The Design and Synthesis of a New Class of Nonmacrocyclic Alkali Metal Host Compounds

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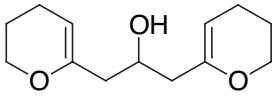
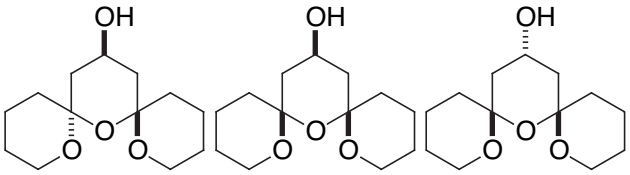
Supporting Information



1,3-*Bis*[(3,4-dihydro-2*H*-pyran-2-yl)]propan-2-ol (9)
To a 1-necked 500 mL round-bottom flask equipped with a magnetic stirring bar and a reflux condenser fitted with a septum was introduced 3,4-dihydro-2*H*-pyran (23.97 g, 0.285 mol) and THF (40 mL). After cooling to –78° C, ^{n}butyllithium (110 mL, 2.58 M, 0.284 mol) was introduced and the result stirred for 10 min then warmed to 60-65° C where it was maintained for 2 h. The resultant blood red solution was then cooled to –78° C then cannulated into a cold (–78° C), vigorously stirred slurry of CuCN (12.20 g, 0.136 mol) and THF (120 mL). After 10 min, the mixture was warmed to 0° C for 10 min, then cooled once again to –78° C to afford a viscous, tan solution of the 3,4-dihydro-2*H*-pyran cuprate.

To the cold solution of the cuprate was added epibromohydrin (8.0 g, 58.0 mmol). At the completion of the addition, the reaction mixture was allowed to warm to room temperature where it was maintained for 16 h. At this time, the mixture was poured into 10% NH_4OH/saturated aqueous NH_4Cl (800 mL) and the layers separated. The aqueous layer was extracted three times with Et_2O and the combined organic layers dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The resulting residue was purified by flash chromatography (SiO_2, 50% Et_2O/hexanes) to afford adduct **9** as a colorless oil (8.15 g, 65%).

R*f* 0.54 (50% Et_2O/hexanes).
IR (NaCl, thin film) 3478, 2883, 1671, 1564, 1535, 1491, 1444, 1400, 1236, 1171, 1039, 973, 936, 900, 871, 773 cm^{-1}.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.61-2.38 (m, 12H), 2.65 (d, J=3.0 Hz, 1H), 3.86-4.06 (m, 5H), 4.37-4.59 (m, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.14, 22.24, 41.64, 66.12, 67.90, 97.91, 151.68 ppm.
Anal. Calcd for $C_{13}H_{20}O_3$: C, 69.61; H, 8.99. Found: C, 69.50; H, 8.95.



10t syn-**10c** anti-**10c**

Spiroketalization of *bis*-dihydropyran 9 to give a mixture of 10t:*syn*-10c:*anti*-10c

To a stirred solution of *bis*-dihydropyran **9** (11.26 g, 50.2 mmol) in THF:H_2O (5:1, 325 mL) at room temperature was added PPTS (2.04 g, 8.10 mmol). After 19 h, the reaction mixture was treated with Et_3N (3 mL) then dried with $MgSO_4$ (50 g). The resultant solution was filtered, concentrated under reduced pressure, then coevaporated with benzene (2 x 200 mL) and placed under vacuum (0.1 torr) for 1.5 h. The result was a yellow solid that could be recrystallized from acetone to afford the pure *bis*-hemiketal as a white solid (mp 64-65° C). The crude hydrate was dissolved in THF (326 mL) and treated with PPTS (3.70 g, 14.7 mmol) and stirred for two h at which time 4Å molecular sieves (87 g) were introduced and the result stirred an additional 3 h. The solution was then filtered through Celite, the filter cake thoroughly washed with Et_2O (900 mL), and the combined filtrates treated with Et_3N and dried over $MgSO_4$. The result was filtered and concentrated under reduced pressure to afford a residue which is purified by chromatrography (linear gradient of 20-40% EtOAc/hexanes) to give a mixture of inseparable **10t** with *syn*-**10c** (7.69 g, 63%) and *anti*-**10c** (2.49 g, 20%) as colorless oils.

10t

R*f* 0.55 (50% EtOAc/hexanes).

IR (NaCl, thin film) 3439, 2940, 2868, 1440, 1385, 1251, 1219, 1162, 1100, 1082, 1064, 1044, 1000, 987, 977, 939, 785 cm^{-1}.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.44-1.66 (m, 8H), 1.68-1.79 (m, 3H), 1.88-2.04 (m, 2H), 2.22 (dd, J=6.0, 14.0 Hz, 1H), 2.31 (dd, J=8.2, 13.7 Hz, 1H), 2.58 (d, J=10.5 Hz, 1H), 3.49-3.55 (m, 1H), 3.58-3.63 (m, 1H), 3.79 (ddd, J=3.33, 11.4, 11.4 Hz, 1H), 3.88 (ddd, J=3.2, 11.5, 11.7 Hz, 1H), 4.01-4.09 (m, 1H) ppm.

^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.40, 18,56, 25.05, 35.98, 36.56, 41.36, 43.11, 60.84, 95.99, 97.37 ppm.

Anal. Calcd for $C_{13}H_{24}O_4$: C, 64.44; H, 9.15. Found: C, 64.28; H, 9.20.

Syn-**10c**

R*f* 0.55 (50% EtOAc/hexanes).

IR (NaCl, thin film) 3409, 2929, 2860, 2773, 2709, 1441, 1373, 1345, 1273, 1254, 1215, 1196, 1164, 1145, 1100, 1071, 1045, 980, 936, 927, 882, 816, 773, 745, 645, 618, 571, 518 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.49 (ddd, J=3.3, 12.2, 12.2 Hz, 2H), 1.49-1.53 (m, 4H), 1.56 (dd, J=4.0, 13.5 Hz, 2H), 1.53-1.62 (m, 2H), 1.58-1.64 (m, 2H), 1.83-1.92 (m, 2H), 2.16 (dd, J=3.7, 13.8 Hz, 2H), 3.65 (dddd, J=1.5, 3.6, 3.8, 10.9 Hz, 2H), 3.89 (d, J=11.5 Hz, 1H), 3.99 (dtt, J=3.6, 3.6, 11.0 Hz, 1H), 3.99-4.15 (m, 2H) ppm.

^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.91, 25.16, 37.60, 40.33, 62.17, 64.01, 97.19 ppm.

Anal. Calcd for $C_{13}H_{24}O_4$: C, 64.44; H, 9.15. Found: C,64.31; H, 9.09.

Anti-**10c**

R*f* 0.37 (50% Et_2O/hexanes).

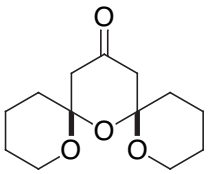
IR (NaCl, thin film) 3351, 2934, 2883, 2727, 2654, 1700, 1642, 1442, 1379, 1218, 1154, 1118, 1078, 1036, 1018, 973, 933, 871, 845, 804, 764 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.30 (dd, J=10.9, 12.9 Hz, 2H), 1.52 (ddd, J=4.3, 13.0, 13.0 Hz, 2H), 1.54-1.62 (m, 6H), 1.69 (ddd, J=3.8, 3.8, 13.1 Hz, 2H), 1.82-1.93 (m, 3H), 2.21 (dd, J=4.5, 12.8 Hz, 2H), 3.64 (dddd, J=1.5, 3.7, 3.7, 11.1 Hz, 2H), 3.97-4.06 (m, 2H), 4.32 (tt, J=4.5, 10.9 Hz, 1H) ppm.

^{13}C NMR ($CDCl_3$, 75 MHz) δ 19.08, 25.34, 45.00, 60.85, 62.31, 98.41 ppm.

Anal. Calcd for $C_{13}H_{24}O_4$: C, 64.44; H, 9.15. Found: C, 64.37; H, 9.19.

Acid catalyzed equilibration of the mixture of 10t and *syn*-10c.
To a stirred solution of a mixture of alcohols **10t** and *syn*-**10c** (7.68 g of 89:11 mixture, 31.7 mmol) in THF (500 mL) was added PPTS (1.66 g, 6.62 mmol) and the result was stored at room temperature for 20 h. At this time, Et_3N (9 mL) was added, the resultant solution concentrated under reduced pressure, and the residue purified by column chromatography (linear gradient of 20-40% EtOAc/hexanes) to afford a mixture of inseparable **10t** with *syn*-**10c** (5.20 g, 89:11 mixture, 68%) and *anti*-**10c** (2.48 g, 32%) as colorless oils.

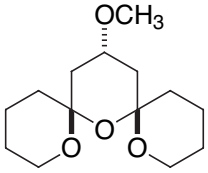


Oxidation of *anti*-10c to ketone 11.
To a stirred solution of alcohol *anti*-**10c** (2.48 g, 10.20 mmol) in CH_2Cl_2 (100 mL) was added pyridine (3.60 g, 46 mmol) and Dess-Martin periodinane (4.81 g, 11.3 mmol). After 6 h, the mixture was poured into saturated aqueous $NaHCO_3$/saturated aqueous $Na_2S_2O_3$ (1:1, 200 mL), the resultant layers separated, and the aqueous layer extracted three times with Et_2O. The combined organic layers were dried over $MgSO_4$, filtered, and concentrated under reduced pressure to afford a residue which was purified by chromatography (50% Et_2O/hexanes) to give the ketone **11** as a white solid (1.98 g, 81%).

R*f* 0.61 (50% Et_2O/hexanes).
Mp 60-63° C.
IR (NaCl, thin film) 2923, 2873, 1726, 1471, 1436, 1373, 1291, 1273, 1229, 1205, 1100, 1073, 1036, 973, 933, 860 cm^{-1}.
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.49 (ddd, J=4.2, 13.2, 13.5 Hz, 2H), 1.54-1.67 (m, 6H), 1.71 (ddd, J=3.0, 3.0, 13.0 Hz, 2H), 1.95 (m, 2H), 2.40 (d, J=17.5 Hz, 2H), 2.87 (d, J=17.0 Hz, 2H), 3.71 (m, 2H), 4.10 (m, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.72, 24.81, 37.02, 49.90, 61.91, 97.68, 206.38 ppm.
Anal. Calcd for $C_{13}H_{20}O_4$: C, 64.98; H, 8.39. Found: C, 64.94; H, 8.34.

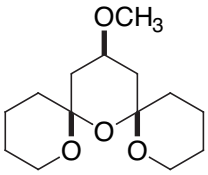
Reduction of ketone 11 using $LiAlH_4$.
A solution of the ketone (0.25 g, 1.04 mmol) in Et_2O was cooled to 0° C then treated with $LiAlH_4$ (0.070 g, 1.85 mmol). The resultant slurry was stirred 15 min, then sequentially treated with H_2O (0.25 mL), NaOH (4.4 M, 0.25 mL), and H_2O (0.75 mL). A white suspension formed upon stirring which was dried over $MgSO_4$, filtered, and concentrated under reduced pressure to afford a colorless residue (0.25 g, 100%) which could be chromatographically purified (linear gradient of 20-40% EtOAc/hexanes) to obtain *anti*-**10c** (55 mg, 22%) and *syn*-**10c** (194 mg, 77%).

Reduction of ketone 11 using PtO_2, H_2.
To a stirred solution of the ketone (0.25 g, 1.04 mmol) in absolute EtOH (5 mL) was added PtO_2 (25 mg, 0.11 mmol). The reaction vessel was swept with H_2 gas and fitted with a two-way stopcock attached to a H_2 gas filled balloon. The resultant mixture was stirred vigorously for 18 h, diluted with Et_2O, then filtered through a pad of Celite. The filter cake was thoroughly washed with Et_2O and the combined filtrates concentrated under reduced pressure and chromatographically purified to afford *syn*-**10c** (81 mg, 34%).



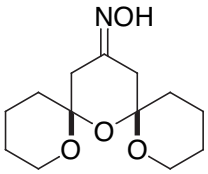
Methylation of alcohol *syn*-10c to afford methyl ether *syn*-12.
To a stirred slurry of alcohol *syn*-**10c** (0.11 g, 0.454 mmol) and Ag_2O (0.22 g, 0.949 mmol) in CH_3CN (10 mL) was added CH_3I (2.01 g, 14.1 mmol). The mixture was heated at 50° C for 48 h, then cooled to room temperature, diluted with Et_2O, and filtered through Celite. The filter cake was thoroughly washed with fresh Et_2O and the combined filtrates concentrated under reduced pressure. The resulting residue was chromatographed (linear gradient of 20-40% Et_2O/hexanes) to afford the methyl ether *syn*-**12** as a white solid (0.11 g, 92%).

R*f* 0.70 (30% Et_2O/hexanes).
Mp 54-57° C.
IR (NaCl, $CDCl_3$) 2945, 2869, 1470, 1447, 1435, 1366, 1335, 1255, 1200, 1159, 1073, 1047, 1021, 994, 976 cm^{-1}.
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.45 (ddd, J=4.2, 13.0, 13.0 Hz, 2H), 1.50-1.63 (m, 6H), 1.68 (ddd, J=2.5, 2.6, 12.6 Hz, 2H), 1.88 (ddddd, J=3.7, 3.7, 13.5, 13.7, 13.5 Hz, 2H), 1.99 (dd, J=0.13, 13.5 Hz, 2H), 2.01 (dd, J=15.37, 13.5 Hz, 2H), 3.30 (s, 3H), 3.53 (quint., J=15.37, 0.13 Hz, 1H), 3.66 (dddd, J=1.8, 2.1, 4.5, 12.0 Hz, 2H), 4.02 (ddd, J=3.0, 12.7, 11.9 Hz, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 19.01, 25.24, 36.93, 39.67, 55.61, 61.48, 71.30, 97.66 ppm.
HRMS Calcd for $C_{14}H_{24}O_4$: 256.1675. Found: 256.1690.



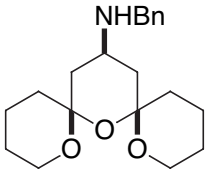
Methylation of alcohol *anti*-10c to afford methyl ether *anti*-12.
To a stirred slurry of alcohol *anti*-**10c** (0.25 g, 1.03 mmol) and Ag_2O (0.26 g, 1.12 mmol) in CH_3CN (5 mL) was added CH_3I (2.28 g, 16.1 mmol). The mixture was heated at 80° C for 20 h, then cooled to room temperature, diluted with Et_2O, and filtered through Celite. The filter cake was thoroughly washed with fresh Et_2O and the combined filtrates concentrated under reduced pressure. The resulting residue was chromatographed (linear gradient of 20-50% Et_2O/hexanes) to afford the methyl ether *anti*-**12** as a white solid (0.18 g, 68%).

R*f* 0.70 (30% Et_2O/hexanes).
Mp 39-43° C.
IR (NaCl, thin film) 3018, 2925, 2858, 1708, 1518, 1430, 1358, 1220, 1154, 1082, 1036, 973, 936, 900, 876, 844, 745, 664 cm^{-1}.
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.49 (ddd, J=4.3, 12.6, 12.8 Hz, 2H), 1.51-1.62 (m, 6H), 1.70 (ddd, J=3.7, 3.9, 12.5 Hz, 2H), 1.75-1.93 (m, 2H), 2.28 (ddd, J=1.2, 4.5, 12.2 Hz, 2H), 3.34 (s, 3H), 3.64 (dddd, J=1.6, 3.7, 3.7, 10.9 Hz, 2H), 3.79 (tt, J=4.5, 10.7 Hz, 1H), 4.99-4.06 (m, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 19.01, 25.34, 37.86, 41.70, 55.51, 62.24, 69.58, 98.25 ppm.
Anal. Calcd for $C_{14}H_{24}O_4$: C, 65.58; H, 9.44. Found: C, 65.53; H, 9.41.



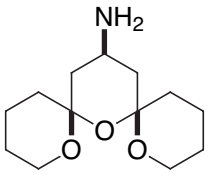
***Cis*-1,7,9-trioxaspiro[5.1.5.3]hexadecane-15-one oxime (13).**
To a cooled (0° C) solution of Na_2CO_3 (0.465 g, 4.39 mmol) and hydroxylamine hydrochloride (0.302 g, 4.39 mmol) in H_2O (5 mL) was introduced a solution of ketone **11** (1.00 g, 4.16 mmol) in Et_2O (5 mL). The stirred mixture was allowed to slowly warm to room temperature where it was maintained for 16 h. The reaction mixture was then partitioned between Et_2O and brine, the layers separated, and the combined organic layers dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The resulting solid was purified by chromatography (50% Et_2O/hexanes) to afford oxime **13** as a white solid (0.88 g, 83%).

R*f* 0.42 (70% Et_2O/hexanes).
Mp 128-132° C.
IR (NaCl, thin film) 3587, 3298, 2924, 1442, 1475, 1298, 1250, 1217, 1137, 1098, 1067, 1035, 950 cm^{-1}.
^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.29-2.03 (m, 12H), 2.40 (d, J=14.6 Hz, 1H), 2.53 (d, J=18.6 Hz, 1H), 2.68 (d, 18.6 Hz, 1H), 3.05 (d, J=14.6 Hz, 1H), 3.59-3.74 (m, 2H), 3.94 (ddd, J=4.3, 10.8, 11.0 Hz, 1H), 4.17 (ddd, J=3.3, 11.1, 11.1 Hz, 1H), 8.44-8.72 (br s, 1H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) d 18.67, 18.98, 24.94, 25.05, 36.17, 37.01, 38.77, 61.67, 61.78, 97.16, 97.23, 154.47 ppm.
Anal. Calcd for $C_{13}H_{21}NO_4$: C, 61.16; H, 8.29; N, 5.49. Found: C, 61.09; H, 9.32; N, 5.51.



Conversion of oxime 13 to benzylamine 14.
To a stirred solution of oxime **13** (0.48 g, 1.88 mmol) and $NiCl_2•(H_2O)_6$ (1.03 g, 4.32 mmol) in MeOH (18 mL) at 0° C was added $NaBH_4$ (0.684 g, 18.1 mmol) in portions over 30 min. The resulting black mixture was stirred at –20° C for 1 h then filtered through Celite. The filter cake was thoroughly washed with fresh MeOH and the combined dark filtrates decolorized with $MgSO_4$. Following filtration, the decolorization using $MgSO_4$ was repeated, filtered, and the result concentrated under reduced pressure. The resulting residue was dissolved in MeOH, filtered through a plug of basic alumina, and the filtrate concentrated under reduced pressure (0.1 torr) to afford the crude amine as an amorphous brown solid. This residue was dissolved in benzaldehyde (0.60 g, 5.65 mmol) and $Ti(O^iPr)_4$ (2.06 g, 7.26 mmol). After stirring for 1 h, absolute EtOH (6 mL) and $NaBH_3CN$ (0.25 g, 3.98 mmol) were introduced and the result stirred for 24 h. At this time, the mixture was treated with H_2O (1 mL) and the resulting solids removed by filtration (sintered glass funnel). The solids were washed with EtOAc, the combined filtrates concentrated under reduced pressure, and the resulting residue purified by chromatography (EtOAc) to afford benzylamine **14** as a white solid (0.38 g, 61%).

R*f* 0.28 (EtOAc).
Mp 152-153° C.
IR (NaCl, $CDCl_3$) 3167, 2953, 2312, 2165, 1434, 1383, 1208, 1170, 1075, 1040, 965, 933 cm^{-1}.
^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.76 (dd, J=3.2, 14.9 Hz, 2H), 1.53-1.96 (m, 12H), 2.49 (dd, J=2.1, 14.7 Hz, 2H), 3.56-3.76 (m, 3H), 3.97-4.06 (m, 2H), 4.15 (s, 2H), 7.31-7.60 (m, 5H), 8.40 (br s, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.18, 24.64, 35.10, 36.72, 49.04, 50.61, 62.80, 96.79, 129.49, 129.70, 129.75, 130.73 ppm.
HRMS calcd for $C_{20}H_{29}NO_3$: 331.2149. Found: 331.2160.



Data for crude amine intermediate:
^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.99-2.15 (m, 12H), 2.54 (d, J=14.1 Hz, 2H), 3.40-4.45 (m, 5H), 8.29 (br s, 2H) ppm.
^{13}C NMR ($CDCl_3$, 75 MHz) δ 18.61, 24.74, 35.33, 36.90, 44.25, 62.61, 96.84 ppm.

X-Ray Crystallographic Data for Compound *anti*-12

A. Crystal Data

Emperical Formula	$C_{14}H_{24}O_4$
Formula Weight	256.34
Crystal Color, Habit	colorless, prism
Crystal Dimensions (mm)	0.460 x 0.320 x 0.250
Crystal System	orthorhombic
Lattice Parameters:	a = 9.278 (3) Å b = 15.418 (5) Å c = 9.443 (3) Å
	V = 1351 (1) $Å^3$
Space Group	$Pna2_1$ (#33)
Z	4
D_{calc}	1.260 g/cm^3
$\mu_{(MoK\alpha)}$	0.85 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC6S
Radiation	MoKα (λ = 0.71069 Å)
Temperature	-100° C
Scan Type	ω-2θ
$2\theta_{max}$	50.0°
Number of Reflections Measured	Total: 1406
Corrections	Lorentz-polarization Secondary Extinction (coefficient: 0.26072E-05)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR88)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w (\|Fo\| - \|Fc\|)^2$
Number of Observations ($I > 3.00\sigma(I)$)	927
Number of Variables	163
Reflection/Parameter Ratio	5.69
Residuals: R; R_w	0.053; 0.065
Goodness of Fit Indicator	2.28
Maximum Peak in Final Diffraction Map	0.29 $e^-/Å^3$

Table II. Positional Parameters

Atom	x	y	z
O(1)	0.1399 (4)	1.1029 (2)	0.0336
O(2)	-0.2449 (4)	0.9707 (3)	0.1644 (6)
O(7)	0.2034 (3)	0.9999 (2)	0.2099 (5)
O(9)	0.1706 (4)	0.8983 (2)	0.0239 (6)
C(2)	0.2837 (6)	1.1297 (4)	-0.0062 (8)
C(3)	0.3369 (6)	1.2034 (4)	0.0820 (8)
C(4)	0.3348 (6)	1.1774 (4)	0.2376 (8)
C(5)	0.1824 (6)	1.1480 (4)	0.2787 (8)
C(6)	0.1243 (6)	1.0771 (3)	0.1785 (7)
C(8)	0.1464 (6)	0.9163 (3)	0.1698 (8)
C(10)	0.3181 (6)	0.8898 (4)	-0.0175 (8)
C(11)	0.3951 (7)	0.8218 (4)	0.0685 (8)
C(12)	0.3847 (7)	0.8425 (4)	0.2267 (7)
C(13)	0.2252 (6)	0.8494 (4)	0.2630 (8)
C(14)	-0.0141 (6)	0.9093 (3)	0.1928 (7)
C(15)	-0.0956 (6)	0.9867 (3)	0.1305 (7)
C(16)	-0.0380 (6)	1.0657 (3)	0.1998 (7)
C(17)	-0.3412 (6)	1.0247 (4)	0.0876 (8)
H(2A)	0.2731	1.1444	-0.1017
H(2B)	0.3439	1.0740	-0.0086
H(3A)	0.4342	1.2275	0.0580
H(3B)	0.2610	1.2581	0.0610
H(4A)	0.3974	1.1269	0.2604
H(4B)	0.3781	1.2275	0.3024
H(5A)	0.2001	1.1207	0.3826
H(5B)	0.1277	1.2038	0.2673
H(10A)	0.3285	0.8920	-0.1176
H(10B)	0.3877	0.9458	-0.0106
H(11A)	0.4329	0.7760	0.0387
H(11B)	0.4957	0.8179	0.0430
H(12A)	0.4385	0.8982	0.2664
H(12B)	0.4358	0.7922	0.2791
H(13A)	0.2215	0.8739	0.3721
H(13B)	0.1657	0.7920	0.2594
H(14A)	-0.0368	0.9038	0.2948
H(14B)	-0.0520	0.8584	0.1669
H(15)	-0.0612	0.9659	0.0257
H(16A)	-0.0807	1.1197	0.1681
H(16B)	-0.0578	1.0871	0.2919
H(17A)	-0.4598	1.0117	0.1386
H(17B)	-0.3242	1.0957	0.1024
H(17C)	-0.3384	0.9898	-0.0350

Table III. Bond Lengths (Å)

O1	C2	1.447 (7)	C4	C5	1.535 (8)
O1	C6	1.432 (7)	C5	C6	1.543 (8)
O2	C15	1.442 (6)	C6	C16	1.530 (7)
O2	C17	1.420 (7)	C8	C13	1.541 (8)
O7	C6	1.431 (6)	C8	C14	1.508 (7)
O7	C8	1.443 (6)	C10	C11	1.507 (8)
O9	C8	1.424 (7)	C11	C12	1.530 (8)
O9	C10	1.429 (7)	C12	C13	1.523 (8)
C2	C3	1.493 (8)	C14	C15	1.530 (7)
C3	C4	1.525 (8)	C15	C16	1.482 (7)

Table IV. Bond Angles (deg)

C2	O1	C6	114.9 (4)	O7	C8	C13	105.9 (4)
C15	O2	C17	113.0 (4)	O7	C8	C14	112.8 (4)
C6	O7	C8	120.1 (4)	O9	C8	C13	110.3 (5)
C8	O9	C10	115.7 (4)	O9	C8	C14	106.3 (5)
O1	C2	C3	112.2 (5)	C13	C8	C14	109.8 (5)
C2	C3	C4	109.5 (5)	O9	C10	C11	111.7 (5)
C3	C4	C5	109.5 (5)	C10	C11	C12	110.5 (5)
C4	C5	C6	112.1 (5)	C11	C12	C13	107.2 (5)
O1	C6	O7	112.2 (4)	C8	C13	C12	112.3 (5)
O1	C6	C5	110.8 (4)	C8	C14	C15	112.2 (4)
O1	C6	C16	104.9 (4)	O2	C15	C14	104.9 (4)
O7	C6	C5	106.5 (4)	O2	C15	C16	112.9 (4)
O7	C6	C16	112.4 (4)	C14	C15	C16	107.0 (5)
C5	C6	C16	110.2 (5)	C6	C16	C15	113.1 (4)
O7	C8	O9	111.7 (4)				

Table V. Thermal Displacement Parameters

Atom	U11	U22	U33	U12	U13	U23
O(1)	0.021 (2)	0.026 (2)	0.019 (2)	-0.002 (2)	-0.000 (2)	0.003 (2)
O(2)	0.025 (2)	0.044 (2)	0.030 (2)	-0.010 (2)	-0.001 (2)	0.008 (2)
O(7)	0.022 (2)	0.023 (2)	0.019 (2)	-0.003 (2)	-0.001 (2)	-0.002 (2)
O(9)	0.032 (2)	0.031 (2)	0.014 (2)	0.001 (2)	-0.001 (2)	-0.003 (2)
C(2)	0.037 (3)	0.030 (3)	0.023 (3)	-0.002 (3)	0.002 (3)	0.007 (3)
C(3)	0.031 (4)	0.033 (4)	0.033 (4)	-0.010 (3)	-0.004 (3)	0.009 (3)
C(4)	0.035 (4)	0.028 (3)	0.028 (3)	-0.009 (3)	-0.012 (3)	0.002 (3)
C(5)	0.032 (3)	0.023 (3)	0.025 (3)	0.000 (3)	0.002 (3)	-0.002 (2)
C(6)	0.029 (3)	0.022 (3)	0.016 (3)	-0.000 (2)	0.002 (3)	0.004 (3)
C(8)	0.034 (3)	0.024 (3)	0.019 (3)	-0.003 (2)	-0.004 (3)	-0.002 (3)
C(10)	0.038 (4)	0.040 (4)	0.021 (3)	0.011 (3)	0.003 (3)	-0.002 (3)
C(11)	0.043 (4)	0.042 (4)	0.033 (4)	0.026 (3)	0.005 (3)	-0.005 (3)
C(12)	0.046 (4)	0.033 (3)	0.026 (4)	0.017 (3)	-0.011 (3)	-0.000 (3)
C(13)	0.042 (4)	0.020 (3)	0.019 (3)	-0.003 (3)	-0.007 (3)	-0.000 (2)
C(14)	0.034 (3)	0.023 (3)	0.017 (3)	-0.006 (2)	0.002 (3)	-0.004 (3)
C(15)	0.018 (3)	0.035 (4)	0.022 (3)	-0.002 (2)	0.005 (2)	-0.001 (3)
C(16)	0.021 (3)	0.027 (3)	0.023 (3)	0.006 (2)	0.002 (3)	-0.001 (3)
C(17)	0.028 (3)	0.035 (4)	0.035 (3)	0.000 (3)	-0.003 (3)	0.006 (3)