0.55274 (12)	0.0542 (6)	
O4	0.22649 (11)	0.42225 (13)	0.34204 (13)	0.0548 (6)	
N1	0.47290 (12)	0.31219 (14)	0.35653 (12)	0.0382 (5)	
N2	0.38208 (12)	0.40545 (13)	0.29390 (12)	0.0348 (5)	
O1	0.48377 (13)	0.24315 (13)	0.53913 (13)	0.0561 (6)	
O3	0.34216 (13)	0.47888 (14)	0.47999 (14)	0.0636 (7)	
C1	0.44893 (14)	0.37042 (15)	0.30789 (14)	0.0340 (6)	
N3	0.28526 (15)	0.49256 (17)	0.2328 (2)	0.0572 (8)	
C5	0.35350 (15)	0.46304 (16)	0.24448 (15)	0.0392 (7)	
C2	0.48654 (17)	0.39912 (17)	0.27002 (17)	0.0446 (7)	
H2	0.5319	0.3773	0.2788	0.054*	
C19	0.18511 (17)	0.30613 (19)	0.27335 (17)	0.0486 (8)	
H19A	0.1408	0.2753	0.2427	0.058*	
H19B	0.2053	0.3241	0.2420	0.058*	
C18	0.16240 (17)	0.3741 (2)	0.30315 (19)	0.0532 (8)	
H18A	0.1220	0.4020	0.2623	0.064*	
H18B	0.1445	0.3569	0.3366	0.064*	
C4	0.3887 (2)	0.49176 (18)	0.20632 (18)	0.0512 (8)	
H4	0.3676	0.5317	0.1723	0.061*	
C3	0.4565 (2)	0.45838 (19)	0.22108 (19)	0.0540 (8)	
H3A	0.4819	0.4768	0.1971	0.065*	
C20	0.21142 (17)	0.2138 (2)	0.36939 (17)	0.0505 (8)	
H20A	0.1670	0.1862	0.3333	0.061*	
H20B	0.1972	0.2461	0.3989	0.061*	
C6	0.53333 (19)	0.2108 (2)	0.28003 (18)	0.0547 (8)	
H6A	0.4822	0.2145	0.2400	0.082*	
H6B	0.5498	0.1585	0.2855	0.082*	
H6C	0.5654	0.2423	0.2691	0.082*	
C21	0.2714 (2)	0.15905 (19)	0.41856 (18)	0.0561 (9)	
H21A	0.2511	0.1228	0.4399	0.067*	
H21B	0.2892	0.1307	0.3897	0.067*	
C23	0.45575 (19)	0.2034 (2)	0.58003 (18)	0.0581 (9)	
H23A	0.4378	0.2393	0.6038	0.070*	
H23B	0.4953	0.1723	0.6184	0.070*	
C22	0.39254 (19)	0.1539 (2)	0.5265 (2)	0.0587 (9)	
H22A	0.4094	0.1220	0.4989	0.070*	
H22B	0.3759	0.1207	0.5534	0.070*	
C13	0.5376 (2)	0.3591 (2)	0.6117 (2)	0.0724 (11)	
H13A	0.5204	0.3472	0.6469	0.087*	
H13B	0.5847	0.3873	0.6378	0.087*	
C7	0.63966 (19)	0.2734 (3)	0.4331 (2)	0.0780 (12)	
H7A	0.6517	0.3169	0.4124	0.117*	
H7B	0.6724	0.2316	0.4380	0.117*	
H7C	0.6464	0.2864	0.4813	0.117*	

C8	0.5198 (2)	0.1573 (2)	0.4111 (2)	0.0656 (10)	
H8A	0.5267	0.1696	0.4596	0.098*	
H8B	0.5544	0.1174	0.4158	0.098*	
H8C	0.4689	0.1402	0.3795	0.098*	
C12	0.5499 (2)	0.2879 (3)	0.5811 (2)	0.0786 (12)	
H12A	0.5712	0.3007	0.5493	0.094*	
H12B	0.5868	0.2570	0.6220	0.094*	
C14	0.4656 (2)	0.4693 (3)	0.5824 (3)	0.0738 (13)	
C15	0.4119 (2)	0.5184 (2)	0.5220 (3)	0.0717 (12)	
H15A	0.4037	0.5650	0.5426	0.086*	
H15B	0.4323	0.5323	0.4897	0.086*	
C16	0.2860 (2)	0.5253 (2)	0.4243 (3)	0.0801 (13)	
H16A	0.3025	0.5425	0.3898	0.096*	
H16B	0.2773	0.5701	0.4470	0.096*	
C17	0.2145 (2)	0.4802 (2)	0.3833 (3)	0.0798 (13)	
H17A	0.2013	0.4573	0.4184	0.096*	
H17B	0.1735	0.5134	0.3500	0.096*	
C10	0.2656 (5)	0.6614 (5)	0.2001 (7)	0.0866 (16)	0.727 (7)
H10A	0.2619	0.6747	0.2435	0.130*	0.727 (7)
H10B	0.2366	0.6971	0.1607	0.130*	0.727 (7)
H10C	0.3175	0.6633	0.2119	0.130*	0.727 (7)
C9	0.2135 (4)	0.5557 (4)	0.0737 (3)	0.0866 (16)	0.727 (7)
H9A	0.2615	0.5541	0.0754	0.130*	0.727 (7)
H9B	0.1854	0.5993	0.0451	0.130*	0.727 (7)
H9C	0.1859	0.5097	0.0508	0.130*	0.727 (7)
C11	0.1346 (3)	0.5513 (4)	0.1639 (4)	0.0866 (16)	0.727 (7)
H11A	0.1177	0.4994	0.1506	0.130*	0.727 (7)
H11B	0.0987	0.5854	0.1267	0.130*	0.727 (7)
H11C	0.1389	0.5631	0.2114	0.130*	0.727 (7)
H14A	0.515 (2)	0.493 (2)	0.615 (2)	0.089 (13)*	
H14B	0.448 (3)	0.453 (3)	0.613 (3)	0.108 (17)*	
H3	0.274 (2)	0.474 (2)	0.260 (2)	0.054 (12)*	
C9′	0.1665 (11)	0.5500 (10)	0.0965 (10)	0.113 (6)	0.273 (7)
H9′A	0.1937	0.5148	0.0826	0.169*	0.273 (7)
H9′B	0.1562	0.5960	0.0676	0.169*	0.273 (7)
H9′C	0.1198	0.5270	0.0873	0.169*	0.273 (7)
Si2′	0.2232 (6)	0.5729 (6)	0.1957 (6)	0.068 (2)	0.273 (7)
C10′	0.2770 (15)	0.6586 (13)	0.216 (2)	0.113 (6)	0.273 (7)
H10D	0.2998	0.6695	0.2683	0.169*	0.273 (7)
H10E	0.2445	0.7002	0.1880	0.169*	0.273 (7)
H10F	0.3157	0.6524	0.2025	0.169*	0.273 (7)
C11′	0.1599 (10)	0.5936 (10)	0.2300 (13)	0.113 (6)	0.273 (7)
H11D	0.1404	0.5466	0.2379	0.169*	0.273 (7)
H11E	0.1190	0.6245	0.1942	0.169*	0.273 (7)
H11F	0.1863	0.6210	0.2763	0.169*	0.273 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0336 (3)	0.0448 (4)	0.0361 (3)	−0.0076 (3)	0.0177 (3)	−0.0087 (3)
Si1	0.0349 (4)	0.0534 (5)	0.0350 (4)	0.0100 (4)	0.0104 (4)	−0.0071 (4)
Si2	0.0354 (9)	0.0394 (9)	0.0706 (18)	0.0066 (6)	0.0136 (11)	0.0092 (11)
O5	0.0381 (11)	0.0529 (12)	0.0373 (11)	−0.0103 (9)	0.0175 (9)	−0.0108 (9)
O6	0.0479 (12)	0.0523 (13)	0.0407 (11)	−0.0096 (10)	0.0164 (10)	−0.0067 (10)
O2	0.0497 (13)	0.0575 (14)	0.0470 (13)	−0.0146 (11)	0.0172 (11)	−0.0130 (11)
O4	0.0396 (12)	0.0573 (14)	0.0726 (15)	−0.0078 (10)	0.0316 (11)	−0.0234 (12)
N1	0.0334 (12)	0.0470 (14)	0.0307 (12)	0.0035 (10)	0.0129 (10)	−0.0056 (10)
N2	0.0290 (11)	0.0391 (13)	0.0304 (12)	−0.0017 (10)	0.0102 (10)	−0.0054 (10)
O1	0.0516 (13)	0.0570 (14)	0.0569 (14)	−0.0085 (11)	0.0244 (12)	−0.0044 (11)
O3	0.0507 (14)	0.0711 (16)	0.0800 (17)	−0.0282 (12)	0.0406 (14)	−0.0419 (14)
C1	0.0297 (13)	0.0370 (15)	0.0288 (13)	−0.0040 (12)	0.0093 (11)	−0.0141 (12)
N3	0.0378 (15)	0.0503 (18)	0.076 (2)	0.0042 (12)	0.0225 (15)	0.0138 (16)
C5	0.0361 (15)	0.0354 (15)	0.0344 (15)	−0.0024 (12)	0.0083 (13)	−0.0071 (12)
C2	0.0456 (17)	0.0433 (17)	0.0542 (18)	−0.0031 (14)	0.0316 (15)	−0.0109 (14)
C19	0.0391 (16)	0.059 (2)	0.0389 (17)	−0.0070 (14)	0.0123 (14)	−0.0097 (14)
C18	0.0329 (16)	0.063 (2)	0.061 (2)	−0.0083 (15)	0.0212 (15)	−0.0112 (17)
C4	0.069 (2)	0.0396 (17)	0.0448 (18)	−0.0027 (16)	0.0275 (17)	0.0006 (14)
C3	0.074 (2)	0.0450 (19)	0.060 (2)	−0.0067 (17)	0.0469 (19)	−0.0076 (16)
C20	0.0428 (17)	0.066 (2)	0.0389 (17)	−0.0227 (16)	0.0171 (14)	−0.0134 (15)
C6	0.055 (2)	0.059 (2)	0.0526 (19)	0.0118 (16)	0.0284 (17)	−0.0096 (16)
C21	0.065 (2)	0.054 (2)	0.0481 (19)	−0.0271 (17)	0.0267 (17)	−0.0129 (16)
C23	0.0481 (19)	0.079 (2)	0.0417 (18)	0.0054 (17)	0.0177 (16)	0.0048 (17)
C22	0.057 (2)	0.057 (2)	0.063 (2)	−0.0006 (17)	0.0295 (18)	0.0078 (17)
C13	0.063 (2)	0.080 (3)	0.054 (2)	−0.025 (2)	0.0122 (19)	−0.008 (2)
C7	0.0398 (19)	0.100 (3)	0.072 (3)	0.014 (2)	0.0098 (18)	−0.026 (2)
C8	0.079 (3)	0.063 (2)	0.050 (2)	0.0204 (19)	0.0273 (19)	0.0039 (17)
C12	0.046 (2)	0.084 (3)	0.082 (3)	−0.007 (2)	0.013 (2)	−0.004 (2)
C14	0.059 (2)	0.098 (3)	0.075 (3)	−0.043 (2)	0.041 (2)	−0.057 (3)
C15	0.048 (2)	0.065 (2)	0.106 (3)	−0.0242 (19)	0.040 (2)	−0.047 (2)
C16	0.056 (2)	0.064 (3)	0.116 (4)	−0.006 (2)	0.040 (2)	−0.045 (2)
C17	0.048 (2)	0.079 (3)	0.110 (3)	−0.0038 (19)	0.037 (2)	−0.048 (3)
C10	0.064 (2)	0.067 (2)	0.106 (3)	0.0136 (18)	0.024 (2)	0.015 (2)
C9	0.064 (2)	0.067 (2)	0.106 (3)	0.0136 (18)	0.024 (2)	0.015 (2)
C11	0.064 (2)	0.067 (2)	0.106 (3)	0.0136 (18)	0.024 (2)	0.015 (2)
C9′	0.082 (8)	0.059 (6)	0.154 (14)	0.033 (5)	0.024 (8)	−0.001 (7)
Si2′	0.048 (3)	0.067 (4)	0.076 (5)	0.011 (2)	0.021 (3)	0.017 (4)
C10′	0.082 (8)	0.059 (6)	0.154 (14)	0.033 (5)	0.024 (8)	−0.001 (7)
C11′	0.082 (8)	0.059 (6)	0.154 (14)	0.033 (5)	0.024 (8)	−0.001 (7)

Geometric parameters (Å, º)

K1—O1	2.889 (2)	C6—H6C	0.9600
K1—O2	2.831 (2)	C21—H21A	0.9700
K1—O3	3.049 (2)	C21—H21B	0.9700

K1—O4	3.049 (2)	C23—H23A	0.9700
K1—O5	2.770 (2)	C23—H23B	0.9700
K1—O6	2.990 (2)	C23—C22	1.491 (5)
K1—N1	2.822 (2)	C22—H22A	0.9700
K1—N2	2.858 (2)	C22—H22B	0.9700
Si1—N1	1.700 (2)	C13—H13A	0.9700
Si1—C6	1.885 (3)	C13—H13B	0.9700
Si1—C7	1.889 (4)	C13—C12	1.469 (6)
Si1—C8	1.873 (4)	C7—H7A	0.9600
Si2—N3	1.754 (4)	C7—H7B	0.9600
Si2—C10	1.856 (9)	C7—H7C	0.9600
Si2—C9	1.847 (7)	C8—H8A	0.9600
Si2—C11	1.862 (7)	C8—H8B	0.9600
O5—C19	1.433 (4)	C8—H8C	0.9600
O5—C20	1.423 (4)	C12—H12A	0.9700
O6—C21	1.433 (4)	C12—H12B	0.9700
O6—C22	1.407 (4)	C14—C15	1.462 (6)
O2—C13	1.416 (4)	C14—H14A	0.98 (5)
O2—C14	1.414 (5)	C14—H14B	0.91 (5)
O4—C18	1.422 (4)	C15—H15A	0.9700
O4—C17	1.417 (4)	C15—H15B	0.9700
N1—C1	1.339 (4)	C16—H16A	0.9700
N2—C1	1.385 (3)	C16—H16B	0.9700
N2—C5	1.342 (4)	C16—C17	1.497 (5)
O1—C23	1.409 (4)	C17—H17A	0.9700
O1—C12	1.423 (4)	C17—H17B	0.9700
O3—C15	1.425 (4)	C10—H10A	0.9600
O3—C16	1.410 (5)	C10—H10B	0.9600
C1—C2	1.424 (4)	C10—H10C	0.9600
N3—C5	1.386 (4)	C9—H9A	0.9600
N3—H3	0.76 (3)	C9—H9B	0.9600
N3—Si2′	1.788 (11)	C9—H9C	0.9600
C5—C4	1.389 (4)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C2—C3	1.361 (5)	C11—H11C	0.9600
C19—H19A	0.9700	C9′—H9′A	0.9600
C19—H19B	0.9700	C9′—H9′B	0.9600
C19—C18	1.507 (4)	C9′—H9′C	0.9600
C18—H18A	0.9700	C9′—Si2′	1.828 (17)
C18—H18B	0.9700	Si2′—C10′	1.78 (2)
C4—H4	0.9300	Si2′—C11′	1.782 (17)
C4—C3	1.387 (5)	C10′—H10D	0.9600
C3—H3A	0.9300	C10′—H10E	0.9600
C20—H20A	0.9700	C10′—H10F	0.9600
C20—H20B	0.9700	C11′—H11D	0.9600
C20—C21	1.488 (5)	C11′—H11E	0.9600
C6—H6A	0.9600	C11′—H11F	0.9600
C6—H6B	0.9600		

O5—K1—O6	58.49 (6)	H6B—C6—H6C	109.5
O5—K1—O2	150.12 (6)	O6—C21—C20	109.1 (3)
O5—K1—O4	58.92 (6)	O6—C21—H21A	109.9
O5—K1—N1	110.33 (6)	O6—C21—H21B	109.9
O5—K1—N2	96.35 (6)	C20—C21—H21A	109.9
O5—K1—O1	111.93 (6)	C20—C21—H21B	109.9
O5—K1—O3	107.36 (6)	H21A—C21—H21B	108.3
O6—K1—O4	100.02 (6)	O1—C23—H23A	110.2
O6—K1—O3	107.05 (6)	O1—C23—H23B	110.2
O2—K1—O6	99.28 (6)	O1—C23—C22	107.4 (3)
O2—K1—O4	112.34 (7)	H23A—C23—H23B	108.5
O2—K1—N2	110.43 (7)	C22—C23—H23A	110.2
O2—K1—O1	59.19 (7)	C22—C23—H23B	110.2
O2—K1—O3	57.04 (7)	O6—C22—C23	108.8 (3)
O4—K1—O3	55.30 (6)	O6—C22—H22A	109.9
N1—K1—O6	118.60 (7)	O6—C22—H22B	109.9
N1—K1—O2	97.64 (7)	C23—C22—H22A	109.9
N1—K1—O4	126.14 (7)	C23—C22—H22B	109.9
N1—K1—N2	48.21 (6)	H22A—C22—H22B	108.3
N1—K1—O1	84.67 (7)	O2—C13—H13A	109.8
N1—K1—O3	131.24 (7)	O2—C13—H13B	109.8
N2—K1—O6	148.34 (6)	O2—C13—C12	109.3 (3)
N2—K1—O4	79.02 (6)	H13A—C13—H13B	108.3
N2—K1—O1	131.73 (7)	C12—C13—H13A	109.8
N2—K1—O3	98.36 (7)	C12—C13—H13B	109.8
O1—K1—O6	56.58 (6)	Si1—C7—H7A	109.5
O1—K1—O4	149.08 (7)	Si1—C7—H7B	109.5
O1—K1—O3	108.60 (7)	Si1—C7—H7C	109.5
N1—Si1—C6	113.64 (13)	H7A—C7—H7B	109.5
N1—Si1—C7	115.16 (15)	H7A—C7—H7C	109.5
N1—Si1—C8	108.62 (15)	H7B—C7—H7C	109.5
C6—Si1—C7	106.12 (17)	Si1—C8—H8A	109.5
C8—Si1—C6	105.50 (16)	Si1—C8—H8B	109.5
C8—Si1—C7	107.20 (19)	Si1—C8—H8C	109.5
N3—Si2—C10	112.8 (3)	H8A—C8—H8B	109.5
N3—Si2—C9	116.3 (3)	H8A—C8—H8C	109.5
N3—Si2—C11	104.5 (3)	H8B—C8—H8C	109.5
C10—Si2—C11	111.1 (5)	O1—C12—C13	114.5 (3)
C9—Si2—C10	104.6 (5)	O1—C12—H12A	108.6
C9—Si2—C11	107.5 (3)	O1—C12—H12B	108.6
C19—O5—K1	112.66 (16)	C13—C12—H12A	108.6
C20—O5—K1	118.66 (16)	C13—C12—H12B	108.6
C20—O5—C19	114.1 (2)	H12A—C12—H12B	107.6
C21—O6—K1	111.44 (17)	O2—C14—C15	110.1 (3)
C22—O6—K1	115.64 (18)	O2—C14—H14A	105 (2)
C22—O6—C21	113.4 (3)	O2—C14—H14B	108 (3)
C13—O2—K1	120.8 (2)	C15—C14—H14A	115 (2)

C14—O2—K1	122.3 (2)	C15—C14—H14B	113 (3)
C14—O2—C13	109.6 (3)	H14A—C14—H14B	105 (4)
C18—O4—K1	113.71 (18)	O3—C15—C14	109.8 (3)
C17—O4—K1	118.3 (2)	O3—C15—H15A	109.7
C17—O4—C18	112.3 (2)	O3—C15—H15B	109.7
Si1—N1—K1	133.89 (12)	C14—C15—H15A	109.7
C1—N1—K1	98.06 (16)	C14—C15—H15B	109.7
C1—N1—Si1	127.94 (19)	H15A—C15—H15B	108.2
C1—N2—K1	95.27 (16)	O3—C16—H16A	109.8
C5—N2—K1	142.86 (18)	O3—C16—H16B	109.8
C5—N2—C1	120.2 (2)	O3—C16—C17	109.3 (4)
C23—O1—K1	116.16 (18)	H16A—C16—H16B	108.3
C23—O1—C12	116.6 (3)	C17—C16—H16A	109.8
C12—O1—K1	108.0 (2)	C17—C16—H16B	109.8
C15—O3—K1	106.81 (19)	O4—C17—C16	108.0 (3)
C16—O3—K1	112.76 (19)	O4—C17—H17A	110.1
C16—O3—C15	112.5 (3)	O4—C17—H17B	110.1
N1—C1—K1	58.17 (14)	C16—C17—H17A	110.1
N1—C1—N2	116.8 (2)	C16—C17—H17B	110.1
N1—C1—C2	125.4 (3)	H17A—C17—H17B	108.4
N2—C1—K1	59.93 (14)	Si2—C10—H10A	109.5
N2—C1—C2	117.8 (3)	Si2—C10—H10B	109.5
C2—C1—K1	168.24 (18)	Si2—C10—H10C	109.5
Si2—N3—H3	121 (3)	H10A—C10—H10B	109.5
C5—N3—Si2	129.1 (3)	H10A—C10—H10C	109.5
C5—N3—H3	110 (3)	H10B—C10—H10C	109.5
C5—N3—Si2′	143.3 (4)	Si2—C9—H9A	109.5
Si2′—N3—H3	104 (3)	Si2—C9—H9B	109.5
N2—C5—N3	115.7 (3)	Si2—C9—H9C	109.5
N2—C5—C4	123.1 (3)	H9A—C9—H9B	109.5
N3—C5—C4	121.1 (3)	H9A—C9—H9C	109.5
C1—C2—H2	119.8	H9B—C9—H9C	109.5
C3—C2—C1	120.4 (3)	Si2—C11—H11A	109.5
C3—C2—H2	119.8	Si2—C11—H11B	109.5
O5—C19—H19A	109.2	Si2—C11—H11C	109.5
O5—C19—H19B	109.2	H11A—C11—H11B	109.5
O5—C19—C18	112.0 (2)	H11A—C11—H11C	109.5
H19A—C19—H19B	107.9	H11B—C11—H11C	109.5
C18—C19—H19A	109.2	H9′A—C9′—H9′B	109.5
C18—C19—H19B	109.2	H9′A—C9′—H9′C	109.5
O4—C18—C19	108.4 (2)	H9′B—C9′—H9′C	109.5
O4—C18—H18A	110.0	Si2′—C9′—H9′A	109.5
O4—C18—H18B	110.0	Si2′—C9′—H9′B	109.5
C19—C18—H18A	110.0	Si2′—C9′—H9′C	109.5
C19—C18—H18B	110.0	N3—Si2′—C9′	103.4 (7)
H18A—C18—H18B	108.4	C10′—Si2′—N3	109.7 (10)
C5—C4—H4	121.4	C10′—Si2′—C9′	115.2 (16)
C3—C4—C5	117.2 (3)	C10′—Si2′—C11′	102.5 (16)

C3—C4—H4	121.4	C11′—Si2′—N3	119.0 (8)
C2—C3—C4	121.2 (3)	C11′—Si2′—C9′	107.7 (11)
C2—C3—H3A	119.4	Si2′—C10′—H10D	109.5
C4—C3—H3A	119.4	Si2′—C10′—H10E	109.5
O5—C20—H20A	110.1	Si2′—C10′—H10F	109.5
O5—C20—H20B	110.1	H10D—C10′—H10E	109.5
O5—C20—C21	107.9 (3)	H10D—C10′—H10F	109.5
H20A—C20—H20B	108.4	H10E—C10′—H10F	109.5
C21—C20—H20A	110.1	Si2′—C11′—H11D	109.5
C21—C20—H20B	110.1	Si2′—C11′—H11E	109.5
Si1—C6—H6A	109.5	Si2′—C11′—H11F	109.5
Si1—C6—H6B	109.5	H11D—C11′—H11E	109.5
Si1—C6—H6C	109.5	H11D—C11′—H11F	109.5
H6A—C6—H6B	109.5	H11E—C11′—H11F	109.5
H6A—C6—H6C	109.5		

K1—O5—C19—C18	−64.4 (3)	C1—N2—C5—C4	−1.4 (4)
K1—O5—C20—C21	−53.6 (3)	C1—C2—C3—C4	−0.6 (5)
K1—O6—C21—C20	−47.2 (3)	N3—C5—C4—C3	−179.5 (3)
K1—O6—C22—C23	44.1 (3)	C5—N2—C1—K1	168.7 (3)
K1—O2—C13—C12	23.9 (4)	C5—N2—C1—N1	−178.4 (2)
K1—O2—C14—C15	−32.7 (4)	C5—N2—C1—C2	1.9 (3)
K1—O4—C18—C19	−31.6 (3)	C5—N3—Si2′—C9′	85.7 (12)
K1—O4—C17—C16	42.1 (5)	C5—N3—Si2′—C10′	−37.6 (19)
K1—N1—C1—N2	−13.2 (2)	C5—N3—Si2′—C11′	−155.1 (9)
K1—N1—C1—C2	166.5 (2)	C5—C4—C3—C2	1.1 (5)
K1—N2—C1—N1	12.9 (2)	C19—O5—C20—C21	169.9 (2)
K1—N2—C1—C2	−166.8 (2)	C18—O4—C17—C16	177.7 (4)
K1—N2—C5—N3	−20.9 (4)	C20—O5—C19—C18	74.7 (3)
K1—N2—C5—C4	159.7 (2)	C6—Si1—N1—K1	136.60 (17)
K1—O1—C23—C22	57.4 (3)	C6—Si1—N1—C1	−38.6 (3)
K1—O1—C12—C13	59.2 (4)	C21—O6—C22—C23	174.5 (3)
K1—O3—C15—C14	−60.4 (3)	C23—O1—C12—C13	−73.7 (4)
K1—O3—C16—C17	59.7 (4)	C22—O6—C21—C20	−179.8 (3)
K1—C1—C2—C3	−76.7 (10)	C13—O2—C14—C15	177.2 (3)
Si1—N1—C1—K1	176.5 (3)	C7—Si1—N1—K1	−100.7 (2)
Si1—N1—C1—N2	163.35 (19)	C7—Si1—N1—C1	84.1 (3)
Si1—N1—C1—C2	−16.9 (4)	C8—Si1—N1—K1	19.5 (2)
Si2—N3—C5—N2	−175.9 (3)	C8—Si1—N1—C1	−155.7 (2)
Si2—N3—C5—C4	3.5 (5)	C12—O1—C23—C22	−173.6 (3)
O5—C19—C18—O4	64.3 (3)	C14—O2—C13—C12	174.6 (3)
O5—C20—C21—O6	66.8 (3)	C15—O3—C16—C17	−179.4 (3)
O2—C13—C12—O1	−57.3 (5)	C16—O3—C15—C14	175.4 (3)
O2—C14—C15—O3	64.3 (4)	C17—O4—C18—C19	−169.3 (3)
N1—C1—C2—C3	179.4 (3)	C10—Si2—N3—C5	−76.5 (6)
N2—C1—C2—C3	−0.9 (4)	C9—Si2—N3—C5	44.4 (5)
N2—C5—C4—C3	−0.1 (4)	C11—Si2—N3—C5	162.7 (4)
O1—C23—C22—O6	−66.8 (3)	Si2′—N3—C5—N2	163.3 (7)

O3—C16—C17—O4	−68.1 (5)	Si2′—N3—C5—C4	−17.3 (8)
C1—N2—C5—N3	178.0 (2)		