Supporting Information

Modular Terpenoid Construction *via* Catalytic Enantioselective Formation of All-Carbon Quaternary Centers: Total Synthesis of Oridamycin A, Triptoquinone B and C and Isoiresin

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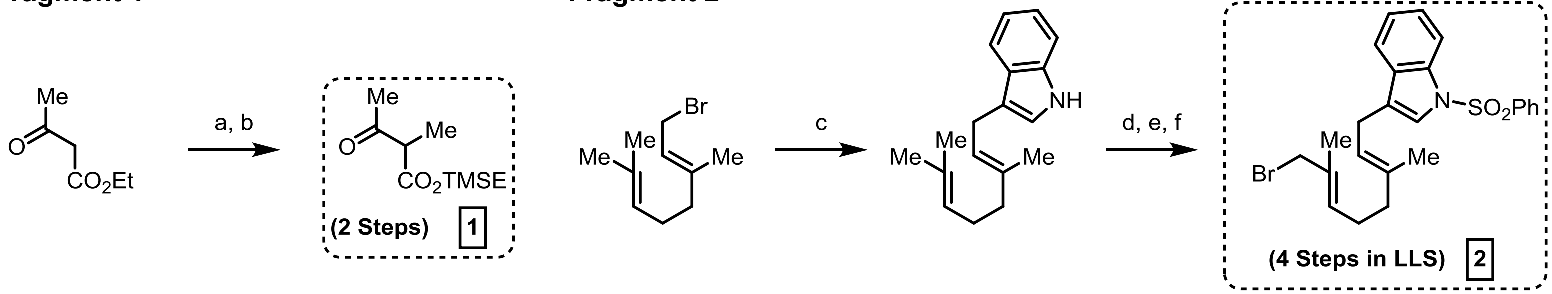
Summaries of Prior Syntheses of Oridamycins, Xiamycins, Triptoquinones, Isoiresin and Iresin

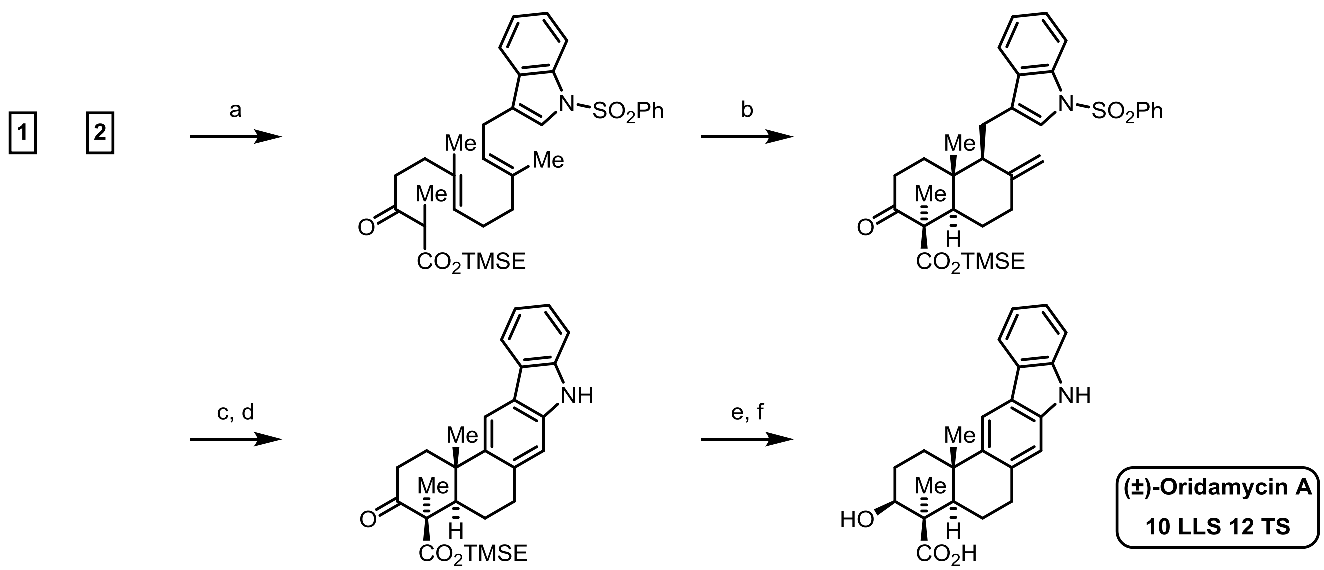
A. Li *et al. Nat. Commun.* **2015**, *6*, 6096. (racemic)

Fragment 1 **Fragment 2**



Key: (a) TMSEOH; (b) MeI, K_2CO_3; (c) Indole, NH_4HCO_3; (d) $PhSO_2Cl$, TBAB, NaOH; (e) SeO_2, TBHP; (f) MsCl, Et_3N, LiBr.

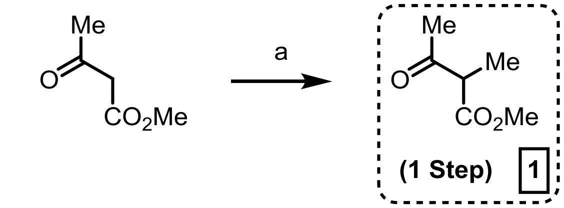
Fragment Union and End Game



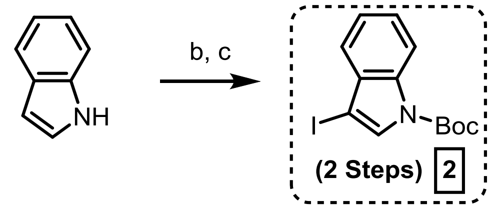
Key: (a) KH, then *n*BuLi, HMPA; (b) $Mn(OAc)_3 \cdot 3H_2O$, $Cu(OAc)_2 \cdot 2H_2O$; (c) Mg, NH_4Cl; (d) $Pd(OAc)_2$, 1,4-benzoquinone; (e) $NaBH_4$, $CeCl_3 \cdot 7H_2O$; (f) TASF.

B. Trotta *Org. Lett.* **2015**, *17*, 3358. (racemic)

Fragment 1

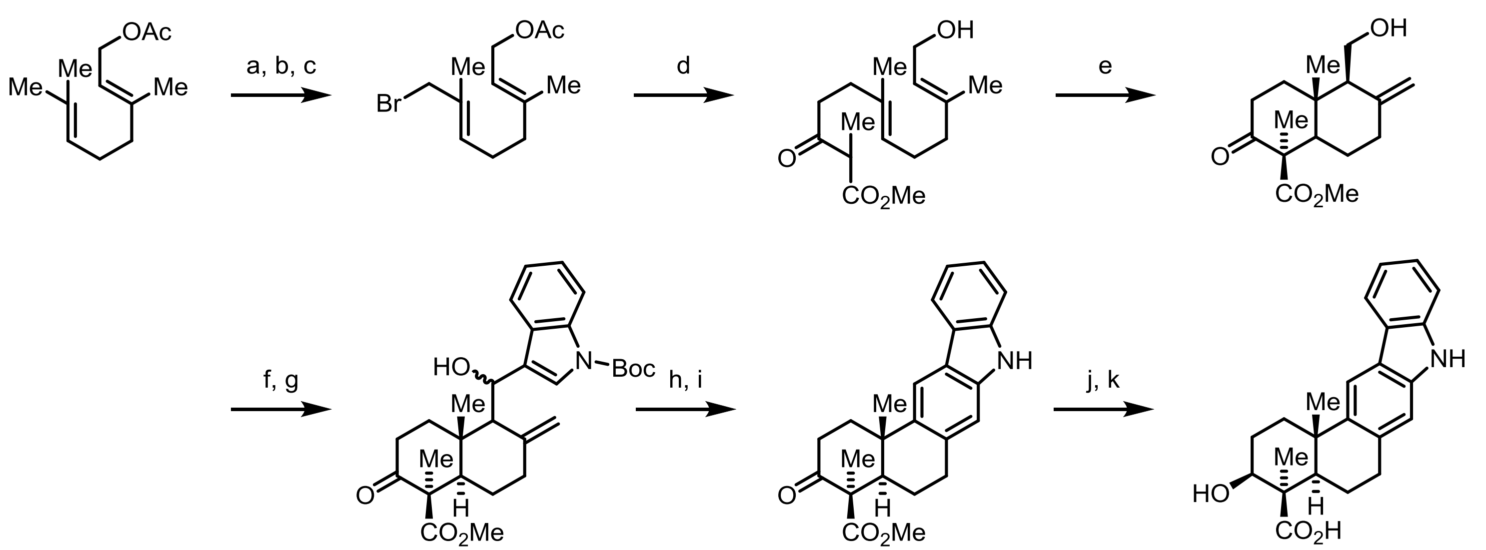


Fragment 2



Key: (a) MeI, K_2CO_3; (b) KOH, I_2; (c) Boc_2O, Et_3N, DMAP.

Fragment Union and End Game

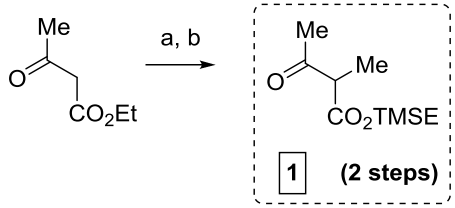


(±)-Oridamycin A
11 LLS 14 TS

Key: (a) SeO_2; (b) $NaBH_4$; (c) MsCl, Et_3N, LiBr; (d) **1**, NaH, then *n*BuLi, HMPA; (e) $Mn(OAc)_3 \cdot 3H_2O$, $Cu(OAc)_2 \cdot 2H_2O$; (f) DMP; (g) **2**, EtMgBr; (h) TFA; (i) Air, then TFA; (j) $NaBH_4$; (k) NaCN.

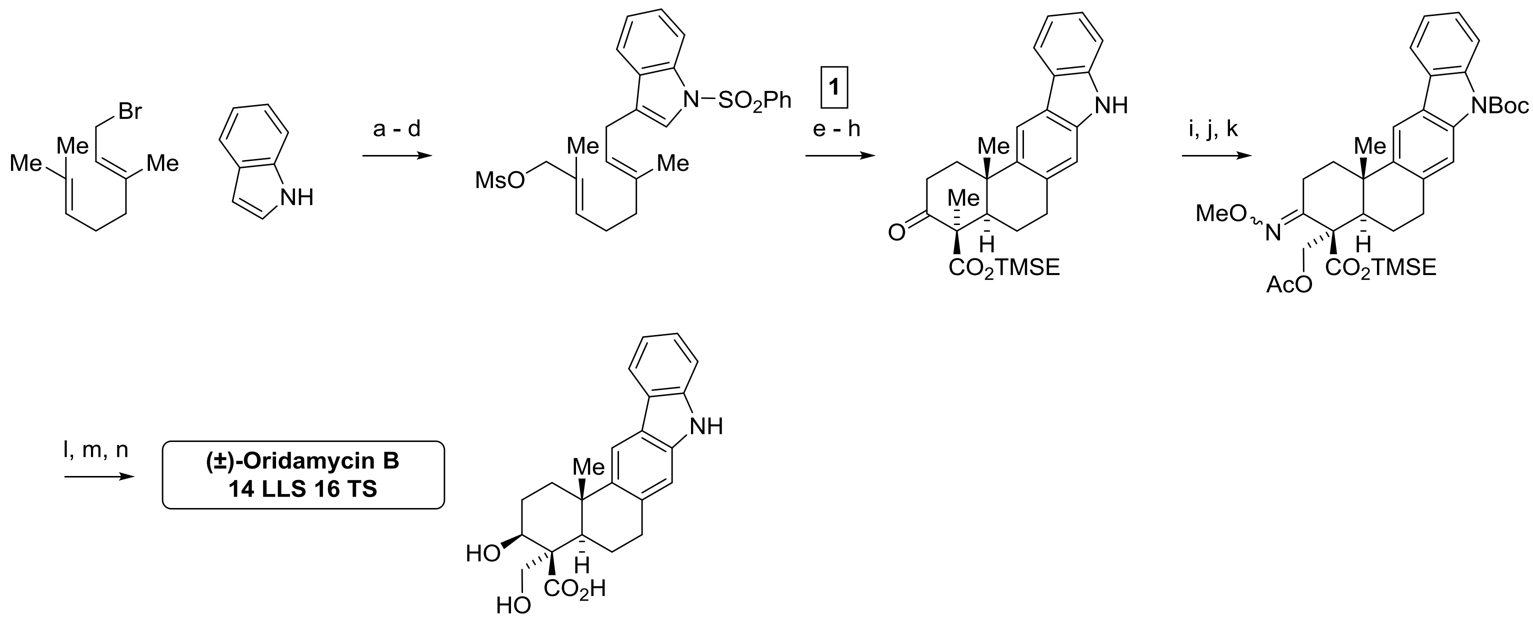
C. Li *et al. Nat. Commun.* **2015**, *6*, 6096. (racemic)

Fragment 1



Key: a) TMSEOH; b) MeI, K_2CO_3.

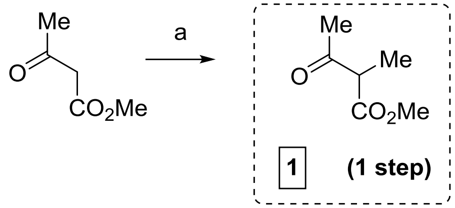
Completion of Synthesis



Key: a) NH_4HCO_3; b) $PhSO_2Cl$, TBAB, NaOH; c) SeO_2, TBHP; d) MsCl, NEt_3, LiBr; e) **1**, KH, then *n*-BuLi; f) $Mn(OAc)_3$-$2H_2O$, $Cu(OAc)_2$-H_2O; g) Mg, NH_4Cl; h) $Pd(OAc)_2$, benzoquinone; i) NH_2OMe; j) Boc_2O, DMAP; k) $Pd(OAc)_2$, $PhI(OAc)_2$; l) $HClO_4$; m) $NaBH_4$; n) TASF.

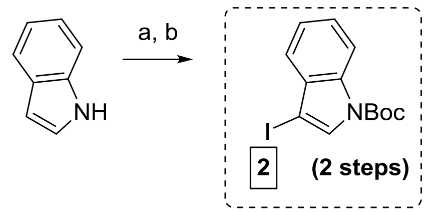
D. Trotta *Org. Lett*. **2015**, *17*, 3358. (racemic)

Fragments 1



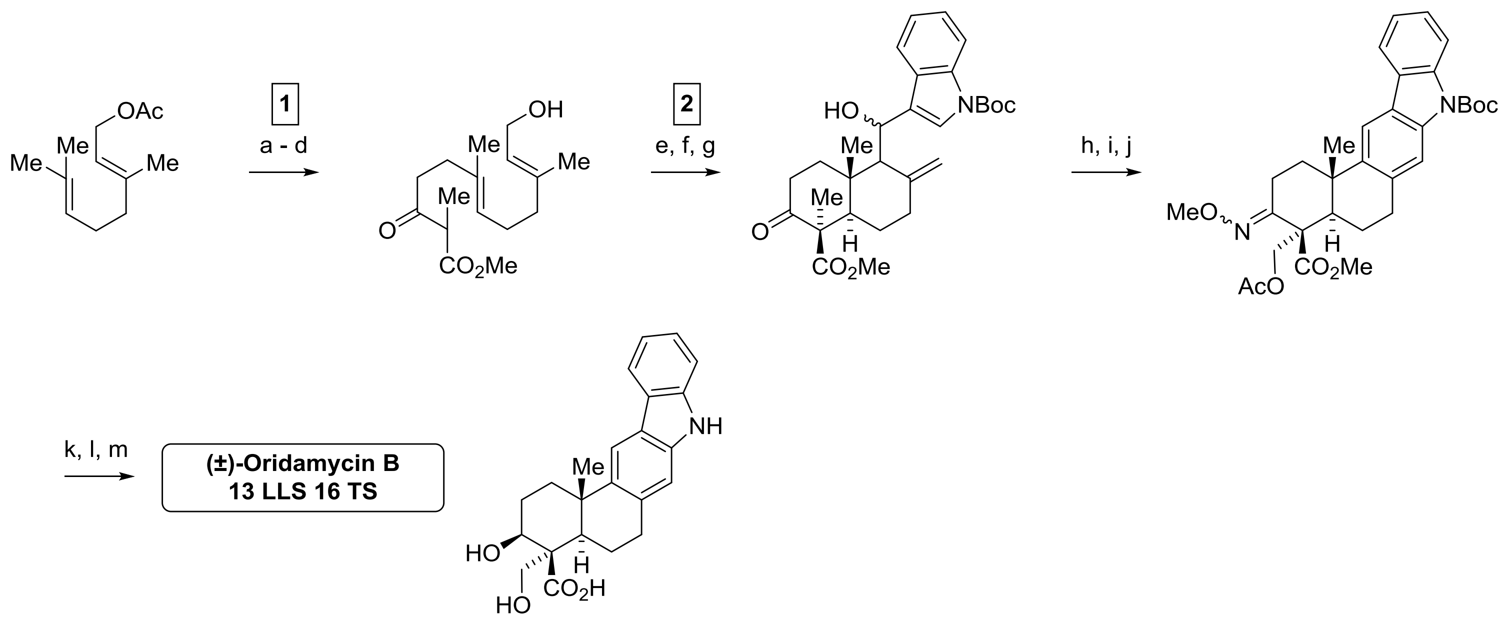
Key: a) MeI, K_2CO_3.

Fragments 2



Key: a) KOH, I_2; b) Boc_2O, Et_3N, DMAP.

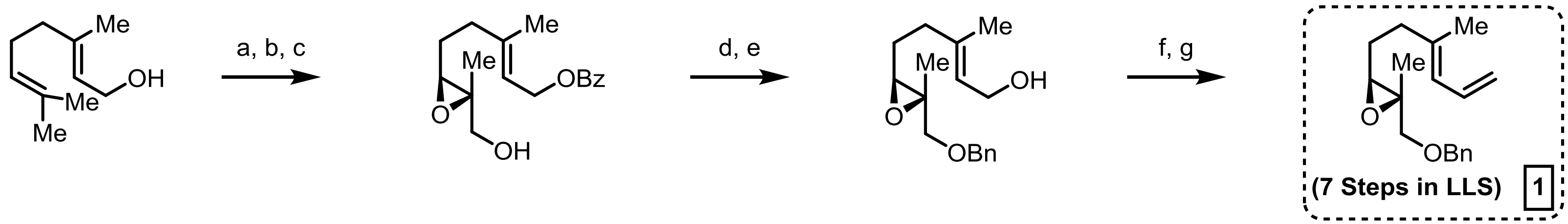
Completion of Synthesis



Key: a) SeO_2; b) $NaBH_4$; c) MsCl, NEt_3, then LiBr; d) **1**, NaH, then *n*-BuLi; e) $Mn(OAc)_2$, $Cu(OAc)_2$; f) DMP; g) **2**, EtMgBr; h) TFA; i) $MeONH_2$-HCl, pyridine; j) $Pd(OAc)_2$, $PhI(OAc)_2$; k) HCl; l) $NaBH_4$; m) NaCN.

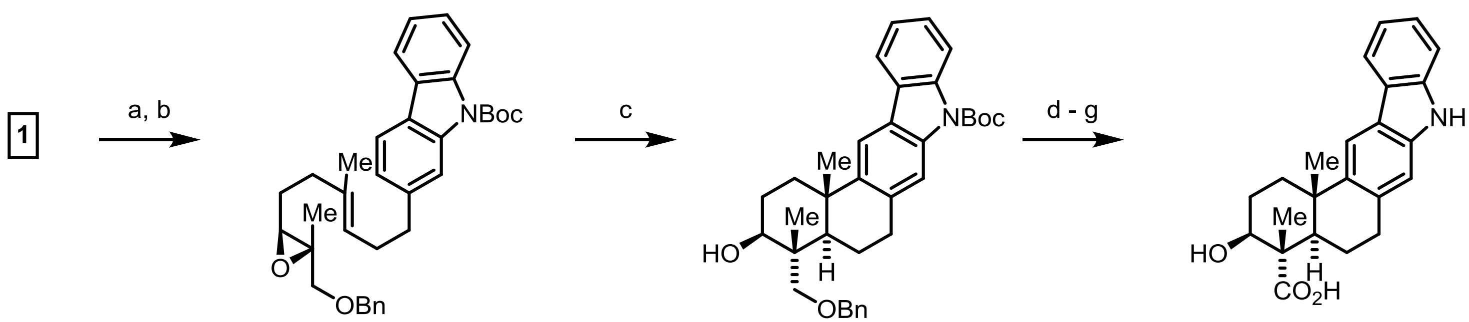
E. Baran *et al. J. Am. Chem. Soc.* **2014**, *136*, 5571.

Fragment 1



Key: (a) BzCl, py, DMAP; (b) SeO_2, TBHP; (c) (+)-DIPT, Ti(O*i*Pr)$_4$, TBHP, 3A MS; (d) BnBr, NaH, TBAI; (e) NaOMe, TBAI; (f) SO_3•py, Et_3N; (g) Ph_3PMeI, *n*BuLi.

Fragment Union and End Game

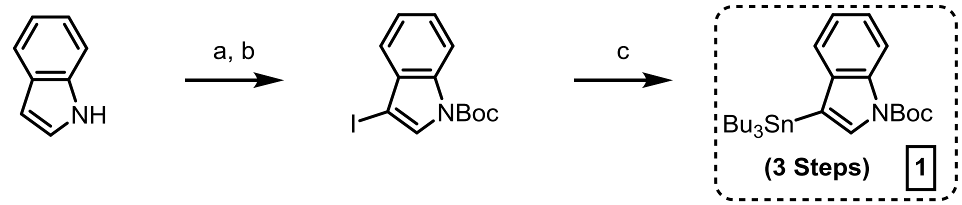


Key: (a) **1**, 9-BBN, then 2-bromo-9H-carbazole, Pd(dppf)Cl_2, NaOH; (b) Boc_2O, Et_3N, DMAP; (c) BF_3•OEt_2; (d) H_2, Pd(OH)$_2$/C; (e) TEMPO, NCS, TBAI, $NaHCO_3$/$KHCO_3$; (f) $NaClO_2$, NaH_2PO_4•H_2O, 2-methyl-2-butene; (g) EtOH, H_2O.

Xiamycin A
14 LLS 14 TS

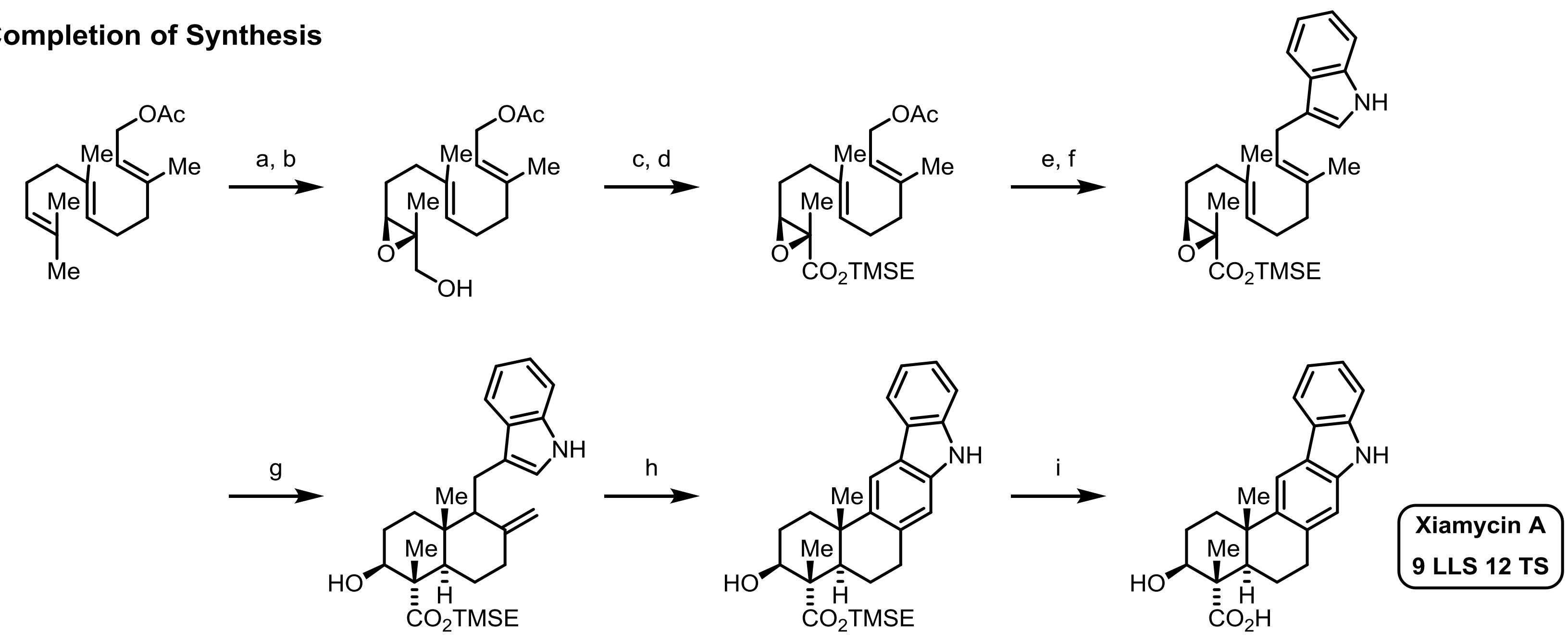
F. Li *et al. Nat. Commun.* **2015**, *6*, 6096.

Fragment 1



Key: (a) ICl, py; (b) Boc_2O, DMAP; (c) *n*BuLi, TMEDA, then Bu_3SnCl.

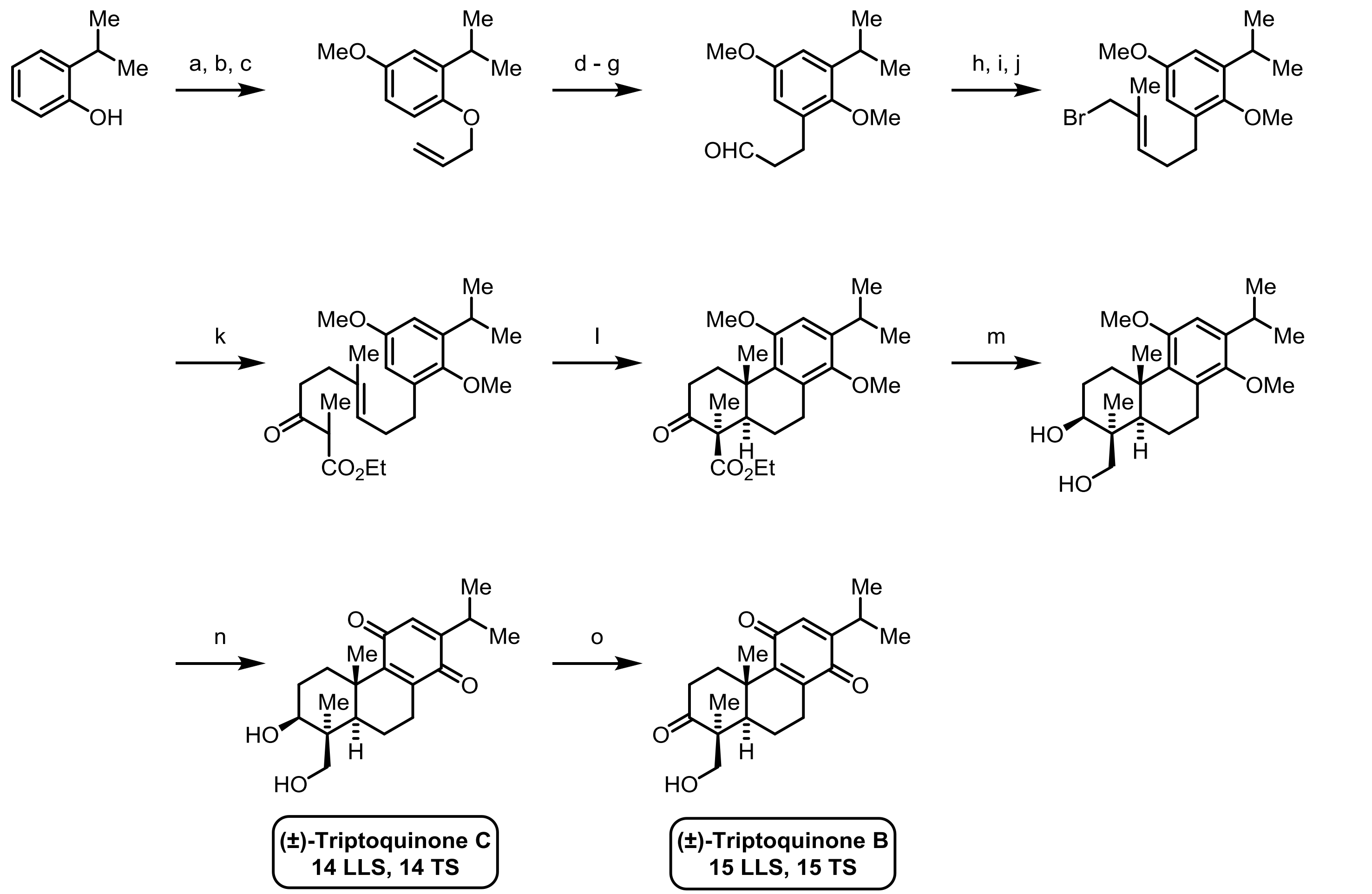
Completion of Synthesis



Key: (a) SeO_2, TBHP; (b) (+)-DET, Ti(O*i*Pr)$_4$, TBHP; (c) AZADO, PhI(OAc)$_2$; (d) TMSEOH, EDC•HCl; (e) **1**, $Pd_2(dba)_3$, LiCl; (f) DMSO, 150 °C; (g) Cp_2TiCl_2, Mn, DIPEA, TMSCl; (h) Pd(OAc)$_2$, 1,4-benzoquinone, AcOH; (i) TASF.

G. Shishido *et al. J. Chem. Soc., Chem. Commun.* **1993**, 793. (racemic)

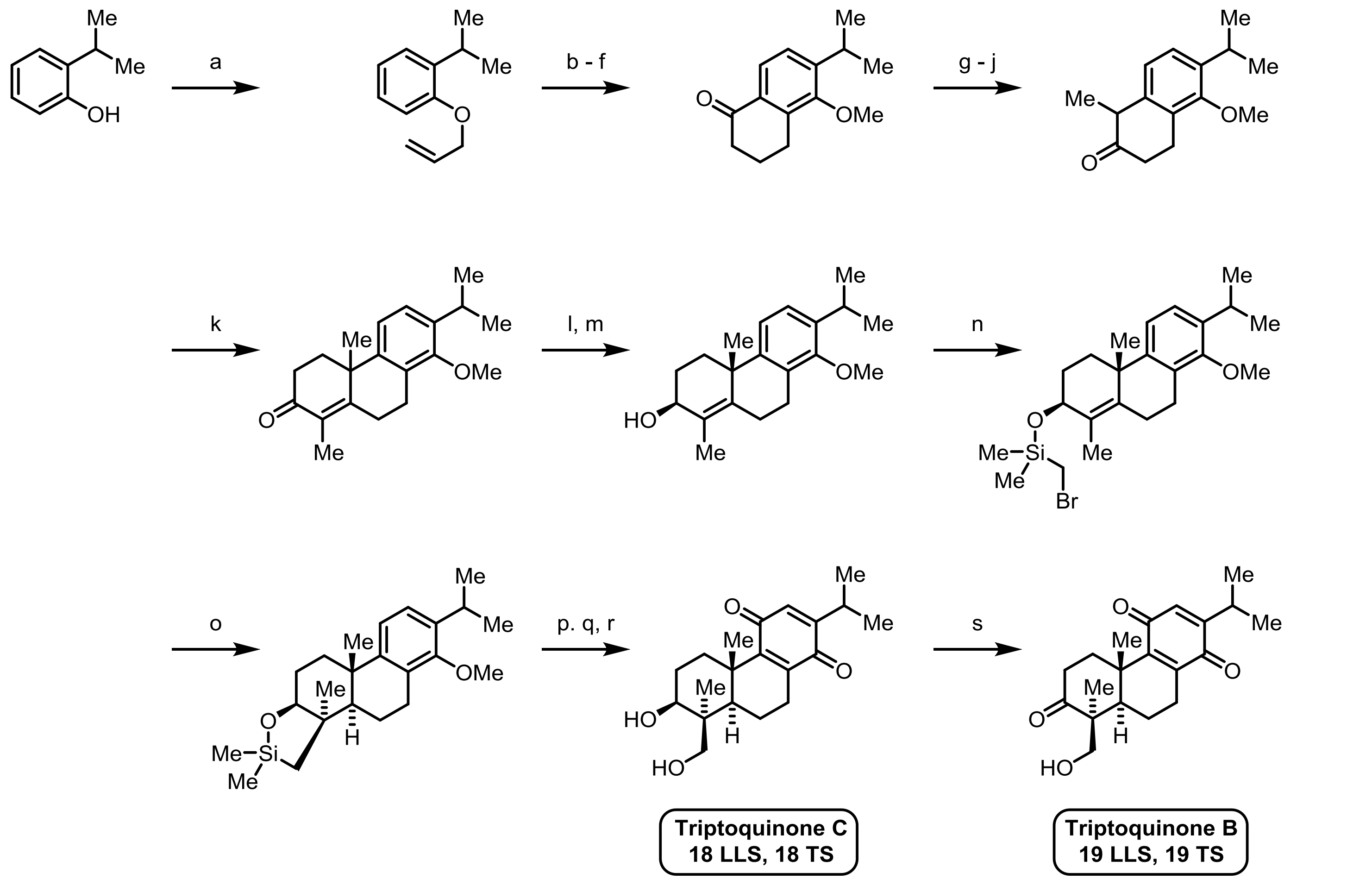
Completion of Synthesis



Key: (a) Br_2; (b) Allyl bromide, K_2CO_3; (c) NaOMe, CuI, MeOH; (d) 200 °C; (e) Me_2SO_4, K_2CO_3; (f) BH_3•SMe_2, then H_2O_2, NaOH; (g) $(COCl)_2$, DMSO, Et_3N; (h) PPh_3=C(Me)CO_2Et; (i) DIBAL; (j) PPh_3, CBr_4; (k) Ethyl methylacetoacetate, NaH, *n*BuLi, HMPA; (l) $Mn(OAc)_3$, AcOH; (m) $LiAlH_4$; (n) $(NH_4)_2Ce(NO_3)_6$; (o) 7% NaClO (aq.), AcOH.

H. Shishido *et al. Tetrahedron Lett.* **1997**, *38*, 4121.

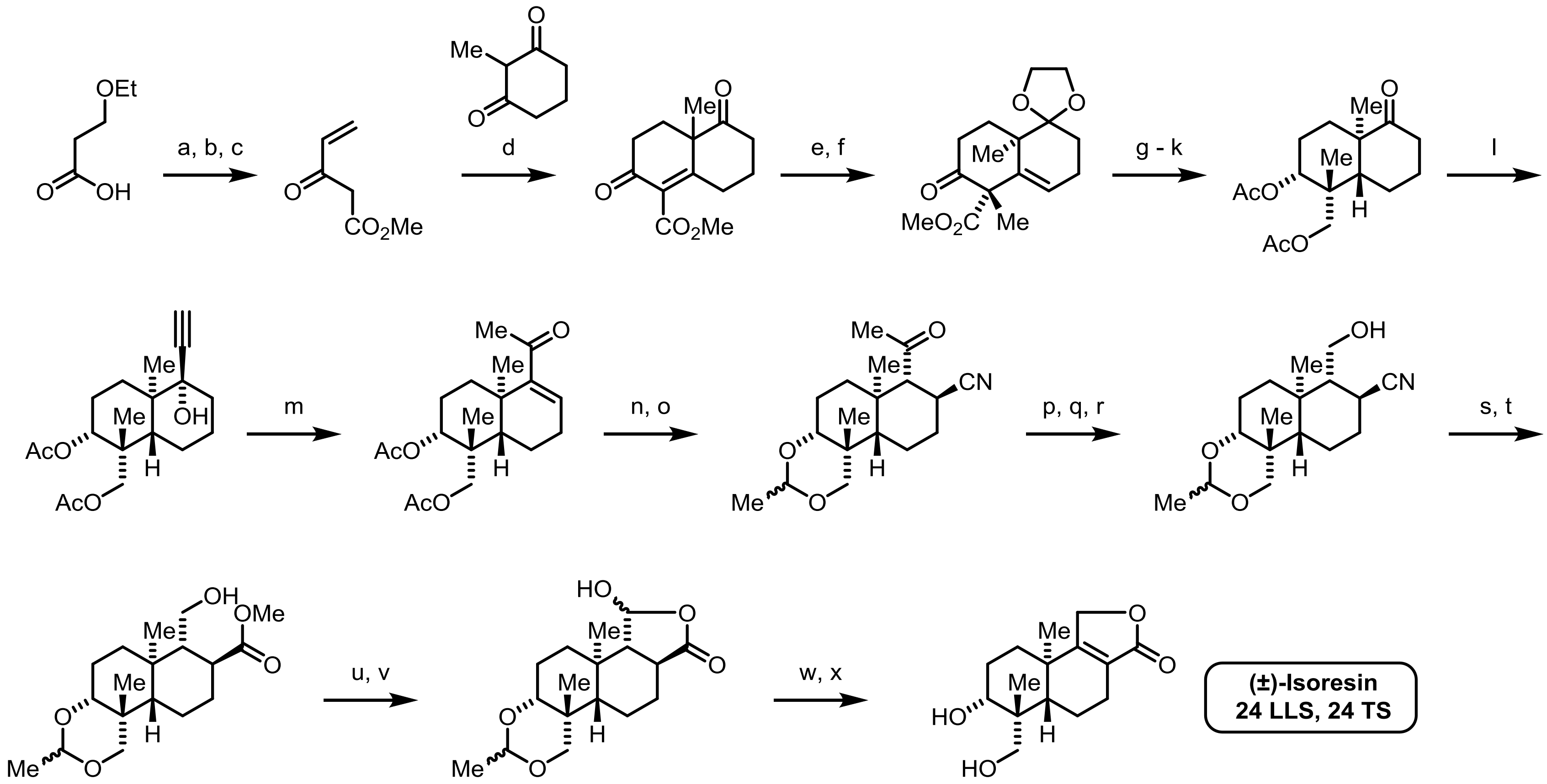
Completion of Synthesis



Key: (a) Allyl bromide, K_2CO_3; (b) 200 °C; (c) Me_2SO_4, K_2CO_3; (d) 9-BBN, CO, $LiAlH(OtBu)_3$, then H_2O_2, NaH_2PO_4, K_2HPO_4•$2H_2O$; (e) H_2NSO_3H, $NaClO_2$; (f) PPA; (g) MeMgI; (h) *p*-TsOH; (i) BH_3•SMe_2, then H_2O_2, NaOH; (j) $(COCl)_2$, DMSO, Et_3N; (k) EVK, KOH; (l) $NaBH_4$, $CeCl_3$; (m) NOVOZYM 435, vinyl acetate; (n) $BrCH_2SiMe_2Cl$, Et_3N; (o) $NaBH_3CN$, Bu_3SnCl, AIBN; (p) Na_2CO_3, H_2O_2; (q) EtSH, $AlCl_3$; (r) $(KSO_3)_2NO$, KH_2PO_4; (s) 7% NaClO (aq.), AcOH.

I. Pelletier *et al. J. Am. Chem. Soc.* **1968**, *90*, 5318.

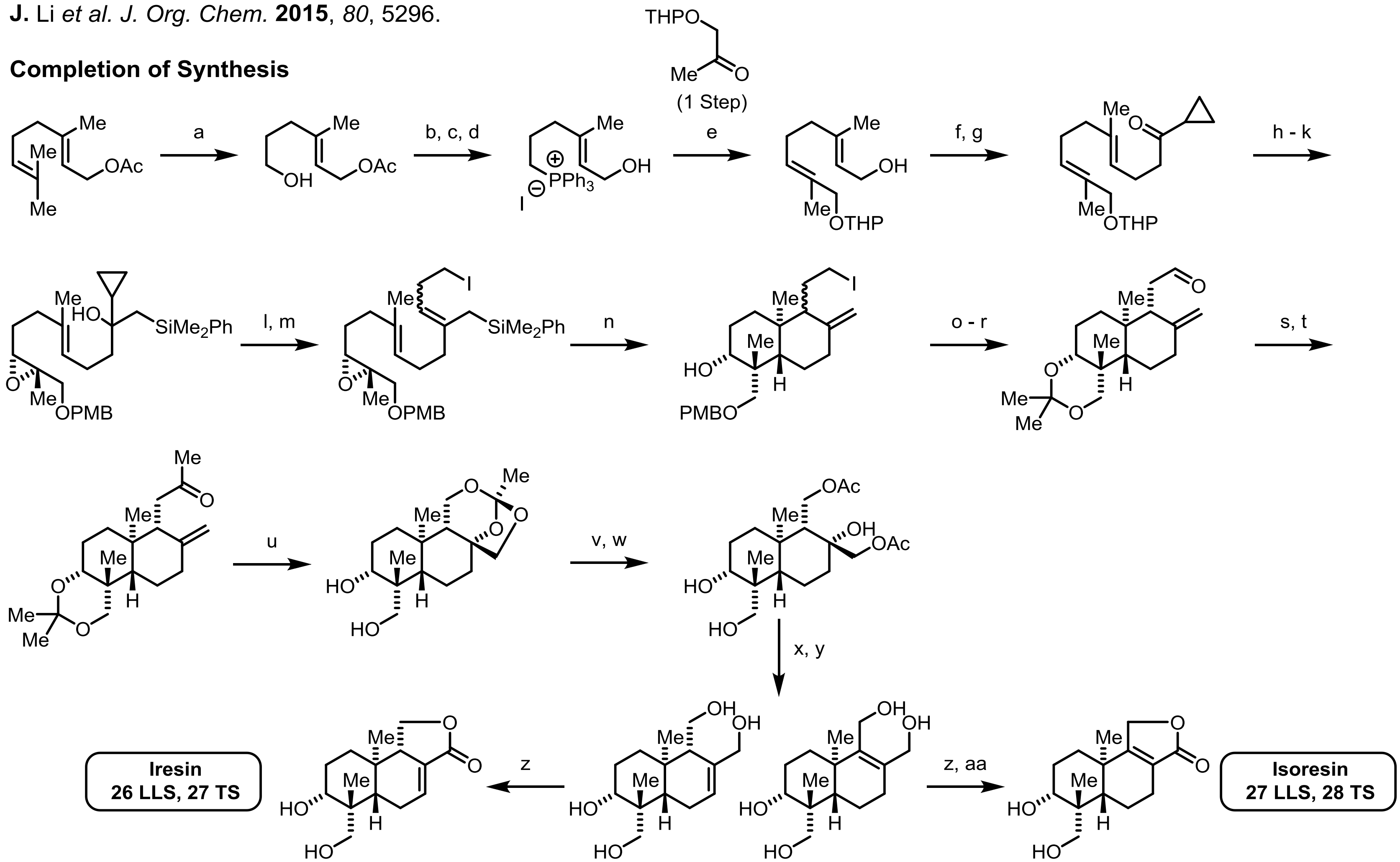
Completion of Synthesis



Key: (a) $SOCl_2$; (b) NaH, methyl acetoacetate, then NH_3 (g); (c) PTSA; (d) KF, MeOH; (e) PTSA; (f) *t*-BuOK, MeI; (g) $NaBH_4$; (h) H_2, Pd/C; (i) $LiAlH_4$; (j) Ac_2O, pyridine; (k) PTSA, acetone-H_2O; (l) C_2H_2, $NaNH_2$; (m) HCO_2H; (n) NaCN, EtOH; (o) CH_3CHO, $ZnCl_2$; (p) NaBrO; (q) base, $(COCl)_2$; (r) $LiAl(O\textit{t}\text{-}Bu)_3H$; (s) base, H_2O; (t) CH_2N_2; (u) DCC, DMSO; (v) K_2CO_3, MeOH; (w) Al_2O_3-pyridine, 235 °C; (x) acid, H_2O.

J. Li *et al. J. Org. Chem.* **2015**, *80*, 5296.

Completion of Synthesis



Key: (a) O_3, pyridine, then $NaBH_4$, MeOH; (b) I_2, Ph_3P, imid; (c) K_2CO_3, MeOH; (d) PPh_3; (e) *n*-BuLi; (f) I_2, Ph_3P, imid; (g) cyclopropyl methyl ketone, LDA; (h) PTSA; (i) Ti(O*i*-Pr)$_4$, L-(+)-DIPT, *t*-BuOOH, CaH_2, silica gel, 4A MS; (j) PMBBr, NaH, TBAI; (k) $PhMe_2SiCH_2MgCl$, $CeCl_3$; (l) MgI_2•$(OEt_2)_n$; (m) K_2CO_3; (n) $SnCl_4$; (o) DDQ; (p) K_2CO_3; (q) $Me_2C(OMe)_2$, PPTS; (r) DMSO, $NaHCO_3$; (s) CH_3Li; (t) $(COCl)_2$, DMSO, Et_3N; (u) TFAA, H_2O_2 (50% aq.), NaH_2PO_4; (v) 6N HCl; (w) Ac_2O, DMAP, pyridine; (x) Burgess reagent; (y) NaOMe, MeOH; (z) Ag_2CO_3/Celite; (aa) $NaBH_4$.

General Information

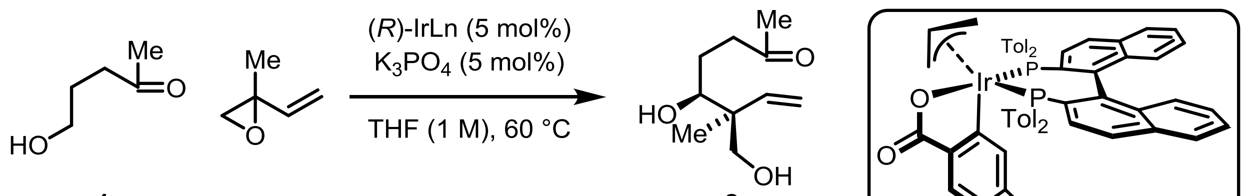
All reactions were performed under an atmosphere of argon, unless specifically noted in detailed procedures. Tetrahydrofuran, diethyl ether and toluene were distilled from sodium-benzophenone immediately prior to use. Dichloromethane, 1,2-dichloroethane were distilled from calcium hydride prior to use. Anhydrous solvents were transferred by oven-dried syringes and needles. Reagents purchased from commercial sources were used as received, or purified via Hickman distillation over appropriate drying agent. Analytical thin-layer chromatography (TLC) was carried out using 0.25 mm commercial silica gel plates (Dynanmic Absorbents F_{254}). Visualization was accomplished with UV light followed by dipping in appropriate stain solution then heating. Flash column chromatography was performed on Sorbent silica gel (40-63 μm, unless indicated specifically) or Sigma-Aldrich aluminum oxide (activated, neutral, Brockmann I, ~150 mesh, 58 Å pore size).

Spectroscopy, Spectrometry, and Data Collection

Infrared spectra were recorded on a Perkin-Elmer 1600 spectrometer. High-resolution mass spectra (HRMS) were obtained on an Agilent Technologies 6530 Accurate Mass Q-Tof LC/MS instrument for electrospray ionisation (ESI) or a Micromass Autospec Ultima instrument for chemical ionization (CI), and are reported as m/z (relative intensity). Accurate masses are reported for the molecular ion (M, M+H, M-H or M+Na), or a suitable fragment ion. ^{1}H Nuclear magnetic resonance spectra were recorded using an Agilent MR (400 MHz), Varian DirectDrive (400, 600 MHz), or Varian INOVA (500 MHz) spectrometer in $CDCl_3$ or CD_3OD solution. Coupling constants are reported in Hertz (Hz) with one decimal place, and chemical shifts are reported as parts per million (ppm) relative to residual solvent peaks ($CDCl_3$ δ_H 7.26 ppm; CD_3OD δ_H 3.31 ppm). ^{13}C Nuclear magnetic resonance spectra were recorded using an Agilent MR (400 MHz), Varian DirectDrive (400, 600 MHz), or Varian INOVA (500 MHz) spectrometer in $CDCl_3$ or CD_3OD solution, and chemical shifts are reported as parts per million (ppm) relative to solvent peaks ($CDCl_3$ δ_C 77.0 ppm; CD_3OD δ_C 49.0 ppm). Specific optical rotations ($[\alpha]_D$) were obtained on an Atago AP-300 automatic polarimeter at the sodium line (589.3 nm) in $CHCl_3$ or CH_3OH solution. Melting points were taken on a Stuart SMP3 melting point apparatus or SRS OptiMelt automated melting point system.

Detailed Procedures and Spectral Data for Asymmetric Synthesis of Oridamycin A

(5*S*,6*S*)-5-Hydroxy-6-(hydroxymethyl)-6-methyloct-7-en-2-one (2)



1 (100 mol%) (400 mol%)

2 90% Yield 34:1 dr, 98% ee

Detailed Procedures

Catalyst (*R*)-IrLn[1] (0.551 g, 0.5 mmol, 5 mol%) and K_3PO_4 (0.109 g, 0.5 mmol, 5 mol%) were added to a flame-dried seal tube and purged with argon. Anhydrous THF (10 mL) was added, followed by 5-hydroxypentan-2-one (1.02 g, 10 mmol, 100 mol%) and isoprene monoxide (3.9 mL, 40 mmol, 400 mol%) via syringe. (**Caution:** K_3PO_4 is hygroscopic. Please make sure all the base solid is placed at the bottom of reaction vial and submerged by solvent) After sealed with cap, the resulting mixture was allowed to stir at 60 °C for 48 hours. The solution was cooled to ambient temperature and concentrated under reduced pressure. The residue was submitted to flash column chromatography on silica gel (pretreated with triethylamine, DCM/acetone = 10:1 to 5:1). *Cautions: product starts converting to ketal upon gently heating (>30 ˚C) under neutral or acidic condition!* The title compound was isolated as a brown oil (1.68 g, 9 mmol) in 90% yield. *Product exists as an equilibrating mixture between hydroxyl ketone and two diastereomeric lactols (equilibrated ratio 0.48:0.30:0.22).*

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.07* (dd, *J* = 17.8, 11.0 Hz, 1H (minor2)), 5.99 (dd, *J* = 17.7, 11.1 Hz, 1H, (major)), 5.93* (dd, *J* = 17.8, 11.0 Hz, 1H, (minor1)), 5.29-5.20 (m, 1H (major, minor1, minor2)), 5.18* (dd, *J* = 8.7, 1.5 Hz, 1H (minor1)), 5.14* (dd, *J* = 8.8, 1.5 Hz, 1H (minor2)), 5.13 (dd, *J* = 17.7, 1.4 Hz, 1H (major)), 4.20* (t, *J* = 7.0 Hz, 1H (minor1)), 4.00* (dd, *J* = 9.9, 5.7 Hz, 1H (minor2)), 3.69 (d, *J* = 10.8 Hz, 1H (major)), 3.61-3.49 (m, 2H (major, minor1, minor2)), 3.26 (br, 1H (major)), 2.99* (br, 2H (minor1, minor2)), 2.66 (td, *J* = 6.7, 4.0 Hz, 2H (major)), 2.18 (s, 3H (major)), 2.13-1.91 (m, 1H (major) + 2H (minor1, minor2)), 1.87-1.54 (m, 2H (major, minor1, minor2)), 1.52* (s, 3H (minor1, minor2)), 1.01 (s, 3H (major)), 0.99* (s, 3H (minor2)), 0.97* (s, 3H (minor1)).

^{13}C NMR (100 MHz, $CDCl_3$) δ 210.5 (major), 140.0 (major), 139.5* (minor2), 138.8* (minor1), 116.0* (minor1), 115.9* (minor2), 115.8 (major), 105.6* (minor1), 105.2* (minor2), 86.6* (minor2), 84.0* (minor1), 76.9 (major), 70.0 (major, minor1, minor2), 45.6 (major), 44.7* (minor1), 44.4* (minor2), 40.9 (major), 38.0* (minor2), 37.0* (minor1), 30.1 (major), 27.2* (minor2), 27.1* (minor1), 26.4* (minor2), 26.2* (minor1), 25.7 (major), 18.2* (minor2), 17.8 (major), 17.7* (minor1).

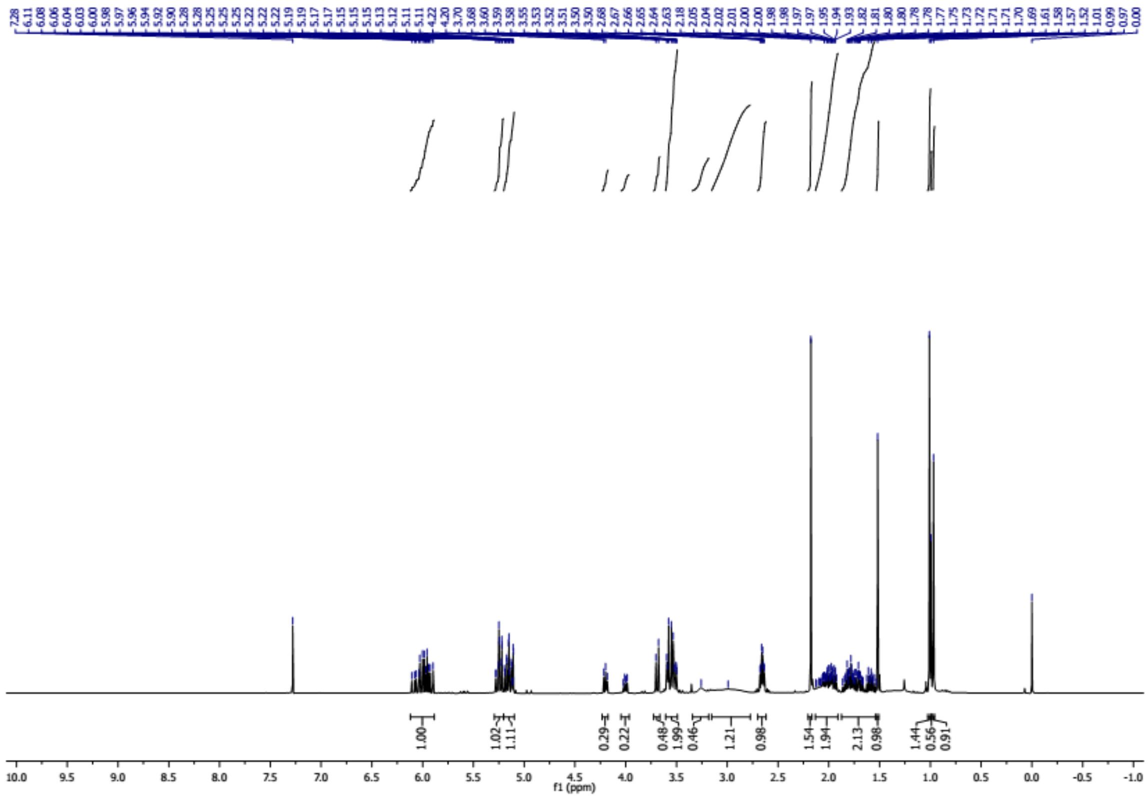
R_f 0.2 (DCM/acetone = 6:1, *p*-anisaldehyde)

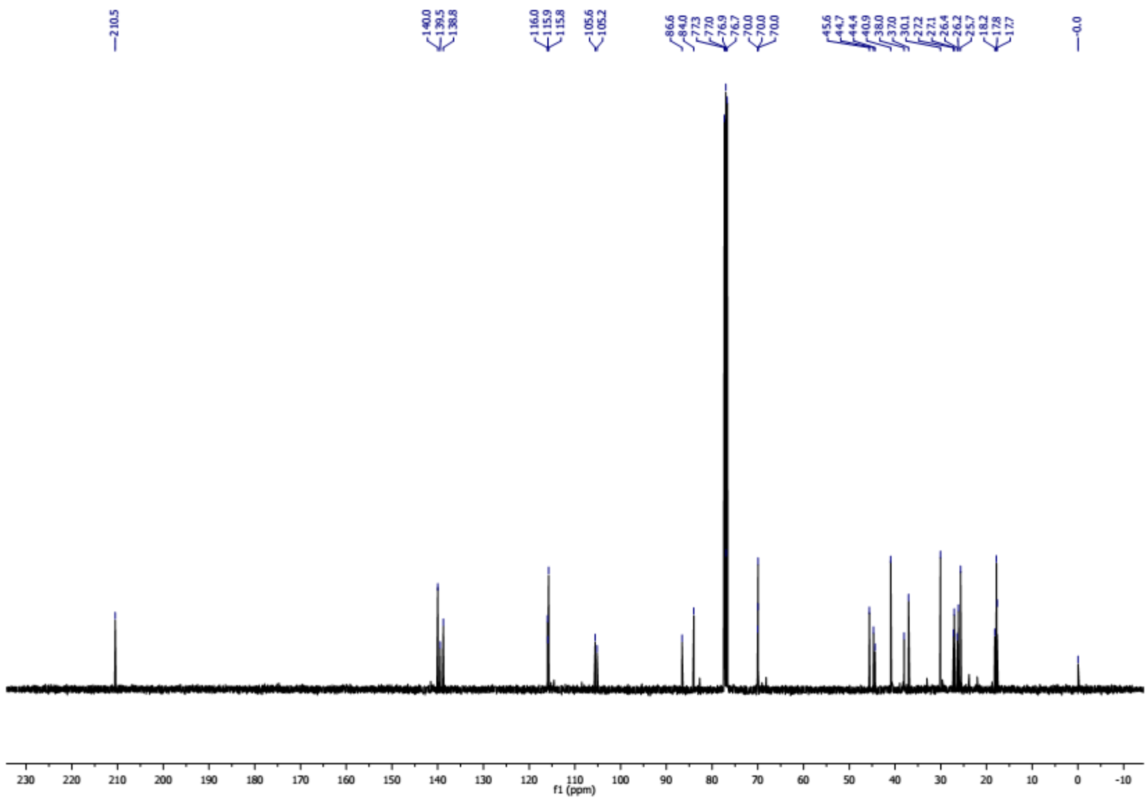
HRMS (ESI) Calcd. for $C_{10}H_{18}O_3$ $[M+Na]^+$: 209.1148, Found: 209.1149.

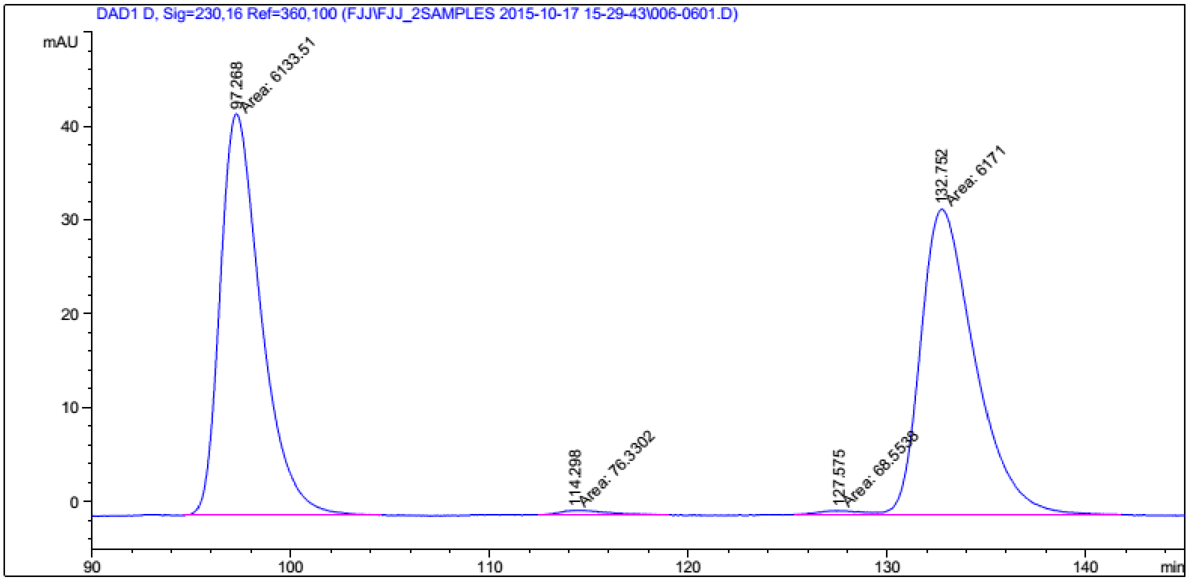
FTIR (neat): 3384, 2978, 2878, 1705, 1638, 1416, 1362, 1215, 1166, 1091, 1011, 917, 674 cm^{-1}.

Optical Rotation $[\alpha]_D^{30}$ = -4.5° (*c* = 1.0, $CHCl_3$)

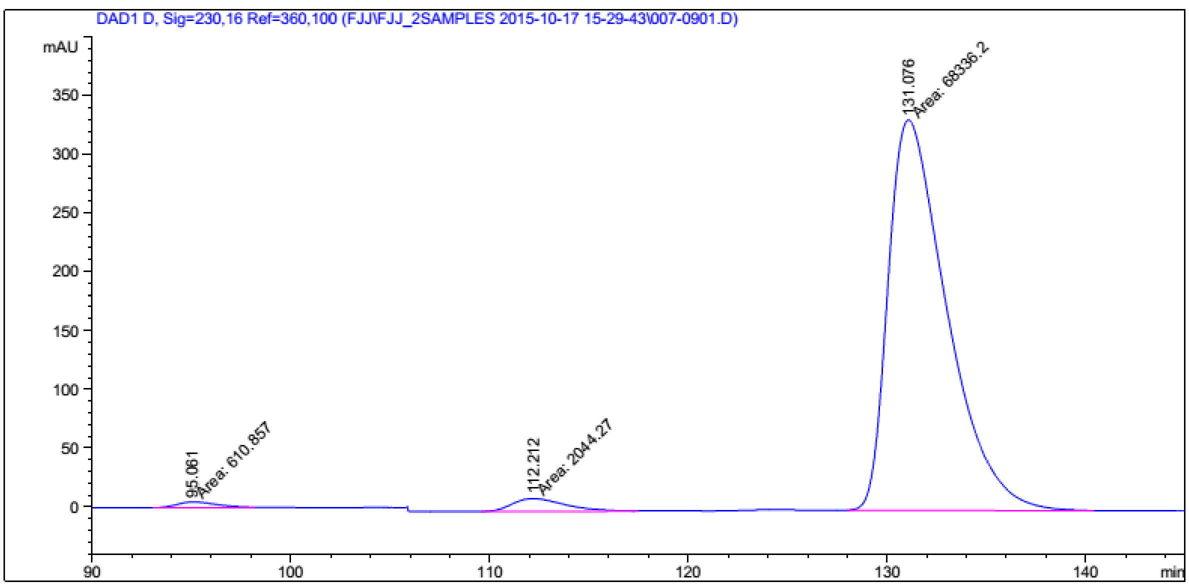
HPLC Diastereomeric ratio and enantiomeric excess was determined by HPLC analysis of the dibenzoate of product (Chiralcel AD-H column, hexanes/*i*-PrOH = 97:3, 0.50 mL/min, 230 nm), *anti*:*syn* = 34:1, *ee* = 98%.


1.00
1.02
1.11
0.29
0.22
0.48
1.99
0.46
1.21
0.98
1.54
1.94
2.13
0.98
1.44
0.56
0.91
f1 (ppm)


210.5
140.0
139.5
138.8
116.0
115.9
115.8
105.6
105.2
86.6
84.0
77.3
77.0
76.9
76.7
70.0
70.0
70.0
45.6
44.7
44.4
40.9
38.0
37.0
30.1
27.2
27.1
26.4
26.2
25.7
18.2
17.8
17.7
0.0
f1 (ppm)

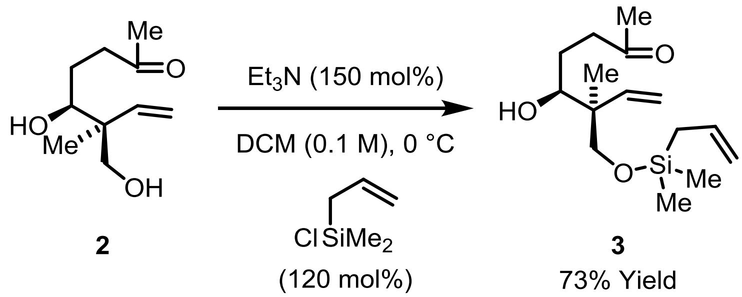


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	97.268	MM	2.3911	6133.50928	42.75188	49.2676
2	114.298	MM	2.6483	76.33024	4.80368e-1	0.6131
3	127.575	MF	2.6135	68.55382	4.37176e-1	0.5507
4	132.752	FM	3.1614	6170.99561	32.53297	49.5687



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	95.061	MM	2.2205	610.85699	4.58498	0.8605
2	112.212	MM	3.1221	2044.26855	10.91295	2.8796
3	131.076	MM	3.4238	6.83362e4	332.64838	96.2599

(5*S*,6*S*)-6-(((Allyldimethylsilyl)oxy)methyl)-5-hydroxy-6-methyloct-7-en-2-one (3)



Detailed Procedures

To a solution of **2** (1.32 g, 7.1 mmol, 100 mol%) in anhydrous DCM (71 mL), freshly distilled triethylamine (1.07 g, 10.6 mmol, 150 mol%) was added and the resulting mixture was cooled to 0 °C. Allyl(chloro)dimethylsilane (1.15 mL, 8.5 mmol, 120 mol%) was added dropwise via syringe. The mixture was allowed to stir for 1 hour at the same temperature and quenched by addition of saturated $NaHCO_3$ aqueous solution. The two layers were separated. The organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (DCM/acetone = 100:1). The title compound was obtained as a light yellow liquid (1.48 g, 5.2 mmol) in 73% yield. *Product exists as an equilibrating mixture between hydroxyl ketone and two diastereomeric lactols (equilibrated ratio 0.70:0.18:0.12).*

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.98 (dd, *J* = 17.8, 11.1 Hz, 1H (major)), 5.95* (dd, *J* = 17.8, 11.0 Hz, 1H (minor1)), 5.88-5.76* (m, 1H (minor2)), 5.79* (dd, *J* = 16.9, 10.1 Hz, 1H (minor2)), 5.77 (dd, *J* = 16.9, 10.1 Hz, 1H (major)), 5.75* (dd, *J* = 17.0, 10.1 Hz, 1H (minor1)), 5.21* (dd, *J* = 11.0, 1.6 Hz, 1H (minor1)), 5.17 (dd, *J* = 11.1, 1.4 Hz, 1H (major)), 5.16* (dd, *J* = 11.0, 1.6 Hz, 1H (minor2)), 5.12* (dd, *J* = 17.8, 1.6 Hz, 1H (minor1)), 5.09* (dd, *J* = 17.7, 1.7 Hz, 1H (minor2)), 5.07 (dd, *J* = 17.8, 1.3 Hz, 1H (major)), 4.96-4.82 (m, 2H (major, minor1, minor2)), 4.23-4.16* (m, 1H (minor2)), 4.05-3.96* (m, 1H (minor1)), 3.71 (d, *J* = 9.7 Hz, 1H (major)), 3.61* (d, *J* = 9.7 Hz, 1H (minor1)), 3.60* (d, *J* = 9.6 Hz, 1H (minor2)), 3.51 (d, *J* = 9.7 Hz, 1H (major)), 3.51-3.46 (m, 1H (major, minor2)), 3.48* (d, *J* = 9.6 Hz, 1H (minor1)), 3.43 (dd, *J* = 4.7, 1.0 Hz, 1H (major)), 3.38* (d, *J* = 9.6 Hz, 1H (minor2)), 2.72 (ddd, *J* = 17.7, 8.4, 5.7 Hz, 1H (major)), 2.58* (br, 1H (minor1)), 2.53 (ddd, *J* = 17.7, 8.3, 6.6 Hz, 1H (major)), 2.15 (s, 3H (major)), 2.11* (t, *J* = 0.9 Hz, 1H (minor2)), 2.07-1.88* (m, 2H (minor1, minor2)), 1.85-1.73 (m, 1H (major, minor1) + 2H (minor2)), 1.73-1.66* (m, 1H (minor1)), 1.65-1.60 (m, 2H (major, minor1, minor2)), 1.59-1.47 (m, 1H (major1)), 1.50 (s, 1H (minor2)), 1.49 (s, 1H (minor1)), 0.98 (s, 3H (major)), 0.97* (s, 3H (minor1)), 0.93* (s, 3H (minor2)), 0.13 (s, 6H (major)), 0.11* (s, 6H (minor1)), 0.10* (s, 6H (minor2)).

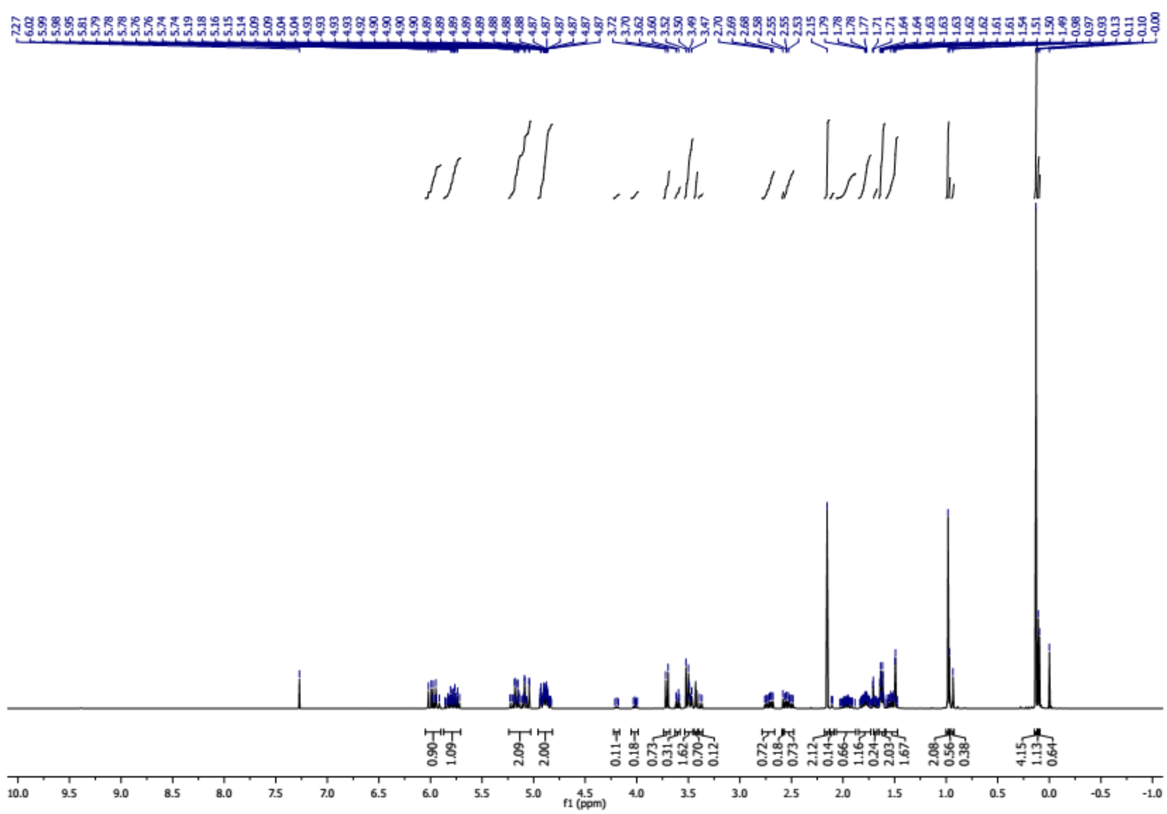
^{13}C NMR (100 MHz, $CDCl_3$) δ 209.6 (major), 140.2* (minor2), 140.1 (major), 140.1* (minor1), 134.3* (minor1), 134.1* (minor2), 133.4 (major), 115.5* (minor1), 115.3* (minor2), 114.9 (major), 114.1 (major), 113.6* (minor2), 113.4* (minor1), 105.2* (minor2), 104.7* (minor1), 83.6* (minor2), 81.2* (minor1), 77.2 (major), 70.8 (major), 68.2* (minor2), 68.0* (minor1), 45.3* (minor2), 44.7 (major), 44.7* (minor1), 40.6 (major), 38.7* (minor1), 37.4* (minor2), 30.1 (major), 27.1* (minor1), 26.9* (minor2), 25.9 (major), 25.8* (minor2), 25.7* (minor1), 24.4* (minor1), 24.3* (minor2), 24.0 (major), 18.5 (major), 17.8* (minor2), 16.95* (minor1), -2.5* (minor1), -2.6* (minor2), -2.8 (major).

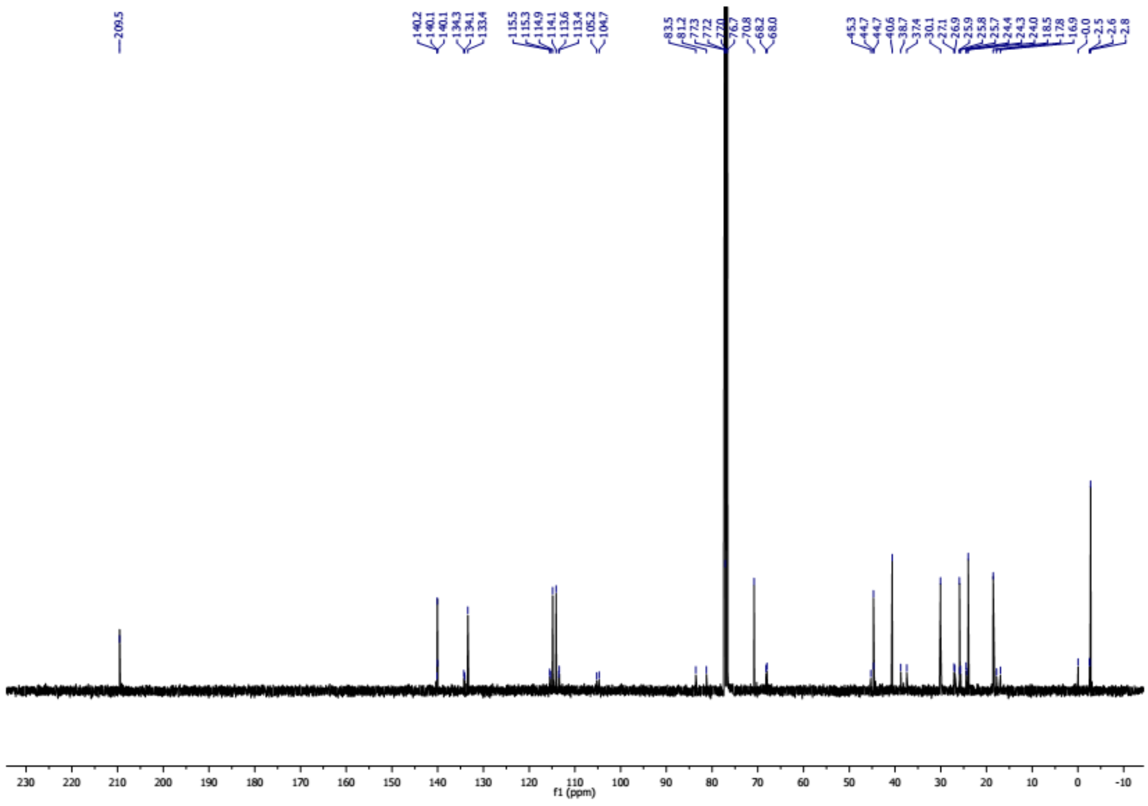
R_f 0.3 (hexanes/acetone = 6:1, *p*-anisaldehyde)

HRMS (ESI) Calcd. for $C_{15}H_{28}O_3Si$ $[M+Na]^+$: 307.1700, Found: 307.1704.

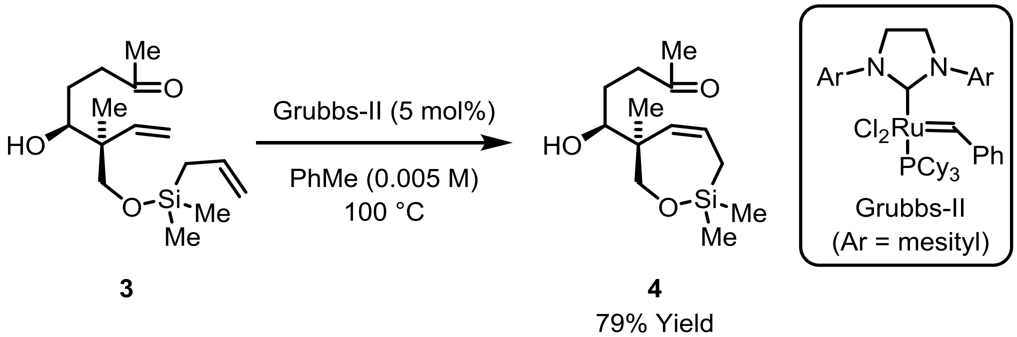
FTIR (neat): 3442, 2970, 1717, 1631, 1417, 1364, 1252, 1216, 1158, 1081, 893, 859, 834, 751 cm^{-1}.

Optical Rotation $[\alpha]_D^{30}$ = -8.0° (*c* = 1.0, $CHCl_3$)


f1 (ppm)


f1 (ppm)

(*S*)-5-Hydroxy-5-((*S*)-2,2,6-trimethyl-2,3,6,7-tetrahydro-1,2-oxasilepin-6-yl)pentan-2-one (4)



Detailed Procedures

A solution of **3** (0.515 g, 1.8 mmol, 100 mol%) in toluene (350 mL) was degassed by freeze-pump-thaw cycle for three times. It was heated to 100 ˚C, and a freshly made solution of Grubbs-II catalyst (0.0768 g, 0.09 mmol, 5 mol%) in toluene (8 mL) was added via syringe pump in a period of 2 hours. The mixture was allowed to stir at the same temperature for 3 hours. It was cooled to ambient temperature and further cooled in an ice bath, when DMSO (0.32 mL, 4.5 mmol, 250 mol%) was added. After stirred for 12 hours, the solvent was removed under reduced pressure. The residue was submitted to flash column chromatography on silica gel (pretreated with triethylamine, hexanes/acetone = 15:1). The title compound was obtained as a brown oil (0.358 g, 1.4 mmol) in 79% yield, which solidified upon standing in a -20 ˚C freezer. *Product exists as an equilibrating mixture between hydroxyl ketone and two diastereomeric lactols (equilibrated ratio 0.66:0.18:0.16).*

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.78 (dt, *J* = 11.9, 7.3 Hz, 1H (major)), 5.73* (dt, *J* = 11.7, 7.2 Hz, 1H (minor2)), 5.66* (ddd, *J* = 11.8, 7.8, 6.8 Hz, 1H (minor1)), 5.22 (d, *J* = 11.9 Hz, 1H (major)), 5.17* (d, *J* = 12.0 Hz, 1H (minor2)), 5.15* (d, *J* = 11.8 Hz, 1H (minor1)), 4.23* (dd, *J* = 11.9, 0.8 Hz, 1H (minor1)), 4.08* (d, *J* = 11.7 Hz, 1H (minor2)), 4.05* (dd, *J* = 8.1, 5.6 Hz, 1H (minor1)), 4.00 (dd, *J* = 11.9, 1.0 Hz, 1H (major)), 3.89* (dd, *J* = 10.2, 6.4 Hz, 1H (minor2)), 3.82 (d, *J* = 11.9 Hz, 1H (major)), 3.65* (dd, *J* = 11.9, 0.7 Hz, 1H (minor1)), 3.58* (dd, *J* = 11.7, 1.0 Hz, 1H (minor2)), 3.40 (ddd, *J* = 11.1, 4.7, 2.2 Hz, 1H (major)), 3.32* (br, 1H (minor1)), 2.97 (dd, *J* = 4.7, 1.0 Hz, 1H (major)), 2.71 (ddd, *J* = 17.8, 7.9, 6.0 Hz, 1H (major)), 2.55 (dt, *J* = 17.9, 7.1 Hz, 1H (major)), 2.25-2.14* (m, 1H (minor1)), 2.16 (s, 3H (major)), 2.13-2.03* (m, 1H (minor2)), 2.01-1.74 (m, 1H (major) + 3H (minor1, minor2)), 1.70-1.56 (m, 3H (major) + 2H (minor1, minor2)), 1.54-1.44* (m, 1H (minor2)), 1.51* (s, 3H (minor2)), 1.50* (s, 3H (minor1)), 1.00* (s, 3H (minor1)), 0.99* (s, 3H (minor2)), 0.92 (s, 3H (major)), 0.16* (s, 3H (minor1)), 0.15 (s, 3H (major)), 0.14 (s, 3H (major)), 0.14* (s, 3H (minor1)), 0.13* (s, 3H (minor2)), 0.13* (s, 3H (minor2)).

^{13}C NMR (100 MHz, $CDCl_3$) δ 209.7 (major), 133.0* (minor1), 132.6* (minor2), 131.6 (major), 125.3 (major), 124.1* (minor2), 123.8* (minor1), 105.4* (minor1), 104.9* (minor2), 86.0* (minor2), 84.1* (minor1), 79.0 (major), 70.0 (major), 67.4* (minor1), 67.4* (minor2), 48.0 (major), 47.0* (minor1), 46.9* (minor2), 40.9 (major), 38.9* (minor1), 37.4* (minor2), 30.1 (major), 27.1* (minor1), 26.5* (minor2), 26.1* (minor1), 25.5 (major, minor2), 22.4* (minor2), 21.1 (major), 20.9* (minor1), 16.1* (minor1), 16.0

(major), 16.0* (minor2), -1.1* (minor1), -1.2* (minor1), -1.2* (minor2), -1.3 (major), -1.4 (major, minor2).

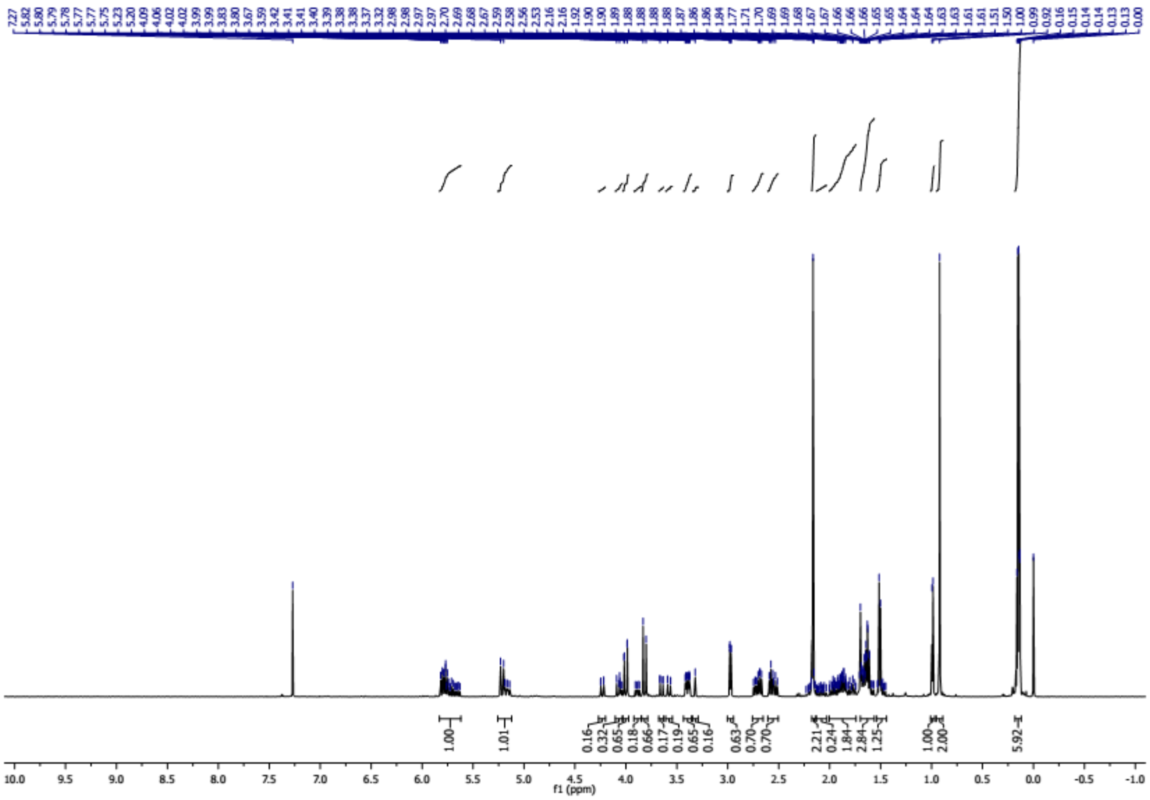
R_f 0.1 (DCM/acetone = 50:1, *p*-anisaldehyde)

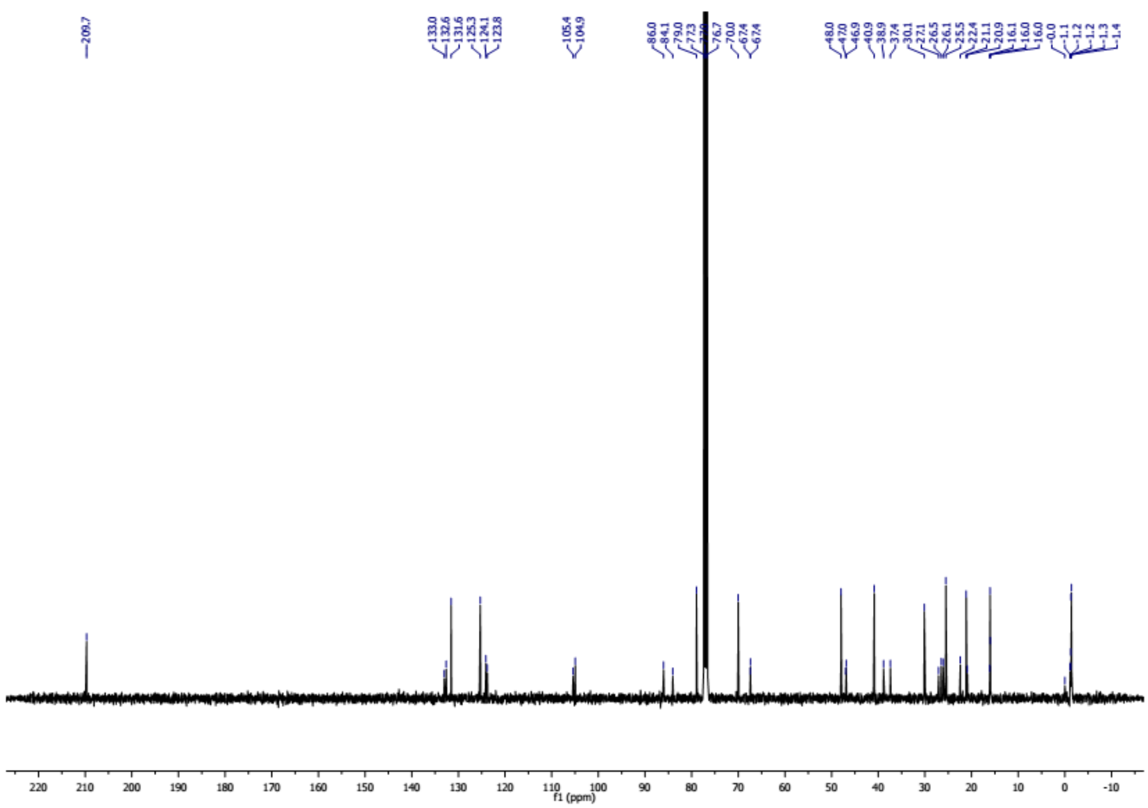
HRMS (ESI) Calcd. for $C_{13}H_{24}O_3Si$ $[M+Na]^+$: 279.1387, Found: 279.1386.

FTIR (neat): 3419, 2957, 2879, 1715, 1406, 1375, 1250, 1084, 859, 839, 809, 752, 730, 678 cm^{-1}.

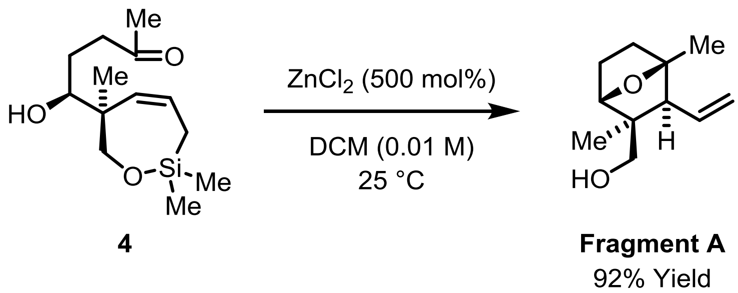
MP 55.2-55.5 ˚C (CH_2Cl_2)

Optical Rotation $[\alpha]_D^{30}$ = +13.6˚ (c = 1.3, $CHCl_3$)


f1 (ppm)


f1 (ppm)

((1*S*,2*S*,3*S*,4*R*)-2,4-Dimethyl-3-vinyl-7-oxabicyclo[2.2.1]heptan-2-yl)methanol (Fragment A)



Detailed Procedures

To a solution of **4** (0.128 g, 0.5 mmol, 100 mol%) in DCM (50 mL) at -78 ˚C, $ZnCl_2$ (1M solution in Et_2O, 2.5 mL, 2.5 mmol, 500 mol%) was added. The resulting mixture was allowed to warm to ambient temperature, and stirred for 2 hours. The reaction was quenched by addition of saturated $NaHCO_3$ (aq., 50 mL), and stirred for 10 min. The mixture was filtered through Celite, and the precipitate was washed with DCM. The filtrate was separated, and the aqueous layer was extracted with DCM (50 mL). The combined organic layers were dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/acetone = 15:1). The title compound was obtained as a light yellow oil (0.0843 g, 0.46 mmol) in 92% yield, which solidified upon standing in freezer.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.91 (ddd, *J* = 16.9, 10.8, 10.0 Hz, 1H), 5.11 (dd, *J* = 10.0, 2.3 Hz, 1H), 5.02 (ddd, *J* = 16.9, 2.3, 0.6 Hz, 1H), 4.23 (d, *J* = 5.4 Hz, 1H), 3.54-3.42 (m, 2H), 2.75 (dd, *J* = 6.6, 3.6 Hz, 1H), 2.02-1.92 (m, 2H), 1.80 (tt, *J* = 12.5, 5.3 Hz, 1H), 1.66-1.50 (m, 2H), 1.28 (s, 3H), 1.04 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 136.3, 117.8, 87.0, 82.5, 68.3, 63.1, 50.8, 37.7, 26.2, 20.3, 18.7.

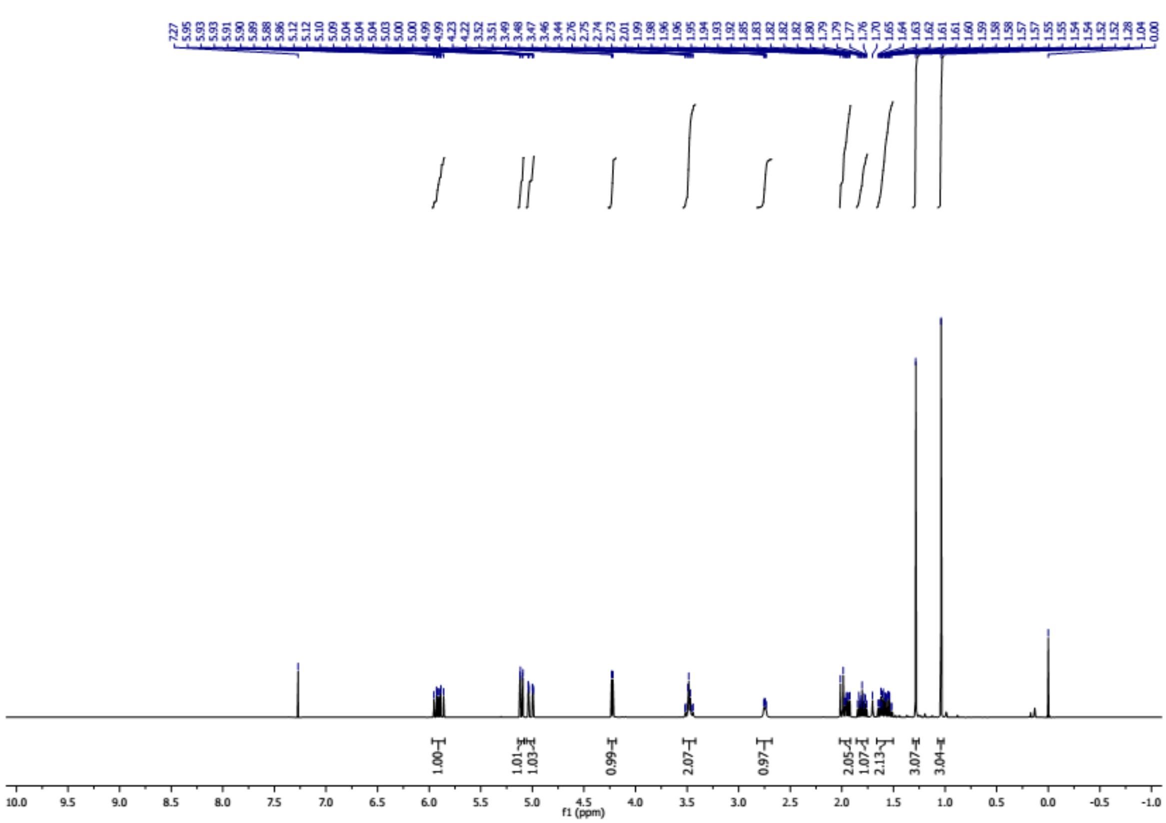
R_f 0.3 (hexanes/EA = 3:1, *p*-anisaldehyde)

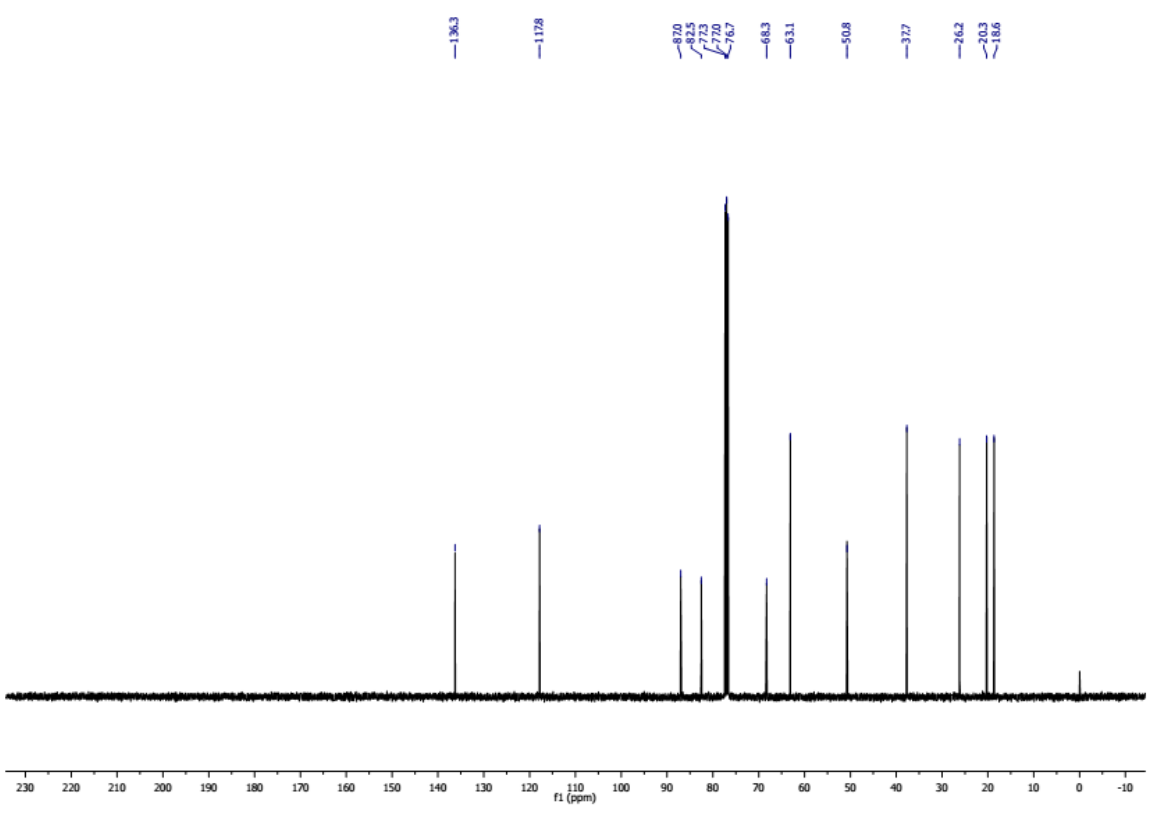
HRMS (CI) Calcd. for $C_{11}H_{18}O_2$ $[M+H]^+$: 183.1380, Found: 183.1383.

FTIR (neat): 3375, 2975, 2914, 1633, 1381, 1187, 1073, 1020, 1015, 1001, 991, 914, 908, 873, 826, 804, 682 cm^{-1}.

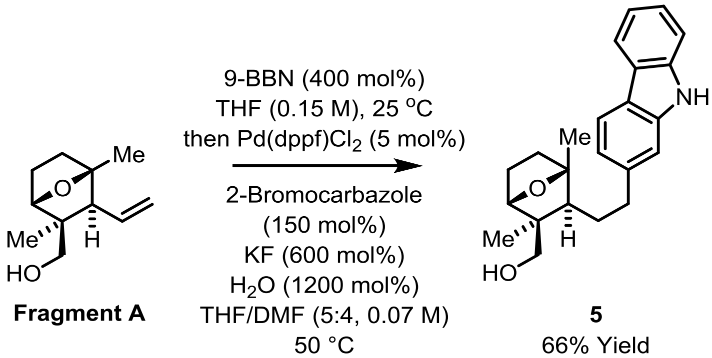
MP 60.9-61.5 ˚C (CH_2Cl_2)

Optical Rotation $[\alpha]_D^{30}$ = +40.0˚ (*c* = 1.0, $CHCl_3$)





((1*S*,2*S*,3*S*,4*R*)-3-(2-(9*H*-Carbazol-2-yl)ethyl)-2,4-dimethyl-7-oxabicyclo[2.2.1]heptan-2-yl)methanol (5)



Detailed Procedures

To a solution of alkene (0.150 g, 0.82 mmol, 100 mol%) in THF (0.8 mL), a solution of 9-BBN (0.403 g, 3.3 mmol, 400 mol%) in THF (4.4 mL) was added slowly at ambient temperature. The mixture was stirred at the same temperature until TLC indicated all the starting alkene was consumed (about an hour). The resulting clear solution was mixed with anhydrous KF (0.286 g, 4.9 mmol, 600 mol%), Pd(dppf)Cl_2 (0.0300 g, 0.041 mmol, 5 mol%) and 2-bromocarbazole (0.303 g, 1.23 mmol, 150 mol%), followed by addition of DMF (4 mL) and water (0.18 mL, 9.8 mmol, 1200 mol%). The degassed heterogeneous mixture was heated to 50 ˚C and vigorously stirred overnight. After cooled to ambient temperature, the reaction mixture was diluted with ethyl acetate (5 mL) and water (5 mL). The separated organic layer was washed with 1 M NaOH (3 mL × 2) and brine (3 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/acetone = 10:1 to 5:1). The title compound was obtained as a pale yellow solid (0.191 g, 0.55 mmol) in 66% yield.

^{1}H NMR (400 MHz, CD_3OD) δ 7.98 (dt, *J* = 7.8, 1.1 Hz, 1H), 7.93 (dd, *J* = 8.0, 0.7 Hz, 1H), 7.40 (dt, *J* = 8.1, 1.0 Hz, 1H), 7.31 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.25 (dd, *J* = 1.5, 0.7 Hz, 1H), 7.11 (ddd, *J* = 8.0, 7.1, 1.1 Hz, 1H), 6.99 (dd, *J* = 8.0, 1.5 Hz, 1H), 4.26 (d, *J* = 5.4 Hz, 1H), 3.72 (d, *J* = 10.3 Hz, 1H), 3.54 (d, *J* = 10.4 Hz, 1H), 2.86-2.70 (m, 2H), 1.94 (ddd, *J* = 12.5, 9.1, 4.6 Hz, 1H), 1.79-1.58 (m, 4H), 1.55-1.45 (m, 2H), 1.39 (s, 3H), 1.16 (s, 3H).

^{13}C NMR (100 MHz, CD_3OD) δ 141.9, 141.6, 141.4, 126.1, 124.4, 122.5, 120.8, 120.6, 120.4, 119.6, 111.6, 111.1, 88.4, 82.1, 66.3, 56.7, 50.8, 40.1, 38.0, 29.7, 26.6, 21.5, 19.1.

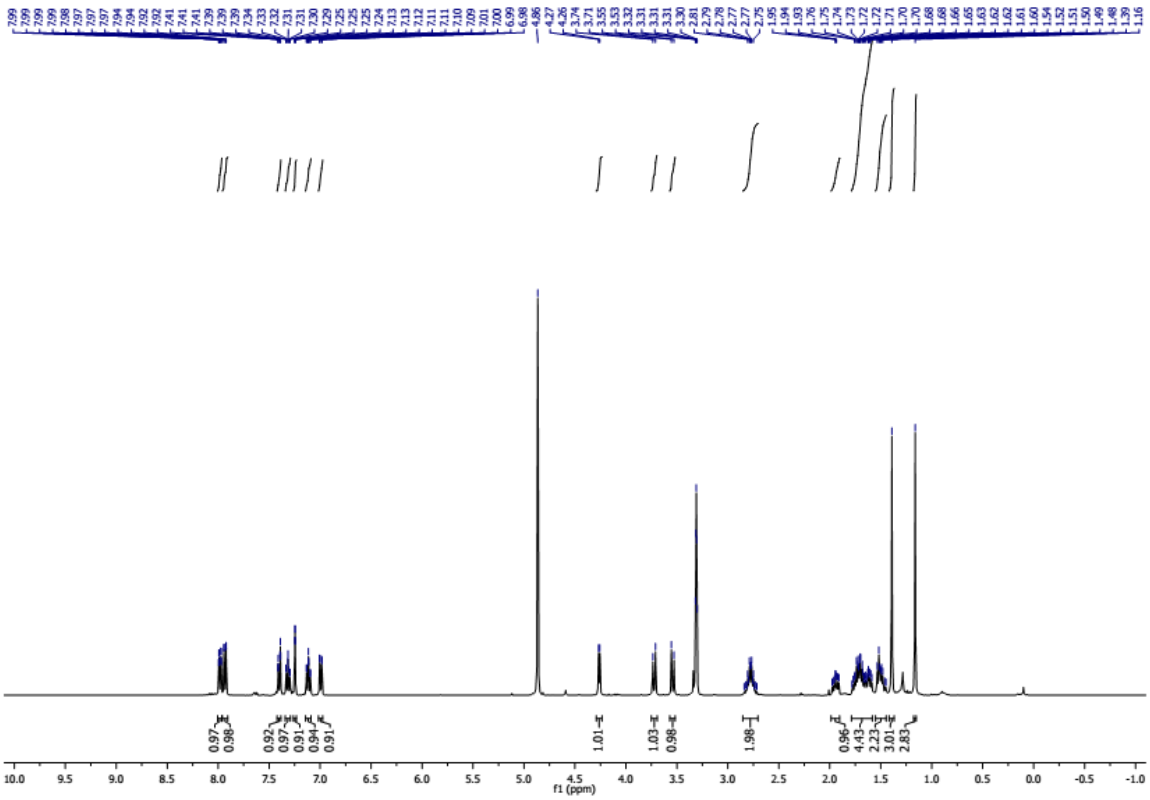
R_f 0.1 (hexanes/acetone = 5:1, UV/*p*-anisaldehyde)

HRMS (CI) Calcd. for $C_{23}H_{27}NO_2$ $[M]^+$: 349.2042, Found: 349.2036.

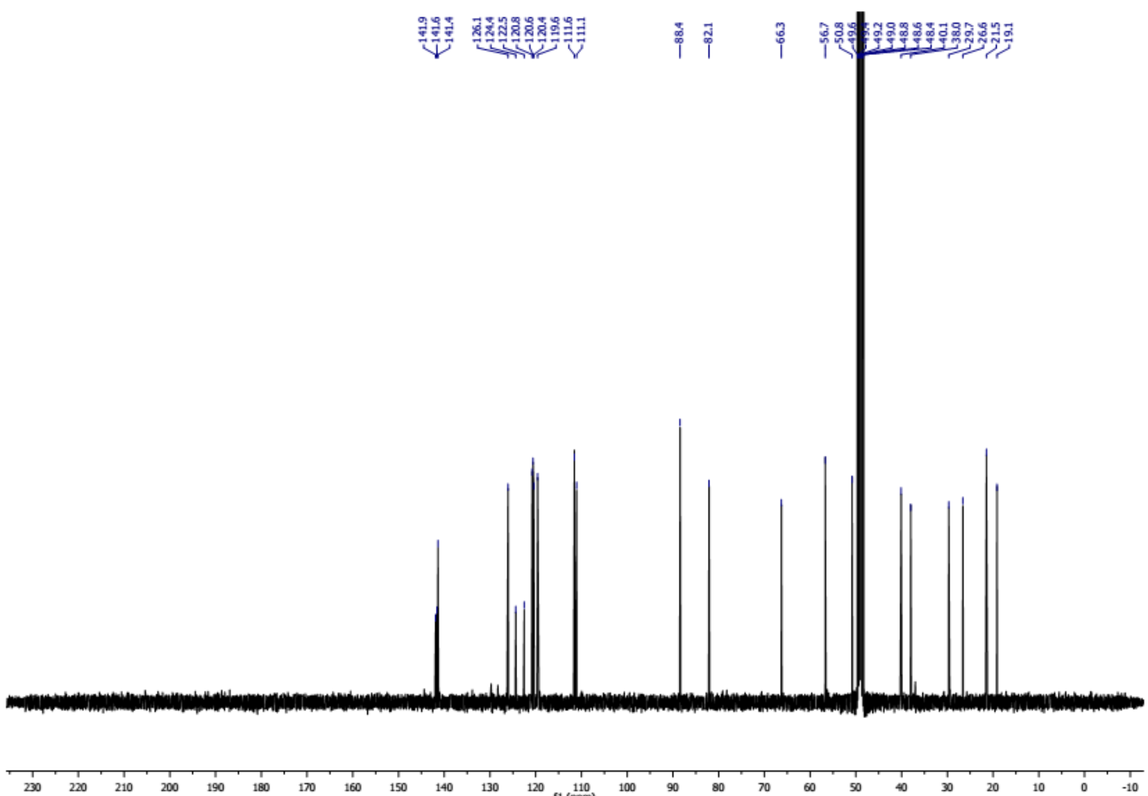
FTIR (neat): 3462, 3416, 2955, 2872, 1608, 1460, 1438, 1380, 1327, 1241, 1040, 1007, 984, 976, 828, 744, 726 cm^{-1}.

MP 215.4-217.0 ˚C ($CHCl_3$)

Optical Rotation $[\alpha]_D^{30}$ = +33.3˚ (*c* = 0.1, CH_3OH)

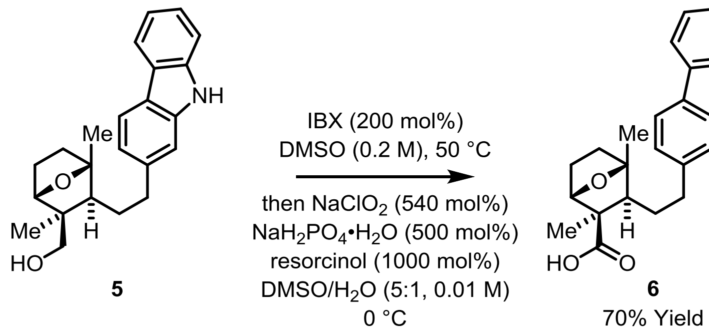


7.99 7.99 7.99 7.98 7.97 7.97 7.94 7.94 7.92 7.92 7.41 7.41 7.39 7.39 7.39 7.34 7.33 7.32 7.31 7.31 7.30 7.29 7.25 7.25 7.24 7.13 7.13 7.12 7.11 7.10 7.09 7.01 7.00 6.99 6.98 4.86 4.27 4.26 3.74 3.71 3.55 3.53 3.32 3.31 3.31 3.30 2.81 2.79 2.78 2.77 2.75 1.95 1.94 1.93 1.76 1.75 1.74 1.73 1.72 1.72 1.71 1.70 1.70 1.68 1.66 1.65 1.63 1.62 1.62 1.61 1.60 1.54 1.52 1.51 1.50 1.49 1.48 1.39 1.16
0.97
0.98
0.92
0.97
0.91
0.94
0.91
1.01
1.03
0.98
1.98
0.96
4.43
2.23
3.01
2.83
f1 (ppm)



141.9 141.6 141.4
126.1 124.4 122.5 120.8 120.6 120.4 119.6 111.6 111.1
88.4
82.1
66.3
56.7 50.8 49.6 49.4 49.2 49.0 48.8 48.6 48.4 40.1 38.0 29.7 26.6 21.5 19.1
f1 (ppm)

(1*S*,2*R*,3*S*,4*R*)-3-(2-(9H-Carbazol-2-yl)ethyl)-2,4-dimethyl-7-oxabicyclo[2.2.1]heptane-2-carboxylic acid (6)



Detailed Procedures

A suspension of alcohol **5** (0.0175 g, 0.05 mmol, 100 mol%) and IBX (0.0280 g, 0.1 mmol, 200 mol%) in DMSO (0.25 mL) was heated to 50 ˚C. The mixture became homogeneous eventually and was stirred at the same temperature for 2 hours. After cooled to ambient temperature, a solution of resorcinol (0.0551 g, 0.5 mmol, 1000 mol%) in DMSO (3.5 mL) was added, followed by dropwise addition of an ice-cooled solution of $NaClO_2$ (80 wt% pure, 0.0305 g, 0.27 mmol, 540 mol%) and $NaH_2PO_4{\cdot}H_2O$ (0.0345 g, 0.25 mmol, 500 mol%) in water (0.75 mL). The resulting mixture was allowed to stir at 0 ˚C for 30 min until all the aldehyde intermediate was consumed. The reaction was quenched by addition of saturated NH_4Cl (aq., 5 mL), and extracted with ethyl acetate (5 mL × 2). The combined organic layers were washed with water (5 mL × 2), and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/acetone/AcOH = 100:20:0.05). The product-containing fractions were concentrated and repurified by column chromatography on silica gel (hexanes/EA/AcOH = 100:30:0.05). The title compound was obtained as a white solid (0.0126 g, 0.035 mmol) in 70% yield.

^{1}H NMR (400 MHz, CD_3OD) δ 7.97 (dt, *J* = 7.8, 1.0 Hz, 1H), 7.91 (dd, *J* = 8.0, 0.6 Hz, 1H), 7.39 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.30 (ddd, *J* = 8.2, 7.1, 1.2 Hz, 1H), 7.23 (dd, *J* = 1.5, 0.7 Hz, 1H), 7.10 (ddd, *J* = 8.0, 7.1, 1.1 Hz, 1H), 6.98 (dd, *J* = 8.0, 1.5 Hz, 1H), 4.46 (d, *J* = 5.2 Hz, 1H), 2.85 (ddd, *J* = 13.5, 10.3, 5.6 Hz, 1H), 2.71 (ddd, *J* = 13.5, 10.0, 6.0 Hz, 1H), 2.00 (ddd, *J* = 12.9, 9.0, 4.0 Hz, 1H), 1.94-1.83 (m, 1H), 1.83-1.63 (m, 4H), 1.56 (td, *J* = 12.1, 4.0 Hz, 1H), 1.44 (s, 3H), 1.36 (s, 3H).

^{13}C NMR (100 MHz, CD_3OD) δ 177.0, 140.5, 140.1, 139.9, 124.6, 122.9, 121.0, 119.3, 119.2, 119.1, 118.1, 110.1, 109.7, 86.5, 82.6, 57.8, 56.8, 37.7, 35.1, 31.7, 26.2, 20.8, 17.6.

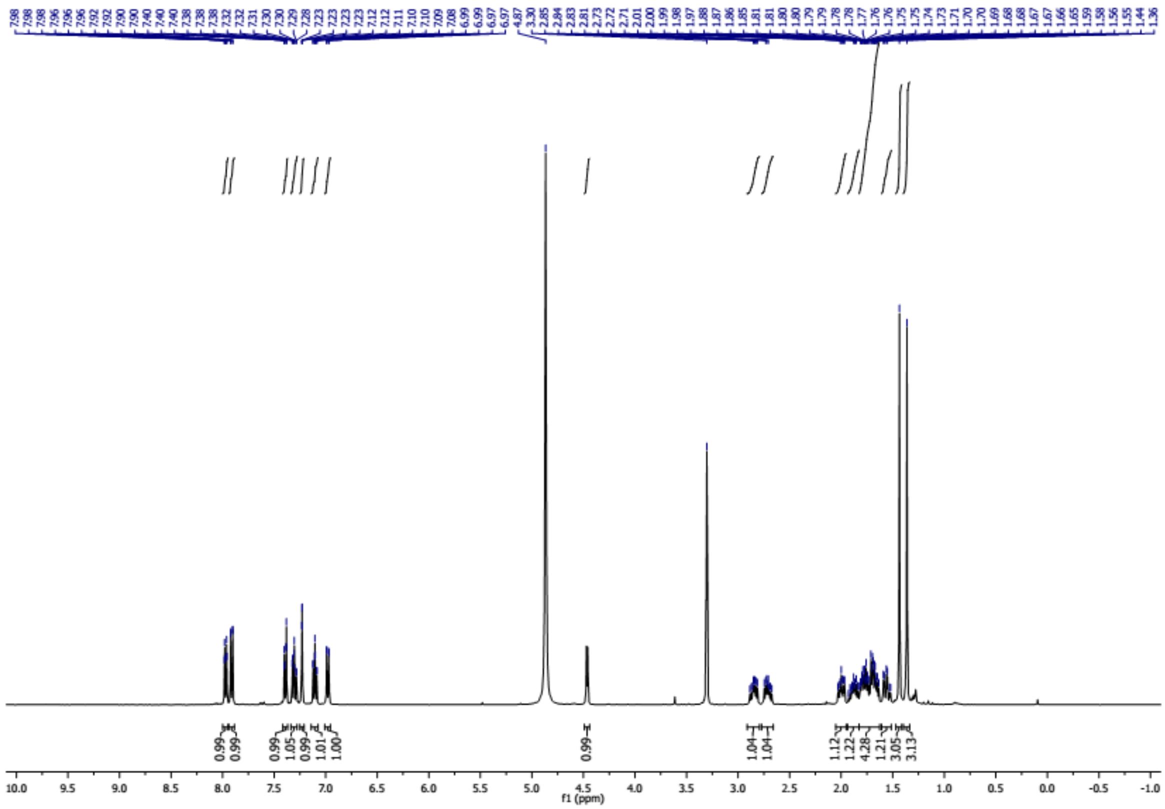
R_f 0.15 (hexanes/acetone/AcOH = 100:40:0.05, UV/*p*-anisaldehyde)

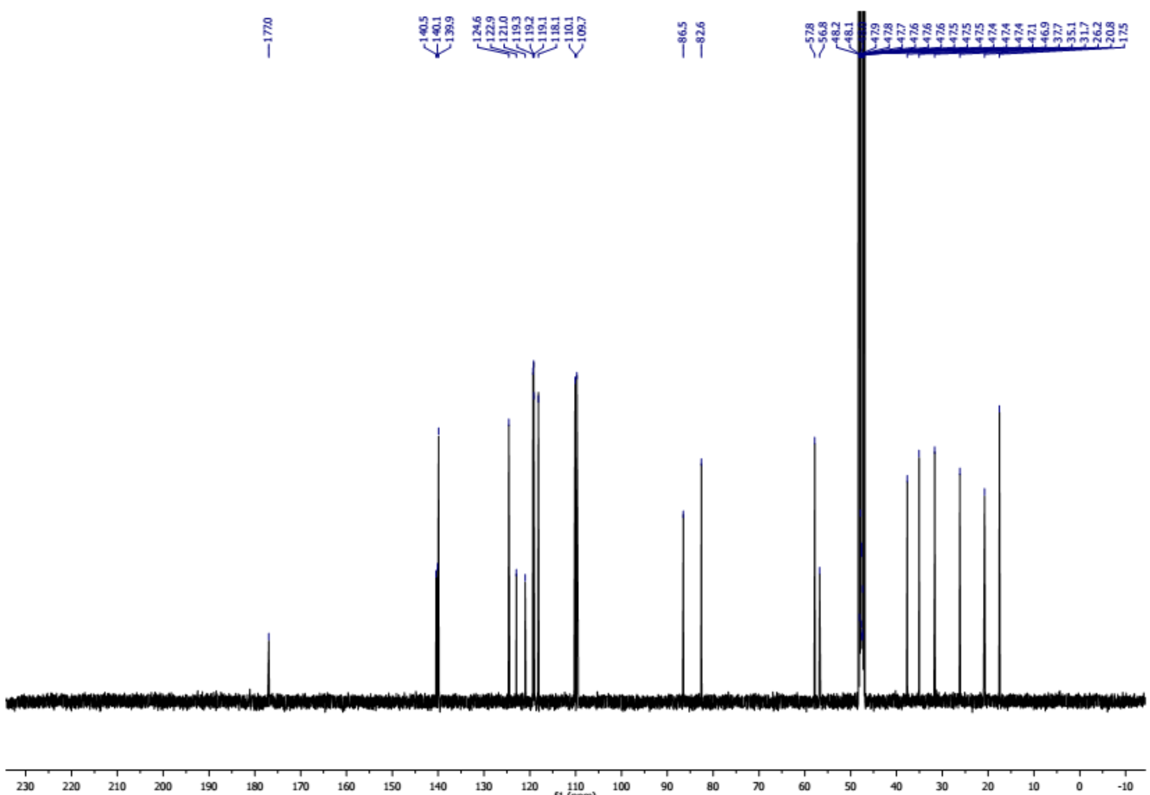
HRMS (ESI) Calcd. for $C_{23}H_{25}NO_3$ $[M+Na]^+$: 386.1727, Found: 386.1732.

FTIR (neat): 3357, 2933, 2871, 2496, 1731, 1607, 1460, 1441, 1401, 1383, 1326, 1240, 1109, 1001, 976, 865, 819, 769, 750, 731 cm^{-1}.

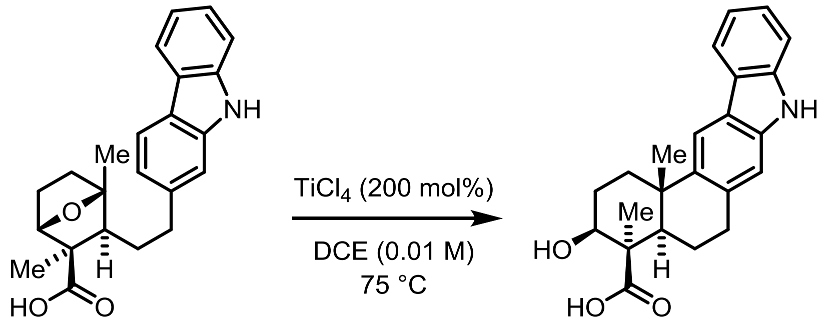
MP 194.3-196.4 ˚C (CH_3OH)

Optical Rotation $[\alpha]_D^{30}$ = +5.4˚ (*c* = 0.6, CH_3OH)





(3*S*,4*R*,4a*R*,13b*S*)-3-Hydroxy-4,13b-dimethyl-2,3,4,4a,5,6,8,13b-octahydro-1H-naphtho[2,1-b]carbazole-4-carboxylic acid ((+)-Oridamycin A)



6

Oridamycin A
81% Yield, 3:1 rr

Detailed Procedures

To a solution of acid **6** (0.0109 g, 0.03 mmol, 100 mol%) in DCE (2.8 mL), a solution of $TiCl_4$ in DCE (0.25 M, freshly prepared, 0.24 mL, 0.06 mmol, 200 mol%) was added dropwise. The mixture was heated to 75 ˚C and allowed to stir for 15 hours. After cooled to ambient temperature, the reaction was poured into saturated $NaHCO_3$ (aq., 20 mL) and allowed to stir for 15 min. Solid $KHSO_4$ was added until the pH was adjusted to 2. The mixture was extracted by DCM (10 mL × 2). The combined organic layers were washed with brine (10 mL), and dried over $MgSO_4$. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (DCM/MeOH/AcOH = 100:1:0 to 100:1:0.05). The title compound was obtained as a pale yellow solid (0.0068 g, 0.019 mmol) along with its regioisomer (0.0023 g, 0.006 mmol) in 81% combined yield and 3:1 regioselectivity.

^{1}H NMR (500 MHz, CD_3OD) δ 7.96 (dt, *J* = 8.1, 1.0 Hz, 1H), 7.96 (s, 1H), 7.34 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.28 (ddd, *J* = 8.1, 7.1, 1.2 Hz, 1H), 7.08 (ddd, *J* = 8.1, 7.1, 1.2 Hz, 1H), 7.06 (s, 1H), 3.25 (dd, *J* = 12.2, 4.4 Hz, 1H), 3.08 (ddd, *J* = 17.2, 5.6, 1.9 Hz, 1H), 2.97 (dddd, *J* = 16.5, 12.7, 6.2, 1.3 Hz, 1H), 2.60 (dt, *J* = 13.2, 3.7 Hz, 1H), 2.32 (qd, *J* = 13.3, 3.8 Hz, 1H), 2.28-2.19 (m, 1H), 2.20-2.07 (m, 1H), 1.93 (dq, *J* = 13.1, 3.9 Hz, 1H), 1.61 (td, *J* = 13.5, 3.9 Hz, 1H), 1.52 (dd, *J* = 12.2, 2.1 Hz, 1H), 1.50 (s, 3H), 1.28 (s, 3H).

^{13}C NMR (125 MHz, CD_3OD) δ 181.0, 142.1, 140.4, 140.1, 134.5, 126.1, 124.6, 123.3, 120.6, 119.3, 117.5, 111.4, 110.7, 79.1, 54.1, 49.8, 40.0, 39.6, 34.0, 30.3, 24.8, 24.6, 22.5.

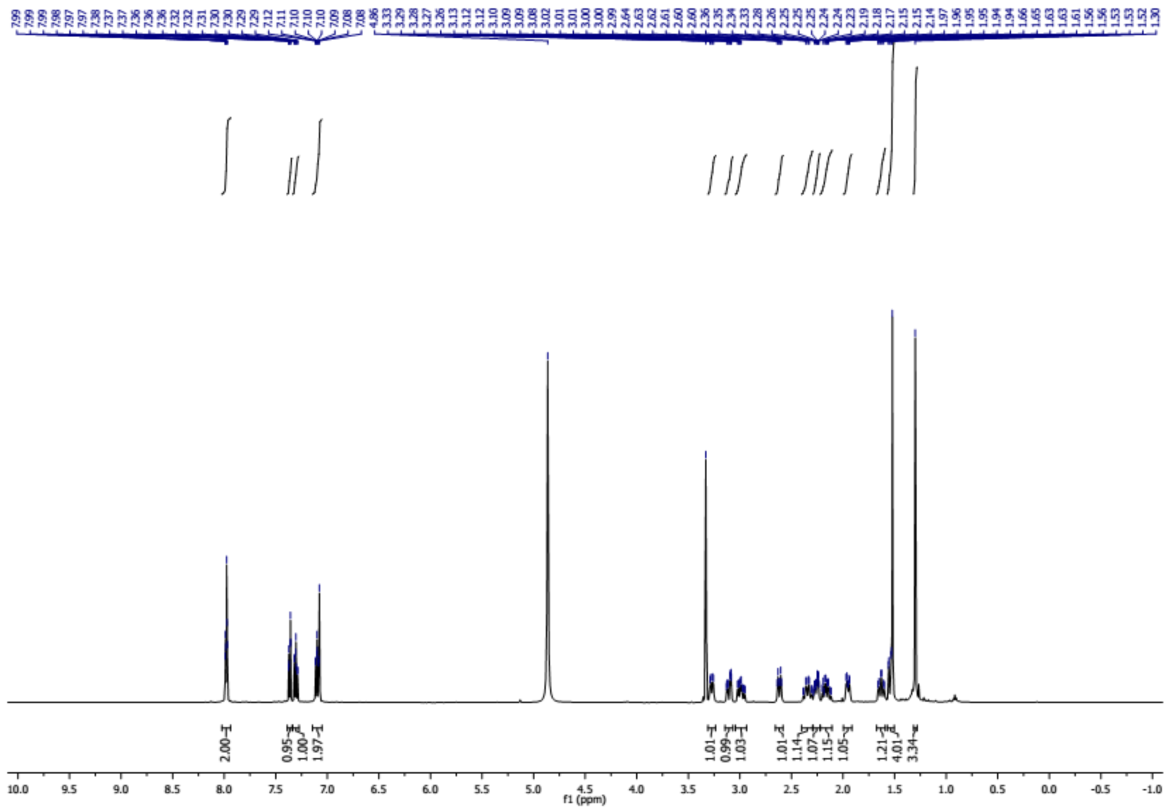
R_f 0.25 (DCM/MeOH = 50:1 (twice), UV/*p*-anisaldehyde)

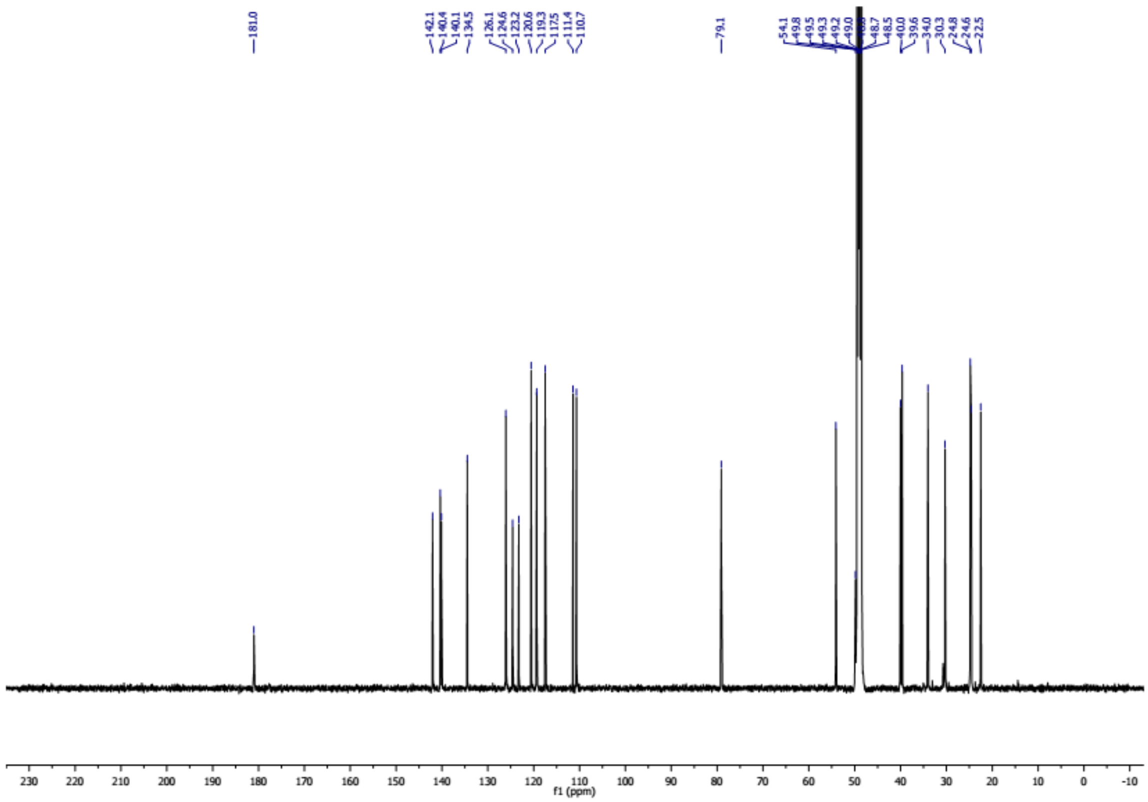
HRMS (ESI) Calcd. for $C_{23}H_{25}NO_3$ $[M+Na]^+$: 386.1727, Found: 386.1725.

FTIR (neat): 3406, 2918, 2851, 1702, 1612, 1466, 1439, 1319, 1240, 1186, 1088, 1071, 1025, 889, 859, 749, 736 cm^{-1}.

MP 183 ˚C (decomp.)

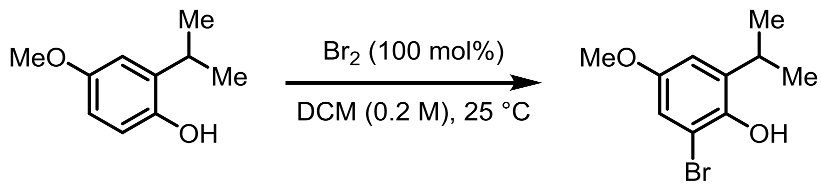
Optical Rotation $[\alpha]_D^{30}$ = +93.3° (c = 0.2, CH_3OH)





Detailed Procedures and Spectral Data for Asymmetric Synthesis of Triptoquinone C

2-Bromo-6-isopropyl-4-methoxyphenol (Fragment B-II)



S1
(100 mol%)

Fragment B-II
67% Yield

Detailed Procedures

To a solution of phenol **S1**[2] (3.11 g, 18.7 mmol, 100 mol%) in DCM (73.5 mL) at 0 ˚C, a solution of Br_2 (0.96 mL, 18.7 mmol, 100 mol%) in DCM (20 mL) was added dropwise in a period of 10 min. The resulting mixture was allowed to warm to ambient temperature and stirred overnight. The reaction was quenched by addition of saturated $NaHCO_3$ (aq., 100 mL), and kept stirring until the solution turned yellow (about 10 min). The organic layer was separated, washed with brine (100 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/EA = 50:1). The title compound was obtained as a yellow to red-brown oil (3.08 g, 12.6 mmol) in 67% yield.

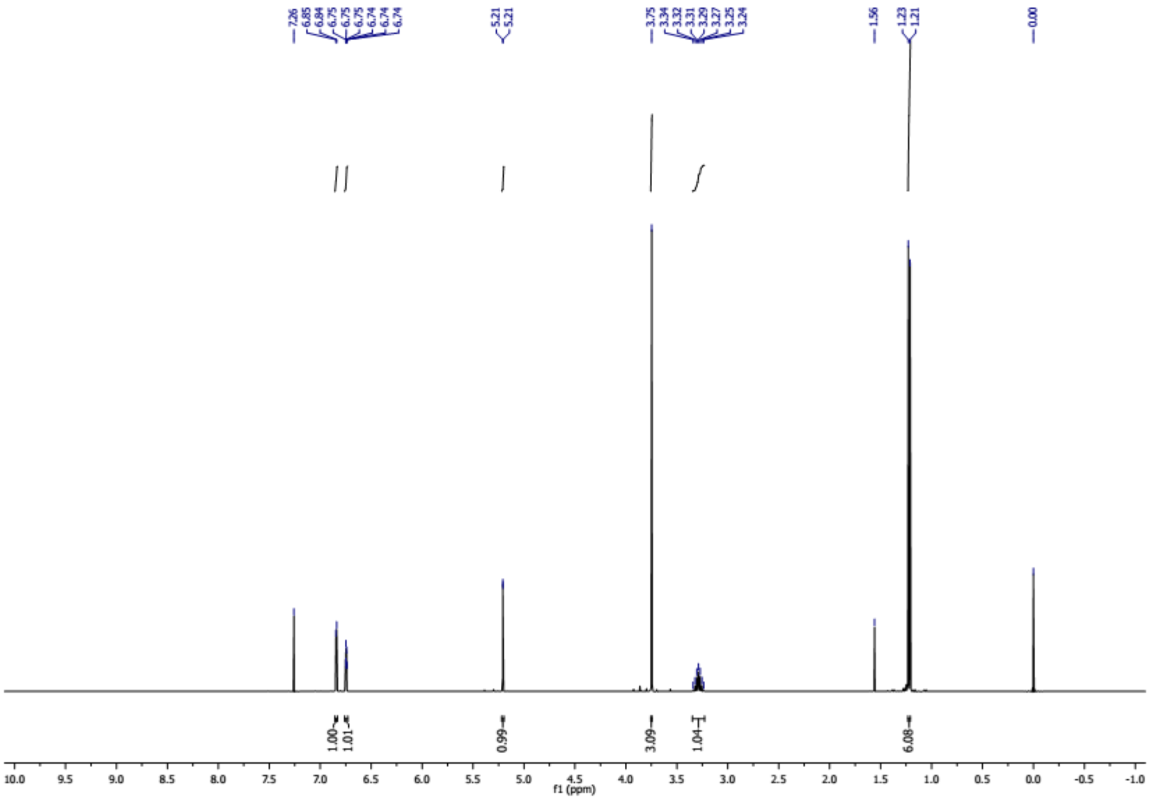
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (d, *J* = 2.9 Hz, 1H), 6.74 (dt, *J* = 2.9, 0.6 Hz, 1H), 5.21 (d, *J* = 0.5 Hz, 1H), 3.75 (s, 3H), 3.29 (hept, *J* = 6.9 Hz, 1H), 1.22 (d, *J* = 6.9 Hz, 6H).

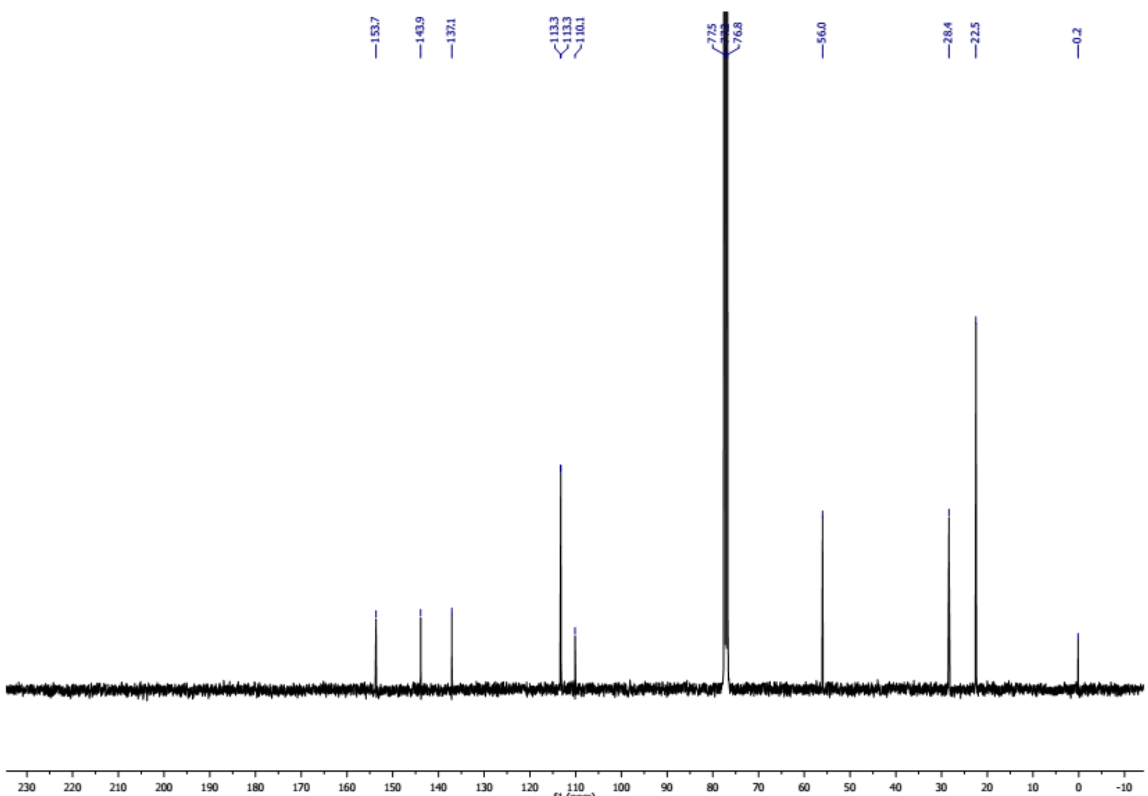
^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7, 143.9, 137.1, 113.3, 113.3, 110.1, 56.0, 28.4, 22.5.

R_f 0.6 (hexanes/EA = 10:1, UV)

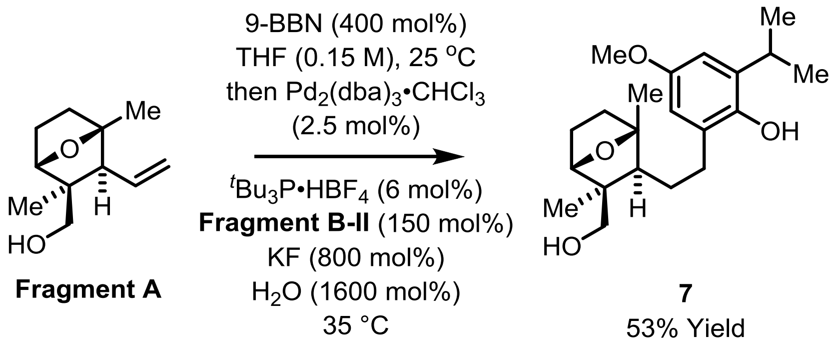
HRMS (ESI) Calcd. for $C_{10}H_{13}BrO_2$ $[M+Na]^+$: 243.0026, Found: 243.0022.

FTIR (neat): 3516, 2961, 1606, 1576, 1474, 1425, 1330, 1292, 1198, 1175, 1157, 1092, 1039, 938, 877, 849, 824, 764, 733 cm^{-1}.


7.26
6.85
6.84
6.75
6.75
6.75
6.74
6.74
6.74
5.21
5.21
3.75
3.34
3.32
3.31
3.29
3.27
3.25
3.24
1.56
1.23
1.21
0.00
1.00
1.01
0.99
3.09
1.04
6.08
f1 (ppm)


153.7
143.9
137.1
113.3
113.3
110.1
77.5
76.8
56.0
28.4
22.5
0.2
f1 (ppm)

2-(2-((1*R*,2*S*,3*S*,4*S*)-3-(Hydroxymethyl)-1,3-dimethyl-7-oxabicyclo[2.2.1]heptan-2-yl)ethyl)-6-isopropyl-4-methoxyphenol (7)



Detailed Procedures

To a solution of alkene (0.0547 g, 0.3 mmol, 100 mol%) in THF (0.3 mL), a solution of 9-BBN (0.146 g, 1.2 mmol, 400 mol%) in THF (1.6 mL) was added slowly at ambient temperature. The mixture was stirred at the same temperature until TLC indicated all the starting alkene was consumed (about an hour). The resulting clear solution was mixed with anhydrous KF (0.139 g, 2.4 mmol, 800 mol%), $Pd_2(dba)_3$·$CHCl_3$ (0.0078 g, 0.0075 mmol, 2.5 mol%) and $^tBu_3P$·HBF_4 (0.0052 g, 0.018 mmol, 6 mol%), followed by addition of water (0.086 mL, 4.8 mmol, 1600 mol%) and the bromophenol (**Fragment B-II**, 0.110 g, 0.45 mmol, 150 mol%). The degassed heterogeneous mixture was heated to 35 ˚C and vigorously stirred overnight. After cooled to ambient temperature, the reaction mixture was diluted with ethyl acetate (2 mL) and water (2 mL). The separated organic layer was washed with 1 M NaOH (1 mL × 2) and brine (1 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/EA = 4:1). The title compound was obtained as a colorless oil (0.0550 g, 0.16 mmol) in 53% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.63 (d, *J* = 3.1 Hz, 1H), 6.50 (d, *J* = 3.1 Hz, 1H), 5.96 (br, 1H), 4.01 (d, *J* = 5.5 Hz, 1H), 3.90 (d, *J* = 11.2 Hz, 1H), 3.76 (s, 3H), 3.70 (d, *J* = 11.2 Hz, 1H), 3.20 (hept, *J* = 6.8 Hz, 1H), 3.07 (br, 1H), 2.93 (td, *J* = 12.9, 4.6 Hz, 1H), 2.37 (ddd, *J* = 13.5, 11.8, 5.5 Hz, 1H), 1.95 (ddd, *J* = 12.8, 9.0, 4.9 Hz, 1H), 1.86-1.65 (m, 3H), 1.60 (ddd, *J* = 11.5, 9.0, 4.4 Hz, 1H), 1.54-1.44 (m, 2H), 1.36 (s, 3H), 1.23 (d, *J* = 7.0 Hz, 3H), 1.21 (d, *J* = 7.0 Hz, 3H), 1.13 (s, 3H).

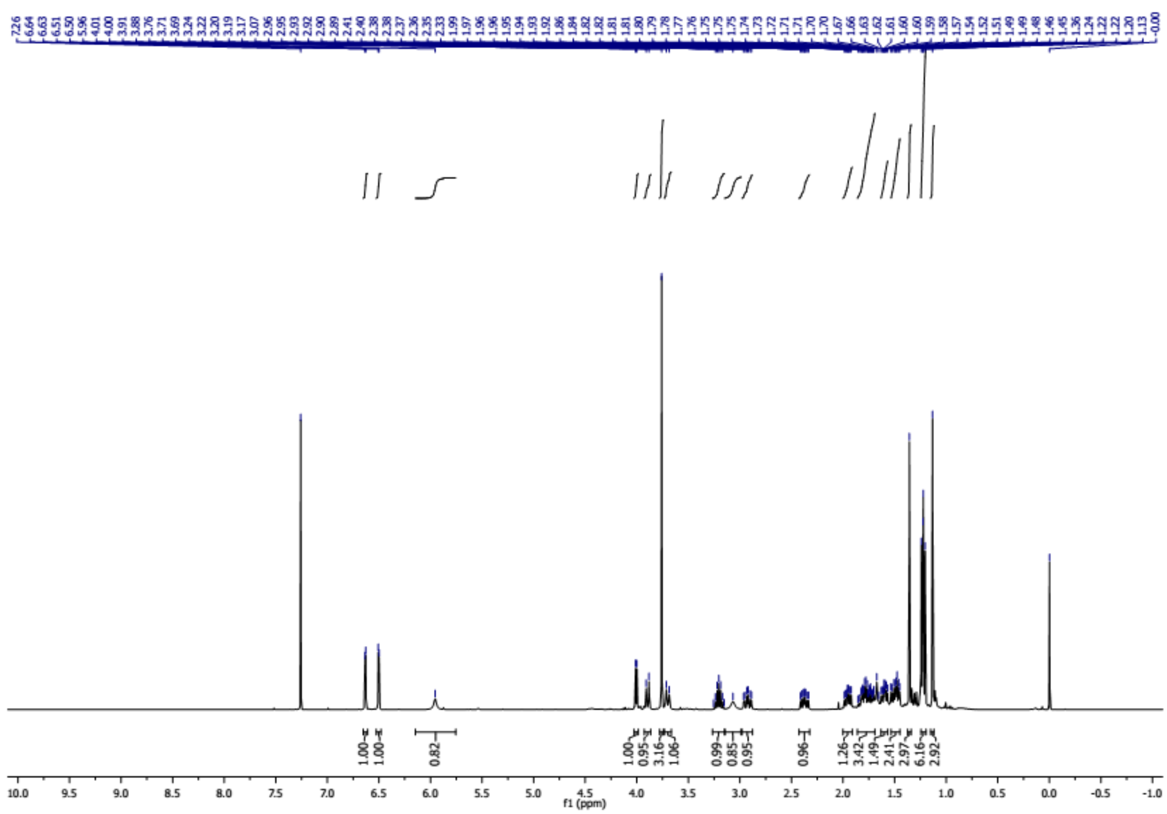
^{13}C NMR (100 MHz, $CDCl_3$) δ 153.3, 145.3, 136.0, 129.0, 112.4, 110.0, 87.4, 83.8, 69.1, 56.5, 55.8, 50.3, 39.4, 32.4, 28.6, 27.4, 25.8, 23.0, 22.8, 20.7, 18.9.

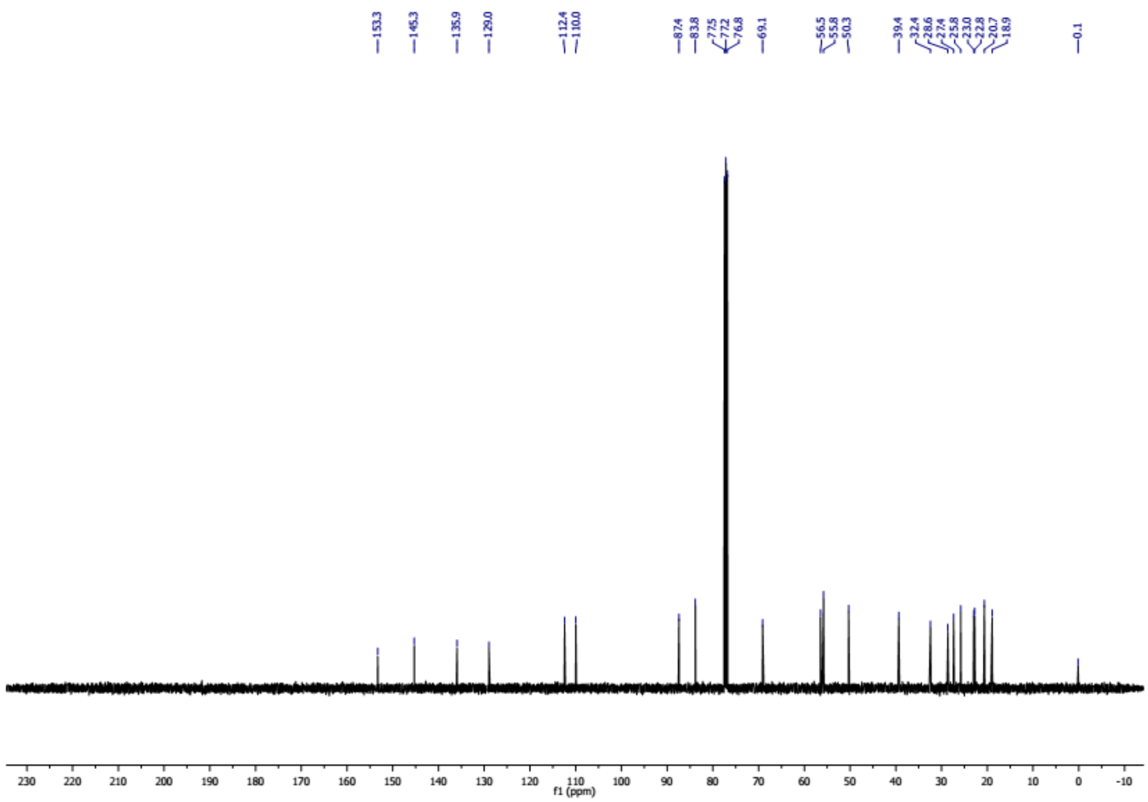
R_f 0.15 (hexanes/acetone = 5:1, *p*-anisaldehyde)

HRMS (ESI) Calcd. for $C_{21}H_{32}O_4$ $[M+Na]^+$: 371.2193, Found: 371.2195.

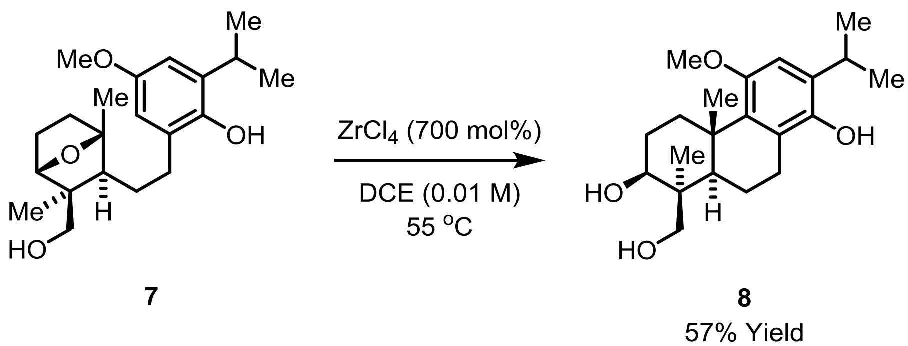
FTIR (neat): 3350, 2960, 2872, 1604, 1467, 1439, 1381, 1310, 1205, 1046, 1005, 988, 868, 828, 757 cm^{-1}.

Optical Rotation $[\alpha]_D^{30}$ = +55.7˚ (*c* = 0.3, $CHCl_3$)




153.3
145.3
135.9
129.0
112.4
110.0
87.4
83.8
77.5
77.2
76.8
69.1
56.5
55.8
50.3
39.4
32.4
28.6
27.4
25.8
23.0
22.8
20.7
18.9
0.1
f1 (ppm)

(4b*S*,7*S*,8*S*,8a*R*)-8-(Hydroxymethyl)-2-isopropyl-4-methoxy-4b,8-dimethyl-4b,5,6,7,8,8a,9,10-octahydrophenanthrene-1,7-diol (8)



Detailed Procedures

To a vigorously stirred suspension of $ZrCl_4$ (0.197 g, 0.84 mmol, 700 mol%) in DCE (10 mL), a solution of **7** (0.0422 g, 0.12 mmol, 100 mol%) in DCE (2 mL) was added in one portion. The mixture was heated to 55 ˚C and allowed to stir for 11 hours. After cooled to ambient temperature, the reaction was poured into saturated $NaHCO_3$ (aq., 20 mL) and allowed to stir for 15 min. The mixture was extracted by DCM (10 mL × 2). The combined organic layers were washed with brine (10 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (DCM/acetone = 10:1). The title compound was obtained as a white solid (0.0239 g, 0.069 mmol) in 57% yield.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.62 (s, 1H), 4.35 (d, *J* = 11.3 Hz, 1H), 4.29 (s, 1H), 3.78 (s, 3H), 3.54 (dd, *J* = 11.8, 4.6 Hz, 1H), 3.41 (d, *J* = 11.3 Hz, 1H), 3.23-3.11 (m, 2H), 2.83 (ddd, *J* = 16.6, 5.6, 1.3 Hz, 1H), 2.56 (ddd, *J* = 16.6, 12.3, 6.9 Hz, 1H), 2.09-1.92 (m, 2H), 1.86-1.77 (m, 1H), 1.55 (qd, *J* = 12.3, 5.5 Hz, 1H), 1.37 (d, *J* = 12.1 Hz, 1H), 1.34 (s, 3H), 1.34-1.28 (m, 1H), 1.28 (s, 3H), 1.27 (d, *J* = 6.9 Hz, 3H), 1.25 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 152.5, 144.6, 135.0, 131.2, 123.7, 108.2, 80.7, 64.4, 55.9, 52.9, 43.6, 38.9, 34.7, 28.9, 27.5, 27.3, 23.0, 22.8, 22.6, 20.7, 18.4.

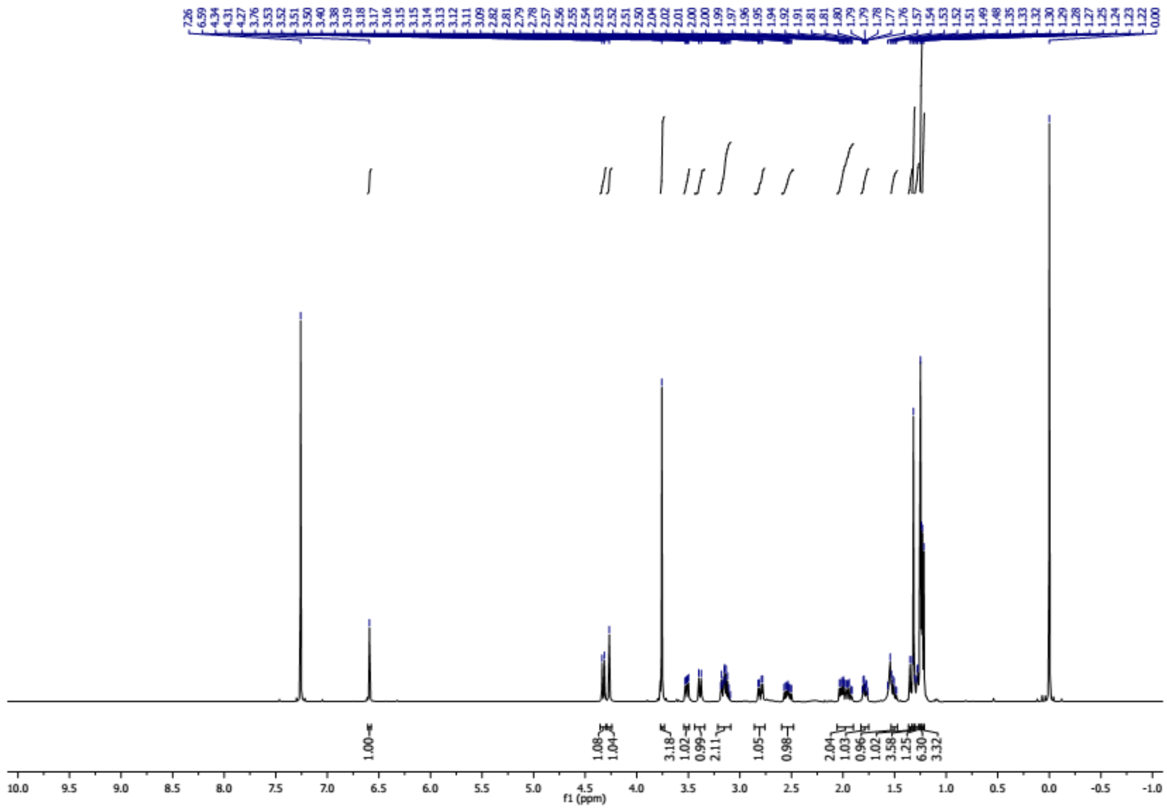
R_f 0.3 (DCM/acetone = 5:1, *p*-anisaldehyde)

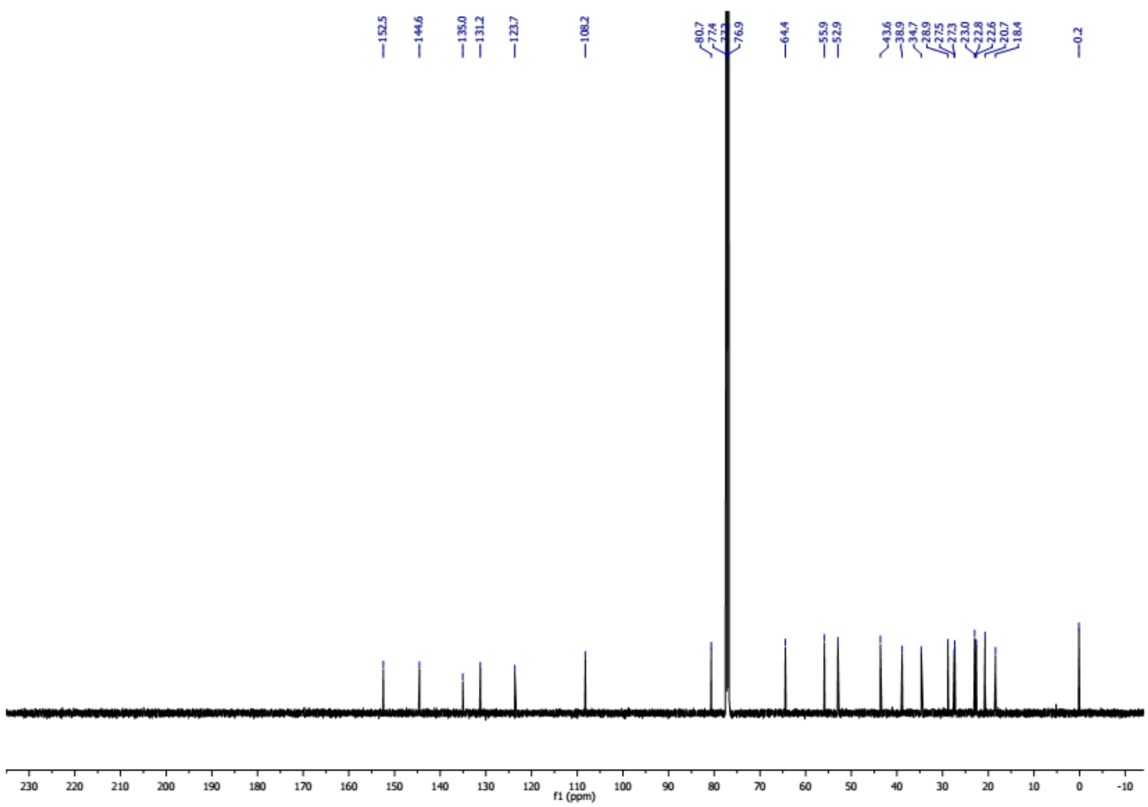
HRMS (ESI) Calcd. for $C_{21}H_{32}O_4$ $[M+Na]^+$: 371.2193, Found: 371.2201.

FTIR (neat): 3381, 2954, 2928, 2868, 1649, 1462, 1413, 1286, 1235, 1101, 1071, 1034, 992, 976, 919, 907, 817, 755 cm^{-1}.

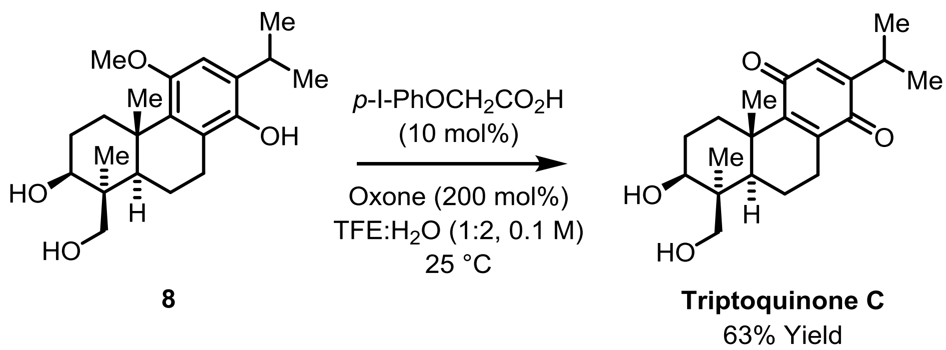
MP 206.4-208.1 ˚C ($CHCl_3$)

Optical Rotation $[\alpha]_D^{30}$ = +75.3˚ (*c* = 0.17, $CHCl_3$)





(4b*S*,7*S*,8*S*,8a*R*)-7-Hydroxy-8-(hydroxymethyl)-2-isopropyl-4b,8-dimethyl-4b,5,6,7,8,8a,9,10-octahydrophenanthrene-1,4-dione ((-)-Triptoquinone C)



Detailed Procedures

To a mixture of **8** (0.0151 g, 0.043 mmol, 100 mol%) and *p*-iodo-phenoxyacetic acid[3] (0.0012 g, 0.0043 mmol, 10 mol%) in 2,2,2-trifluoroethanol (0.14 mL) and water (0.29 mL), Oxone® (0.0533 g, 0.087 mmol, 200 mol%) was added in one portion. The reaction turned yellow slowly, and was allowed to stir at ambient temperature for 2 hours. The mixture was diluted with ethyl acetate (2 mL), washed with water (2 mL) and saturated $NaHCO_3$ (aq., 2 mL), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (hexanes/EA = 3:2). The title compound was obtained as a yellow solid (0.0091 g, 0.027 mmol) in 63% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.32 (d, *J* = 1.3 Hz, 1H), 4.26 (d, *J* = 11.2 Hz, 1H), 3.49 (d, *J* = 11.8 Hz, 1H), 3.35 (t, *J* = 9.6 Hz, 1H), 2.98 (heptd, *J* = 6.9, 1.2 Hz, 1H), 2.81 (dt, *J* = 13.6, 3.6 Hz, 1H), 2.78 (br, 1H), 2.73 (ddd, *J* = 20.3, 5.7, 1.2 Hz, 1H), 2.39 (br, 1H), 2.31 (ddd, *J* = 20.3, 11.6, 7.3 Hz, 1H), 2.03-1.90 (m, 2H), 1.81 (dq, *J* = 13.5, 3.9 Hz, 1H), 1.39 (ddd, *J* = 25.0, 12.5, 5.6 Hz, 1H), 1.29 (s, 3H), 1.28-1.19 (m, 1H), 1.23 (s, 3H), 1.19 (dd, *J* = 12.5, 1.5 Hz, 1H), 1.10 (d, *J* = 6.8 Hz, 3H), 1.09 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 187.9, 187.8, 153.2, 149.7, 142.8, 132.1, 80.3, 64.1, 51.9, 43.3, 38.0, 34.3, 28.3, 26.6, 26.5, 22.8, 21.5, 21.5, 21.0, 17.5.

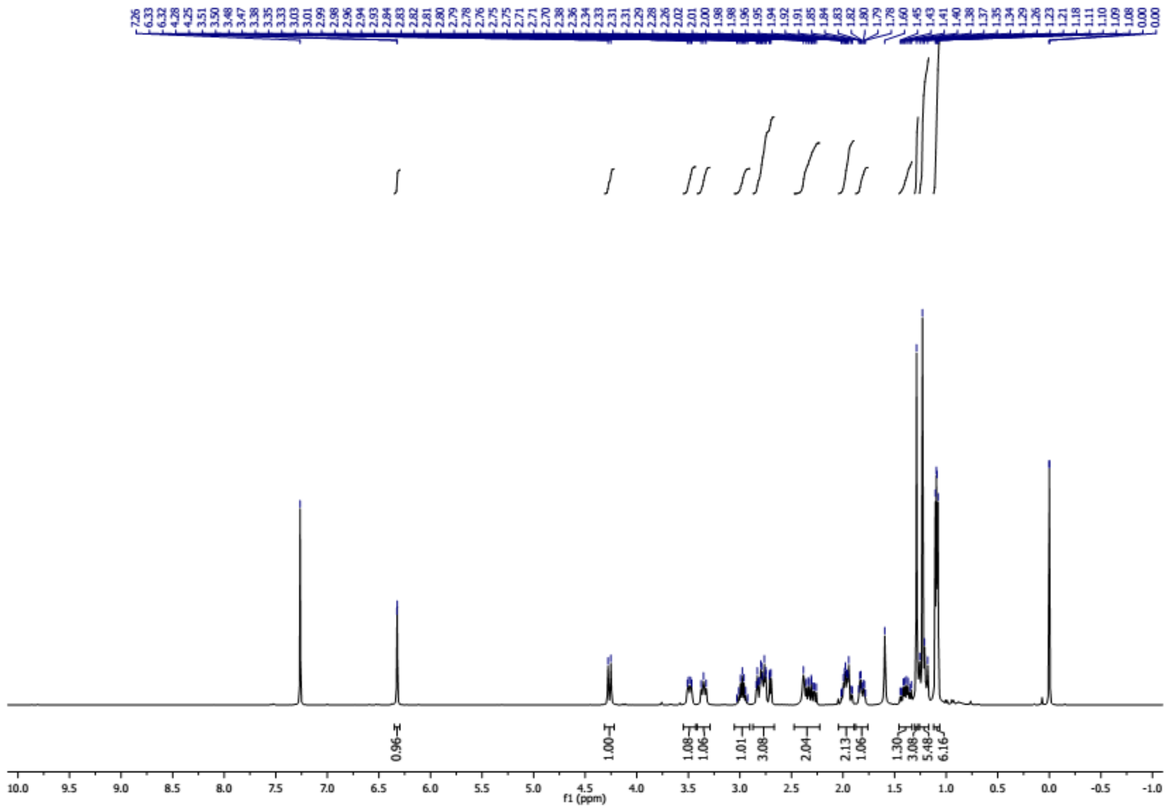
R_f 0.2 (hexanes/EA = 3:2, UV)

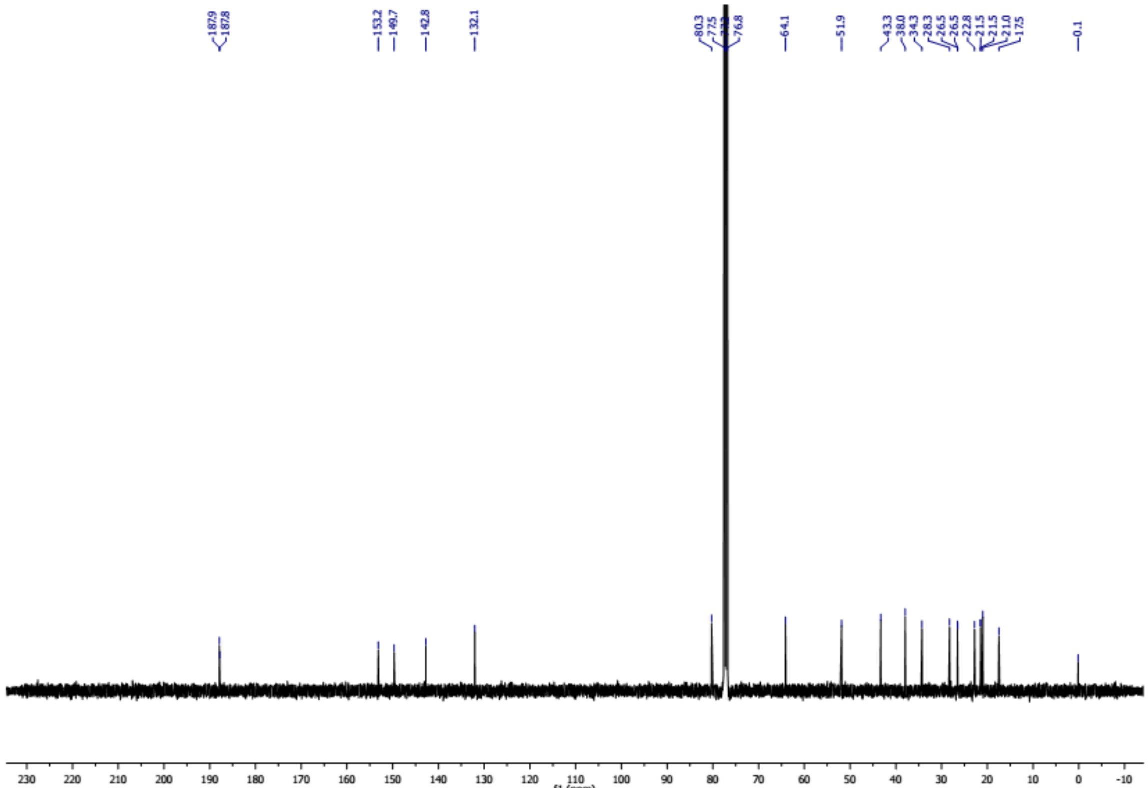
HRMS (ESI) Calcd. for $C_{20}H_{28}O_4$ $[M+Na]^+$: 355.1880, Found: 355.1880.

FTIR (neat): 3382, 2963, 2932, 2873, 1647, 1599, 1460, 1291, 1265, 1230, 1080, 1037, 907, 738 cm^{-1}.

MP 161.0-162.0 ˚C ($CHCl_3$)

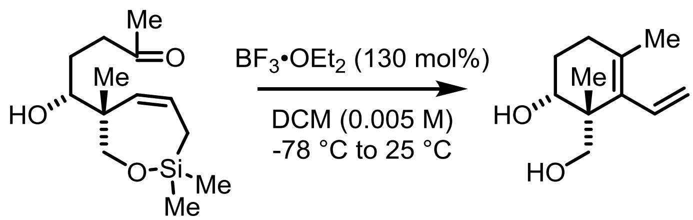
Optical Rotation $[\alpha]_D^{30}$ = -43.0˚ (*c* = 0.3, $CHCl_3$)





Detailed Procedures and Spectral Data for Asymmetric Synthesis of Isoiresin

(1*R*,2*R*)-2-(Hydroxymethyl)-2,4-dimethyl-3-vinylcyclohex-3-en-1-ol (*iso*-Fragment A)



***ent*-4**

***iso*-Fragment A**
63% Yield

Detailed Procedures

To a solution of ***ent*-4**[4] (0.320 g, 1.25 mmol, 100 mol%) in DCM (250 mL) at -78 ˚C, $BF_3·OEt_2$ (0.46 mL, 3.74 mmol, 130 mol%) was added dropwise. The resulting solution was allowed to warm to ambient temperature in an hour, and stirred for 22 hours. The reaction was quenched by addition of saturated $NaHCO_3$ (aq., 250 mL) and vigorously stirred for overnight. The mixture was separated, and the aqueous layer was extracted with DCM (50 mL × 2). The combined organic layer was washed with brine (100 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure (<30 ˚C), and the residue was submitted to flash column chromatography on a short plug of neutral alumina (Et_2O/MeOH = 30:1 to 5:1). The title compound was obtained as a light brown oil (0.143 g, 0.79 mmol) in 63% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.22 (ddtd, *J* = 15.3, 11.2, 2.0, 1.0 Hz, 1H), 5.32 (dd, *J* = 11.2, 2.5 Hz, 1H), 5.05 (dd, *J* = 17.7, 2.5 Hz, 1H), 3.81 (t, *J* = 4.9 Hz, 1H), 3.79 (d, *J* = 9.8 Hz, 1H), 3.59 (d, *J* = 11.5 Hz, 1H), 3.14 (br, 1H), 3.01 (br, 1H), 2.27 (dt, *J* = 16.2, 7.0 Hz, 2H), 2.02 (dt, *J* = 17.4, 5.5 Hz, 1H), 1.87 – 1.80 (m, 2H), 1.74 (d, *J* = 1.0 Hz, 3H), 1.02 (s, 3H).

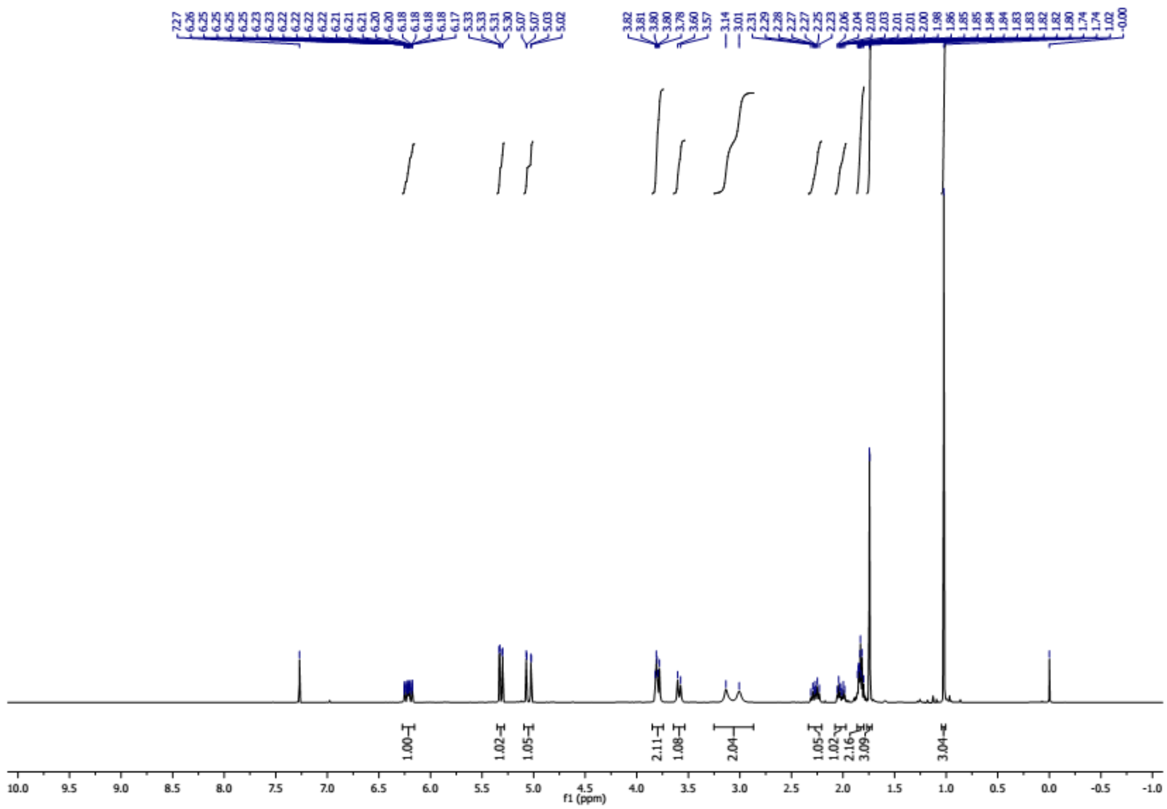
^{13}C NMR (100 MHz, $CDCl_3$) δ 135.1, 132.4, 131.7, 119.5, 76.4, 68.8, 42.4, 28.5, 26.7, 22.0, 21.4.

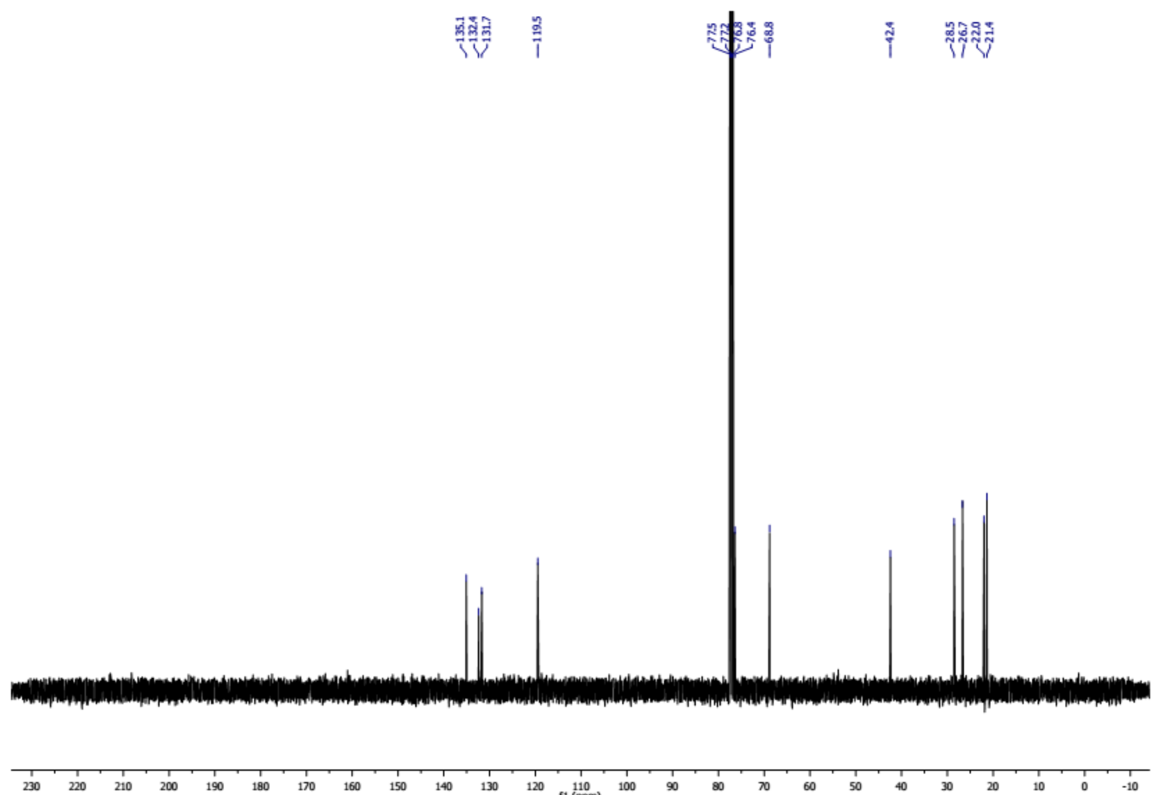
R_f 0.35 (hexanes/EA = 2:1 (twice), UV/*p*-anisaldehyde)

HRMS (ESI) Calcd. for $C_{11}H_{18}O_2$ $[M+Na]^+$: 205.1199, Found: 205.1204.

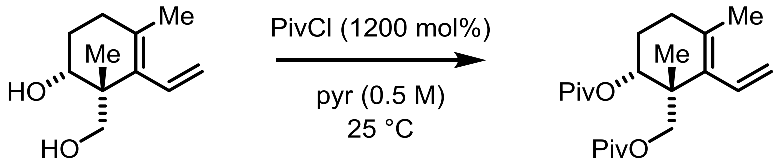
FTIR (neat): 3348, 2969, 2931, 2875, 1431, 1375, 1250, 1231, 1217, 1200, 1093, 1044, 1019, 1003, 918, 836, 809 cm^{-1}.

Optical Rotation $[\alpha]_D^{30}$ = -8.0˚ (*c* = 1.0, $CHCl_3$)


f1 (ppm)


135.1
132.4
131.7
119.5
77.5
77.2
76.8
76.4
68.8
42.4
28.5
26.7
22.0
21.4
f1 (ppm)

(1*R*,2*R*)-2,4-Dimethyl-2-((pivaloyloxy)methyl)-3-vinylcyclohex-3-en-1-yl pivalate (10)



***iso*-Fragment A**

10
81% Yield

Detailed Procedures

To a pyridine (0.8 mL) solution of diol (0.0712 g, 0.39 mmol, 100 mol%) in an ice bath, freshly distilled pivaloyl chloride (0.58 mL, 4.7 mmol, 1200 mol%) was added slowly. White precipitate was formed immediately. The reaction mixture was allowed to warm to ambient temperature, and stirred for 18 hours. Water (1.0 mL) and DCM (1.0 mL) was added to quench the reaction. The resulting two layers were separated, and the aqueous layer was extracted with DCM (5 mL). The combined organic layers were added MeOH (3.2 mL), Et_3N (0.60 mL) and DMAP (0.020 g), and stirred overnight to decompose the excess PivCl. The DCM solution was washed with 0.5 M H_3PO_4 (5 mL) and saturated $NaHCO_3$ (aq., 5 mL × 2), and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the residue was submitted to flash column chromatography on silica gel (Hexanes/EA = 100:1). The title compound was obtained as a colorless oil (0.116 g, 0.33 mmol) in 81% yield, which solidified upon standing in a -20 ˚C freezer.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.11 (dd, *J* = 17.5, 11.2 Hz, 1H), 5.31 (dd, *J* = 11.2, 2.4 Hz, 1H), 5.01 (dd, *J* = 17.6, 2.4 Hz, 1H), 4.93 (dd, *J* = 7.1, 2.7 Hz, 1H), 4.09 (d, *J* = 10.8 Hz, 1H), 4.00 (d, *J* = 10.8 Hz, 1H), 2.20-1.90 (m, 3H), 1.81 (dddd, *J* = 13.7, 8.9, 6.7, 2.7 Hz, 1H), 1.73 (s, 3H), 1.18 (s, 9H), 1.17 (s, 9H), 1.10 (s, 3H).

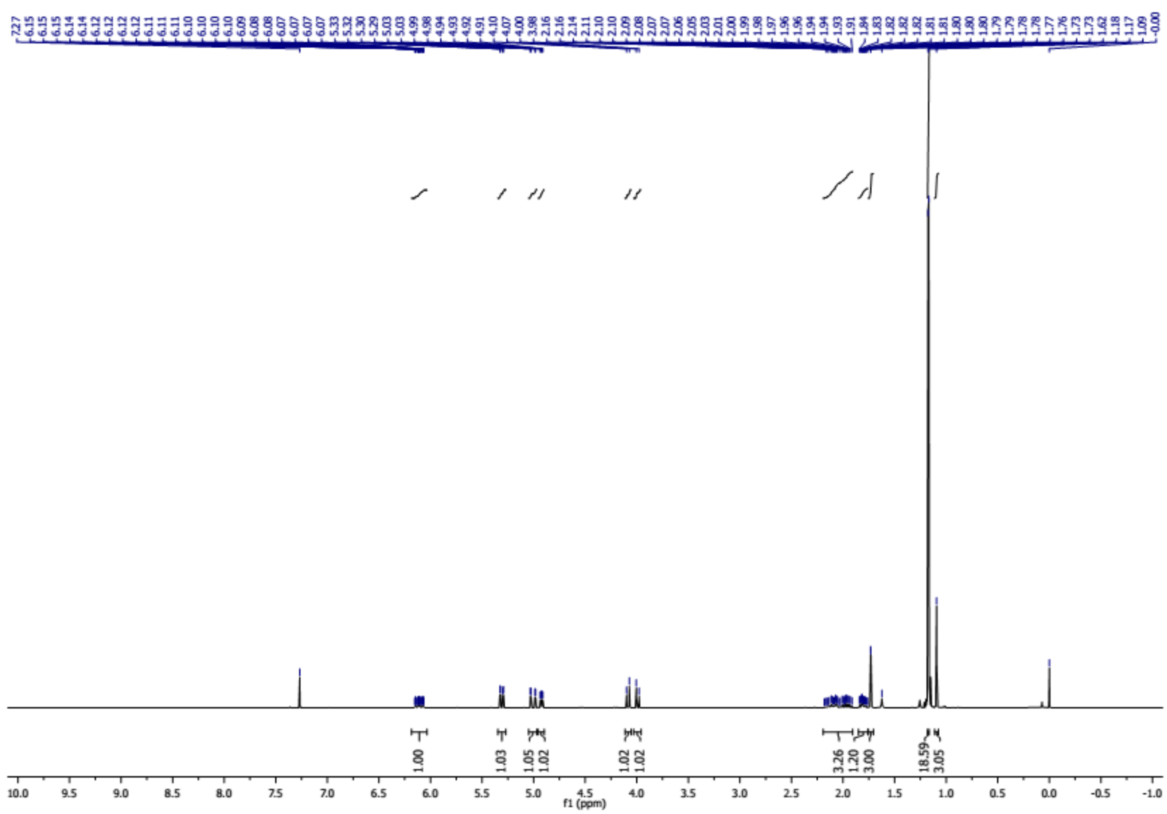
^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 177.7, 134.0, 132.2, 130.7, 120.0, 73.8, 67.4, 41.0, 39.2, 39.0, 28.5, 27.3, 27.3, 22.8, 22.3, 21.3.

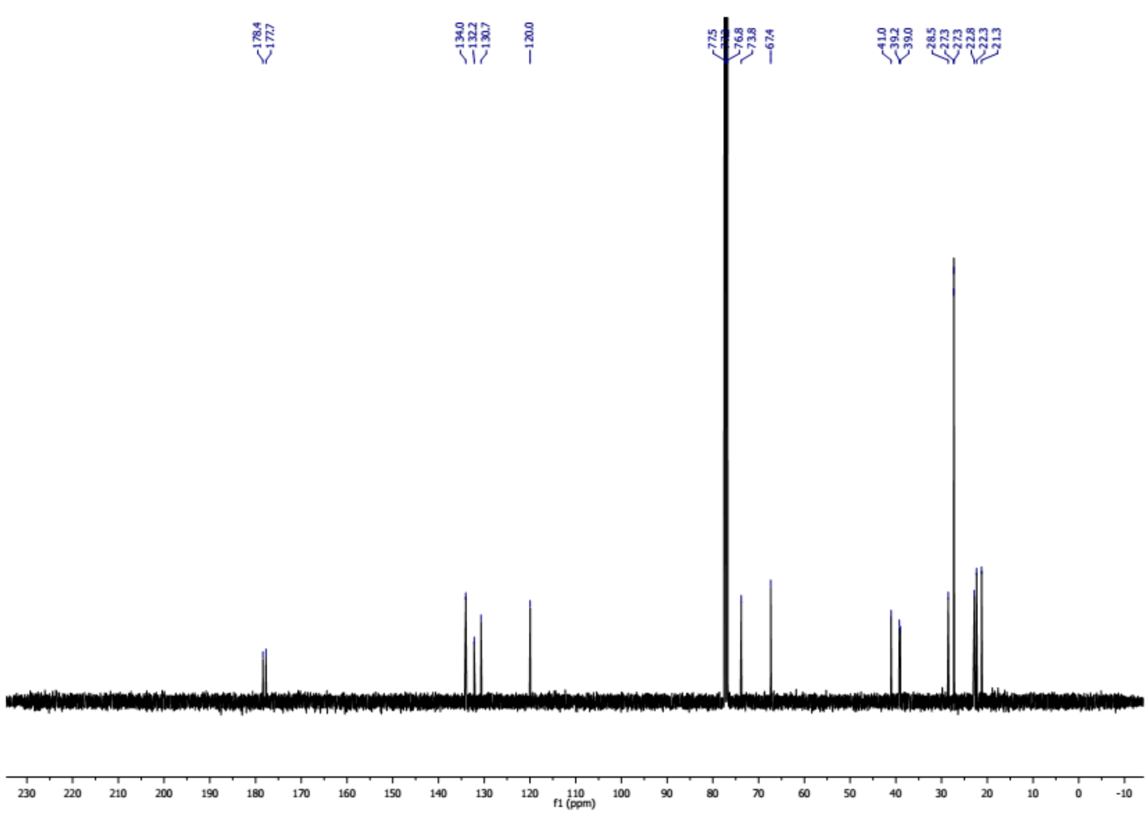
HRMS (ESI) Calcd. for $C_{21}H_{34}O_4$ $[M+Na]^+$: 373.2349, Found: 373.2350.

FTIR (neat): 2970, 2931, 2872, 1727, 1480, 1460, 1397, 1364, 1282, 1164, 1143, 1033, 989, 921 cm^{-1}.

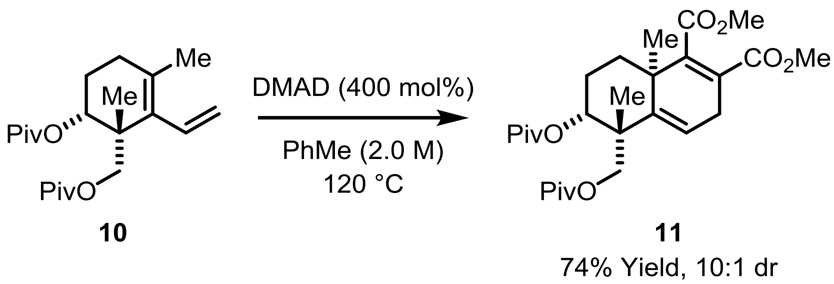
MP 44.4-45.3 ˚C (Et_2O)

Optical Rotation $[\alpha]_D^{30}$ = -2.2˚ (*c* = 0.5, $CHCl_3$)





Dimethyl (5*R*,6*R*,8a*R*)-5,8a-dimethyl-6-(pivaloyloxy)-5-((pivaloyloxy)methyl)-3,5,6,7,8,8a-hexahydronaphthalene-1,2-dicarboxylate (11)



Detailed Procedures

To a solution of **10** (0.0905 g, 0.26 mmol, 100 mol%) in toluene (0.13 mL) was added dimethyl acetylenedicarboxylate (DMAD, 0.13 mL, 1.03 mmol, 400 mol%). The mixture was heated to 120 ˚C in seal tube for 24 hours. After cooled to ambient temperature, the solvent was removed under reduced pressure and the residue was submitted to flash column chromatography on silica gel (hexanes/EA = 15:1). The title compound was obtained with its inseparable diastereomer in 10:1 ratio as a white solid (0.0941 g, 0.19 mmol) in 74% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.83 (dd, *J* = 5.6, 2.3 Hz, 1H (major)), 5.61* (dd, *J* = 5.7, 2.2 Hz, 1H (minor)), 4.94-4.90* (m, 1H (minor)), 4.63 (dd, *J* = 9.7, 5.9 Hz, 1H (major)), 4.46 (d, *J* = 11.3 Hz, 1H (major)), 4.25* (d, *J* = 10.2 Hz, 1H (minor)), 4.05* (d, *J* = 10.3 Hz, 1H (minor)), 4.01 (d, *J* = 11.3 Hz, 1H (major)), 3.85* (s, 3H (minor)), 3.80 (s, 3H (major)), 3.75* (s, 3H (minor)), 3.75 (s, 3H (major)), 3.24 (dd, *J* = 22.8, 5.5 Hz, 1H (major)), 3.21* (dd, *J* = 22.7, 5.7 Hz, 1H (minor)), 2.86 (dd, *J* = 22.8, 2.3 Hz, 1H (major)), 2.83* (dd, *J* = 22.6, 2.2 Hz, 1H (minor)), 2.08-1.71* (m, 4H (minor)), 1.88-1.75 (m, 3H (major)), 1.62 (dd, *J* = 9.6, 3.4 Hz, 1H (major)), 1.47* (s, 3H (minor)), 1.37 (s, 3H (major)), 1.32* (s, 3H (minor)), 1.22 (s, 9H (major)), 1.20 (s, 3H (major)), 1.19* (s, 9H (minor)), 1.17 (s, 9H (major)), 1.13* (s, 9H (minor)).

^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 177.7, 169.3, 166.2, 149.3, 139.3, 125.3, 123.6, 77.3, 66.6, 52.4, 52.3, 44.3, 39.2, 39.1, 38.2, 32.8, 27.4, 27.3, 27.1, 25.1, 23.3, 23.1.

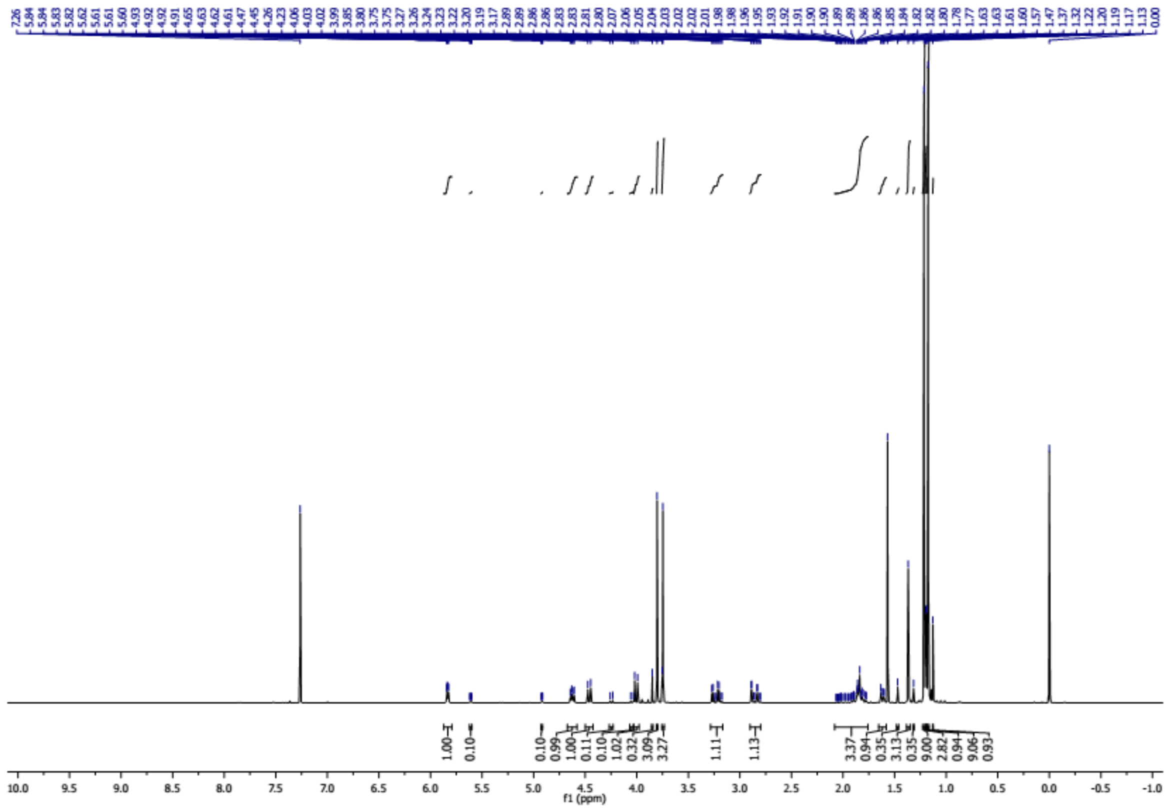
R_f 0.15 (hexanes/EA = 10:1, UV/*p*-anisaldehyde)

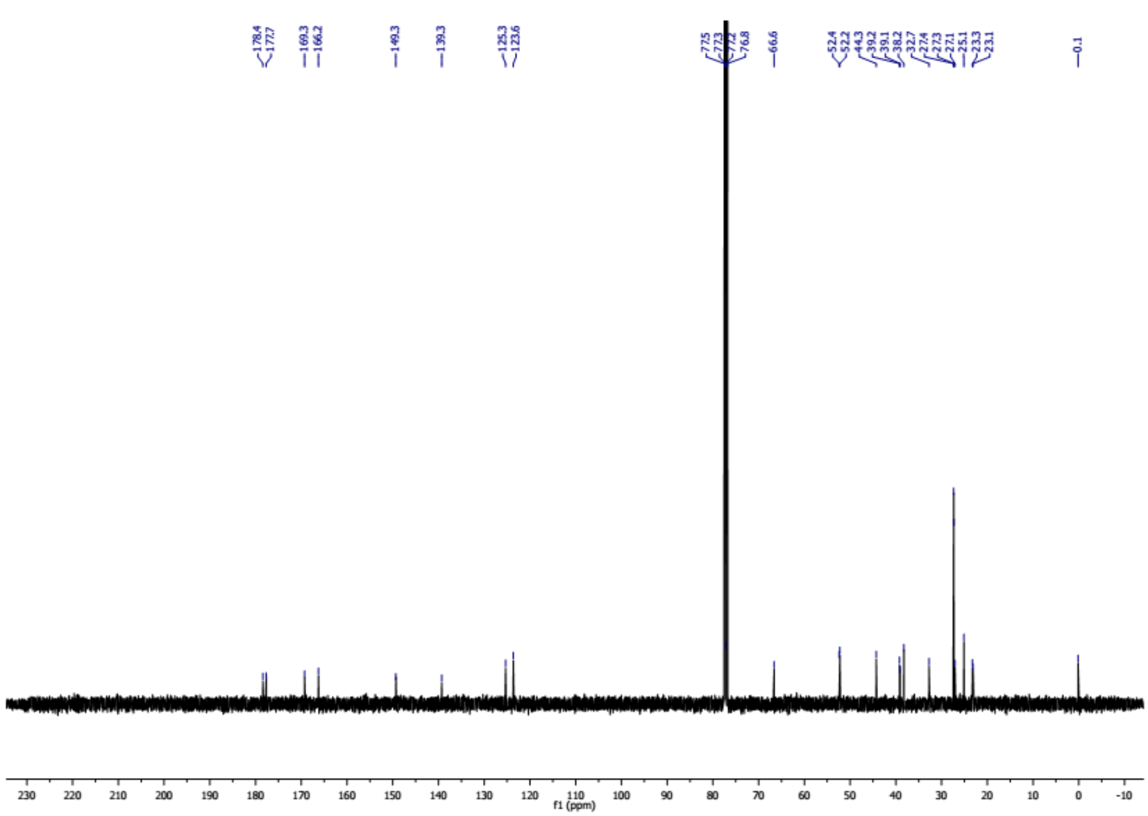
HRMS (ESI) Calcd. for $C_{27}H_{40}O_8$ $[M+Na]^+$: 515.2615, Found: 515.2628.

FTIR (neat): 2975, 2873, 1725, 1639, 1480, 1459, 1434, 1397, 1367, 1281, 1252, 1144, 1023, 993, 768, 743 cm^{-1}.

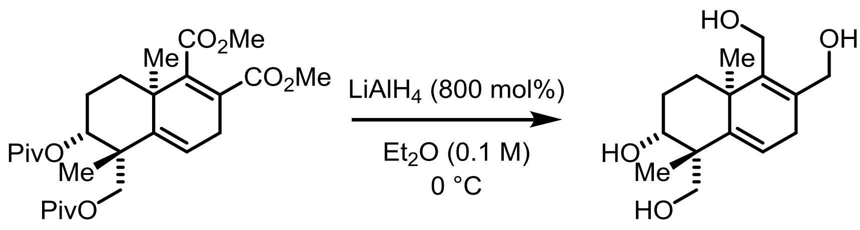
MP 63.6-65.6 ˚C (Et_2O)

Optical Rotation $[\alpha]_D^{30}$ = -18.3˚ (*c* = 0.4, $CHCl_3$)


f1 (ppm)


f1 (ppm)

((5*R*,6*R*,8a*R*)-6-Hydroxy-5,8a-dimethyl-3,5,6,7,8,8a-hexahydronaphthalene-1,2,5-triyl)trimethanol (12)



11

12
78% Yield

Detailed Procedures

To a solution of **11** (0.0507 g, 0.103 mmol, 100 mol%) in diethyl ether (1.0 mL) at 0 ˚C was added $LiAlH_4$ (0.0312 g, 0.823 mmol, 800 mol%). The resulting mixture was stirred vigorously at the same temperature for 2 hours. Water (0.031 mL) was slowly added to quench the reaction, followed by addition of 15% NaOH (aq., 0.031 mL) and water (0.093 mL). The mixture was allowed to stir for 10 min, and was dried over $MgSO_4$. Silica gel was added and solvent was removed under reduced pressure. The resulting residue was directly loaded onto column and submitted to flash chromatography on silica gel (DCM/MeOH = 10:1). The title compound was obtained as a white solid (0.0216 g, 0.0805 mmol) in 78% yield.

^{1}H NMR (400 MHz, CD_3OD) δ 5.83 (dd, *J* = 4.9, 3.0 Hz, 1H), 4.30 (d, *J* = 12.0 Hz, 1H), 4.25 (d, *J* = 12.3 Hz, 1H), 4.14 (d, *J* = 12.0 Hz, 1H), 4.14 (d, *J* = 12.2 Hz, 1H), 3.92 (d, *J* = 11.0 Hz, 1H), 3.59 (d, *J* = 11.1 Hz, 1H), 3.37 (dd, *J* = 11.8, 4.4 Hz, 1H), 2.85 (d, *J* = 3.5 Hz, 1H), 2.84 (d, *J* = 5.3 Hz, 1H), 2.05-1.92 (m, 2H), 1.83-1.76 (m, 1H), 1.64 (td, *J* = 13.9, 3.9 Hz, 1H), 1.31 (s, 3H), 1.18 (s, 3H).

^{13}C NMR (100 MHz, CD_3OD) δ 144.4, 141.8, 134.6, 124.3, 78.6, 67.7, 62.8, 57.5, 47.7, 39.6, 34.8, 30.8, 28.2, 26.4, 23.2.

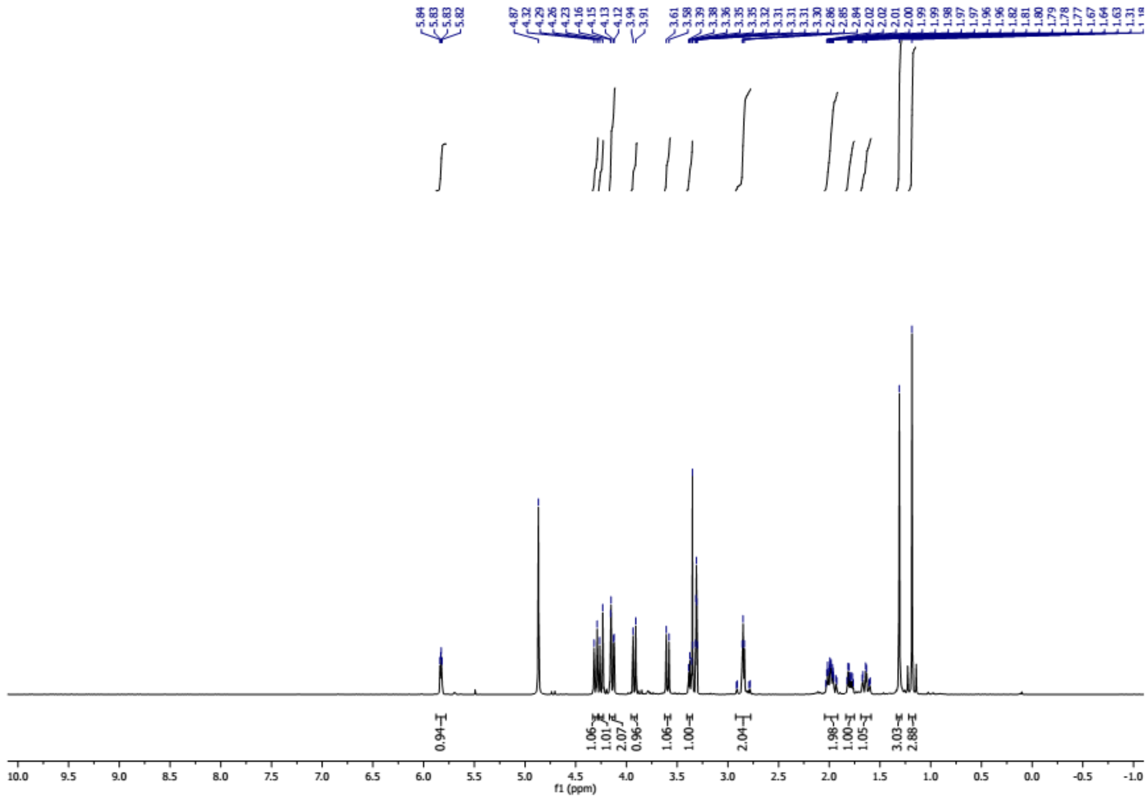
R_f 0.15 (DCM/MeOH = 12.5:1, *p*-anisaldehyde)

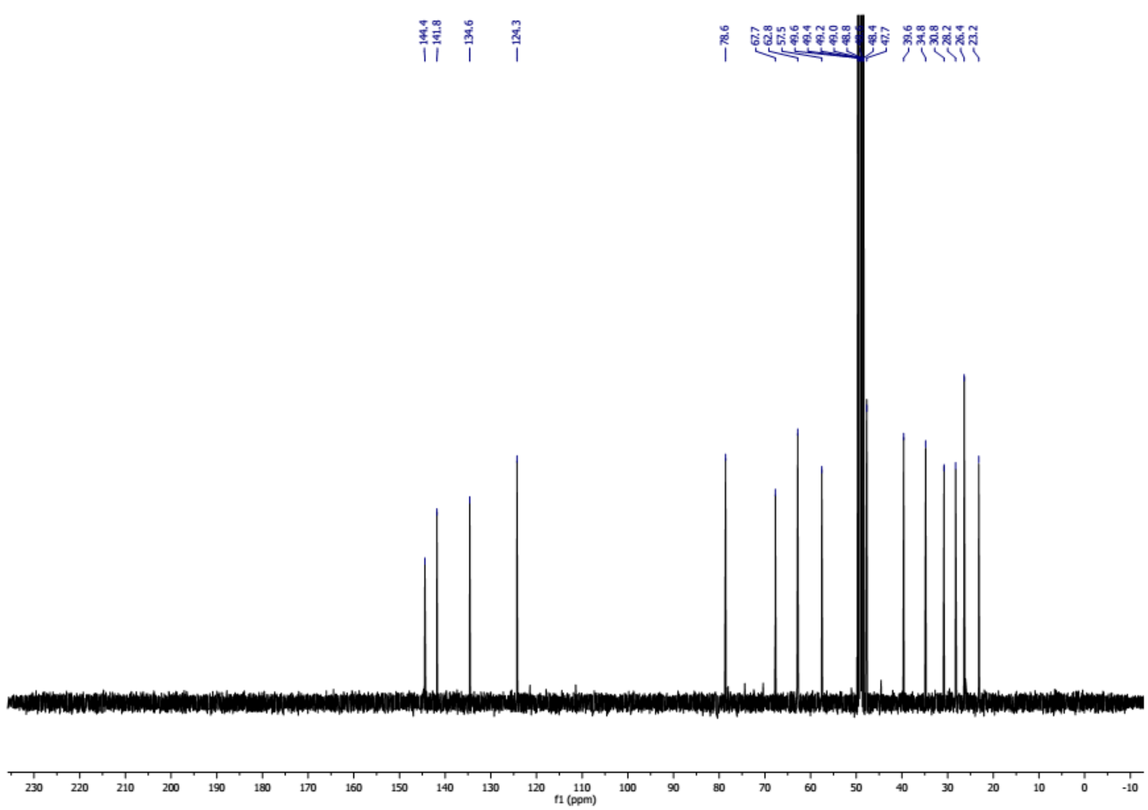
HRMS (ESI) Calcd. for $C_{15}H_{24}O_4$ $[M+Na]^+$: 291.1567, Found: 291.1575.

FTIR (neat): 3356, 2930, 2874, 1562, 1408, 1060, 1030, 1001 cm^{-1}.

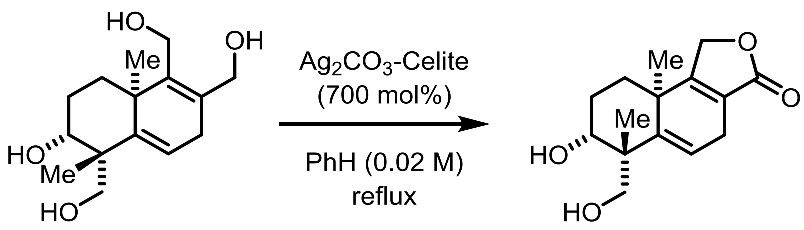
MP 128.0-129.0 ˚C (MeOH)

Optical Rotation $[\alpha]_D^{30}$ = +12.2˚ (*c* = 0.15, CH_3OH)





(6*R*,7*R*,9a*R*)-7-Hydroxy-6-(hydroxymethyl)-6,9a-dimethyl-4,6,7,8,9,9a-hexahydronaphtho[1,2-c]furan-3(1H)-one (S1)



12

S1
70% Yield

Detailed Procedures

A suspension of tetraol **12** (0.0300 g, 0.11 mmol, 100 mol%, dried by azeotropic distillation with benzene) and freshly made Ag_2CO_3-Celite[5] (Fétizon's reagent, 0.4461g, 0.78 mmol, 700 mol%) in anhydrous benzene (5.5 mL) was heated to reflux (a portion of benzene could be removed by distillation to eliminate water in the system). The mixture was vigorously stirred until all the starting material was consumed (about 3.5 hours). The reaction was allowed to cool to ambient temperature and filter through filter paper. The precipitate was washed with MeOH (10 mL × 3), and the combined filtrate was concentrated under reduced pressure. The residue was submitted to flash column chromatography on silica gel (DCM/EA = 15:1 to DCM/MeOH = 25:1). The title compound was obtained as a colorless gel (0.0208 g, 0.079 mmol) in 70% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.97 (dd, *J* = 5.1, 2.5 Hz, 1H), 4.87 (ddd, *J* = 16.8, 3.4, 1.4 Hz, 1H), 4.73 (dt, *J* = 16.9, 2.6 Hz, 1H), 4.20 (d, *J* = 11.1 Hz, 1H), 3.54 (dd, *J* = 11.8, 3.9 Hz, 1H), 3.51 (d, *J* = 11.9 Hz, 1H), 3.00 (dddd, *J* = 22.4, 4.8, 2.6, 1.4 Hz, 2H), 2.88 (dq, *J* = 22.4, 2.6 Hz, 1H), 2.42 (br, 1H), 2.24 (br, 1H), 2.10 (tdd, *J* = 13.6, 11.8, 3.9 Hz, 1H), 1.92 (dq, *J* = 13.5, 3.9 Hz, 1H), 1.74 (dt, *J* = 13.1, 3.6 Hz, 1H), 1.65 (dd, *J* = 13.4, 4.0 Hz, 1H), 1.44 (s, 3H), 1.34 (s, 3H).

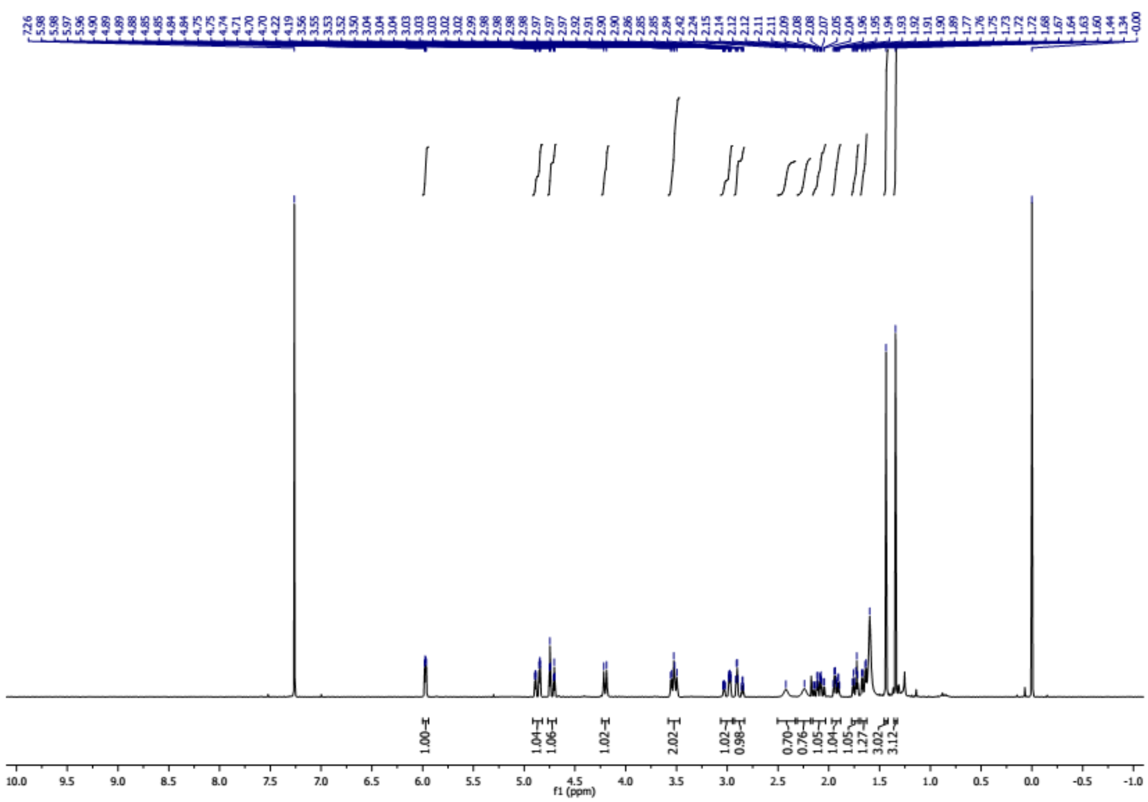
^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 167.4, 142.9, 123.3, 122.2, 77.9, 68.7, 67.9, 47.3, 37.2, 33.7, 27.2, 27.2, 22.9, 22.6.

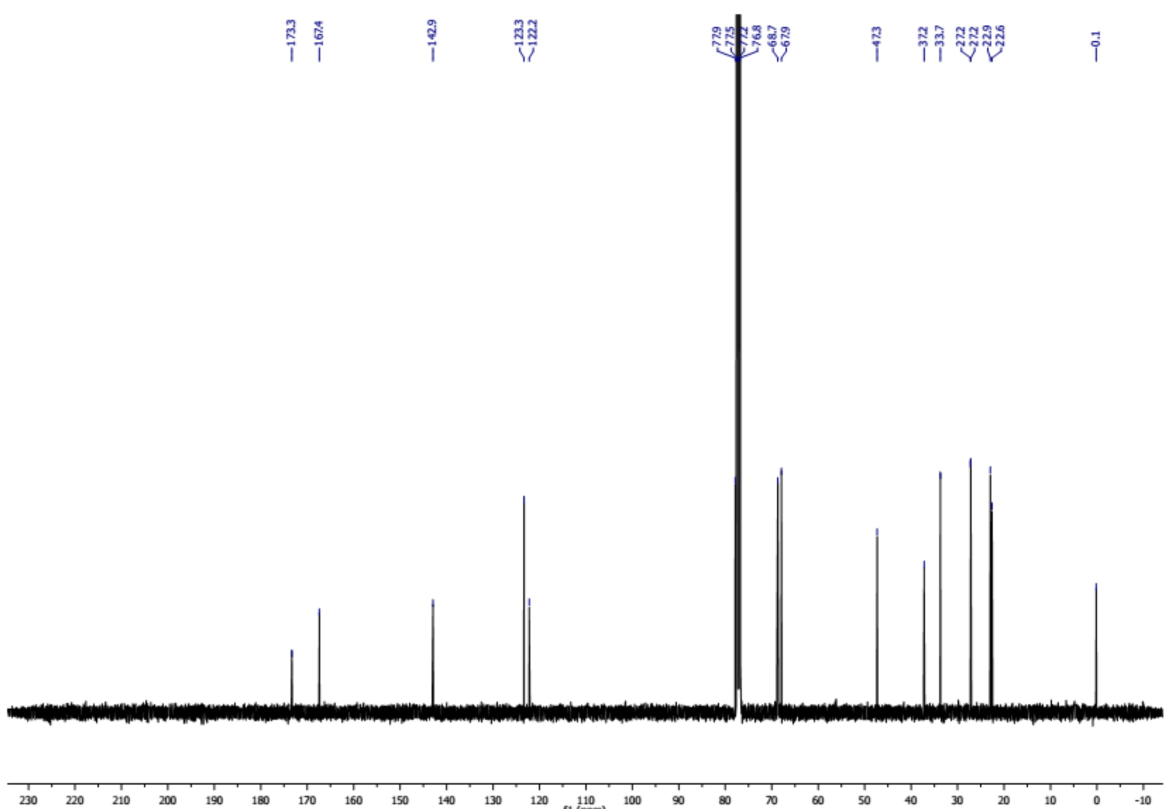
R_f 0.20 (DCM/EA = 1:1, *p*-anisaldehyde)

HRMS (ESI) Calcd. for $C_{15}H_{20}O_4$ $[M+Na]^+$: 287.1254, Found: 287.1262.

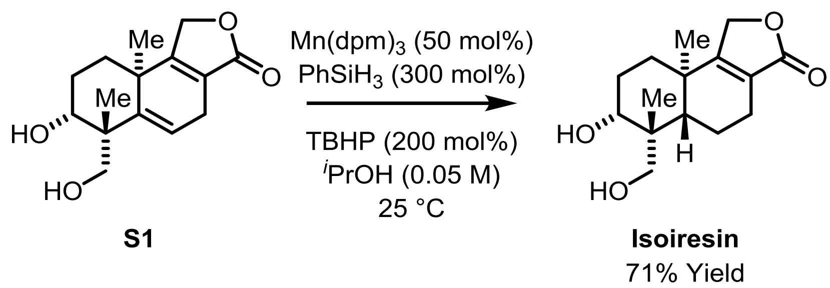
FTIR (neat): 3400, 2927, 2858, 1743, 1696, 1555, 1456, 1260, 1031, 992, 799, 750, 667 cm^{-1}.

Optical Rotation $[\alpha]_D^{30}$ = +1.97° (*c* = 0.18, $CHCl_3$)





(5a*S*,6*R*,7*R*,9a*R*)-7-Hydroxy-6-(hydroxymethyl)-6,9a-dimethyl-4,5,5a,6,7,8,9,9a-octahydronaphtho[1,2-c]furan-3(1H)-one ((-)-Isoiresin)



Detailed Procedures

To a reaction vessel containing **S1** (0.0078g, 0.030 mmol, 100 mol%) was added a degassed solution of $PhSiH_3$ (0.011 mL, 0.089 mmol, 300 mol%) in anhydrous *iso*-propanol (0.18 mL), *tert*-butyl hydroperoxide (5.5 M in decane, 0.011 mL, 0.060 mmol, 200 mol%) and a degassed solution of $Mn(dpm)_3$[6] (0.0089 g, 0.015 mmol, 50 mol%) in *iso*-propanol (0.59 mL). The resulting mixture was degassed by bubbling with argon for 10 seconds, and was allowed to stir at ambient temperature for 5 hours. The solvent was removed under reduced pressure and the residue was submitted to flash column chromatography on silica gel (DCM/MeOH = 50:1). The title compound was obtained as a white solid (0.0055 g, 0.021 mmol) in 71% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.73 (dt, *J* = 16.9, 2.8 Hz, 1H), 4.64 (ddd, *J* = 16.9, 3.7, 1.6 Hz, 1H), 4.23 (d, *J* = 11.2 Hz, 1H), 3.52 (dd, *J* = 11.3, 4.8 Hz, 1H), 3.41 (d, *J* = 11.2 Hz, 1H), 2.49-2.36 (m, 1H), 2.15 (ddddd, *J* = 17.8, 11.0, 6.7, 3.7, 2.8 Hz, 1H), 2.06-1.85 (m, 3H), 1.72 (dt, *J* = 13.0, 3.5 Hz, 1H), 1.58-1.43 (m, 2H), 1.34-1.31 (m, 1H), 1.31 (s, 3H), 1.14 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 169.1, 123.8, 80.1, 68.1, 63.8, 51.0, 42.9, 36.0, 33.8, 27.5, 22.5, 21.7, 21.5, 18.1.

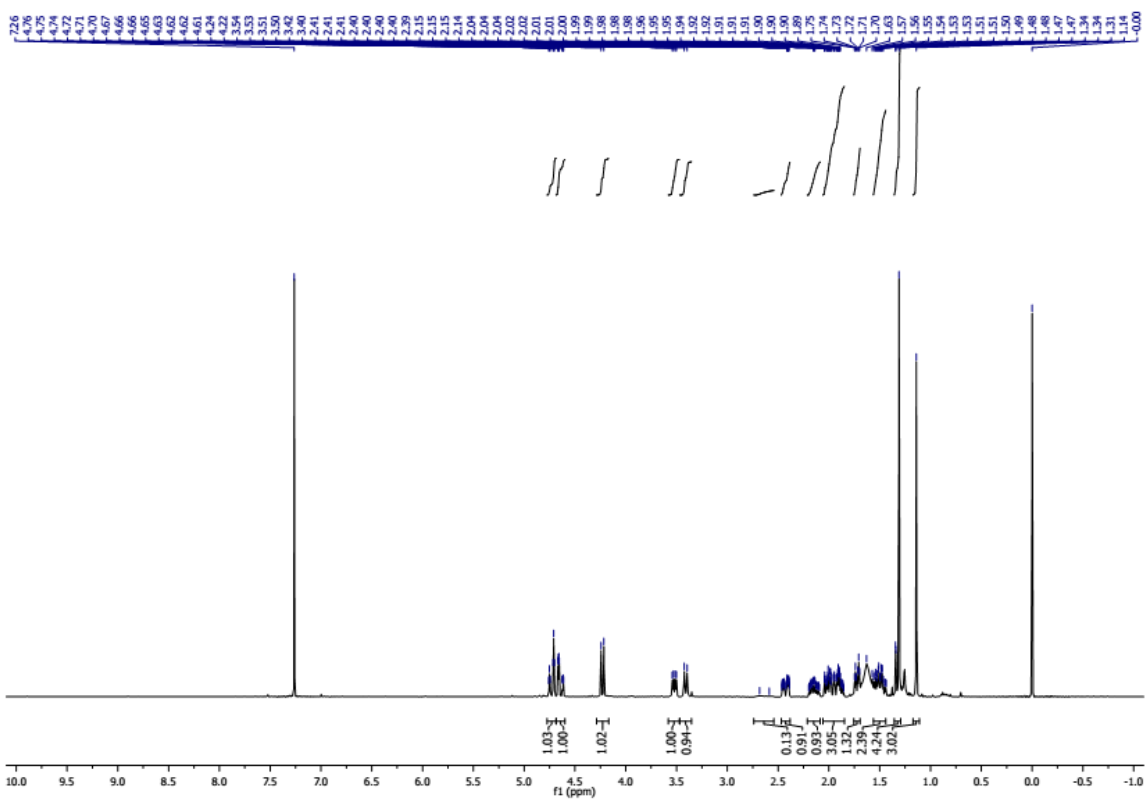
R_f 0.23 (DCM/EA = 1:1, *p*-anisaldehyde)

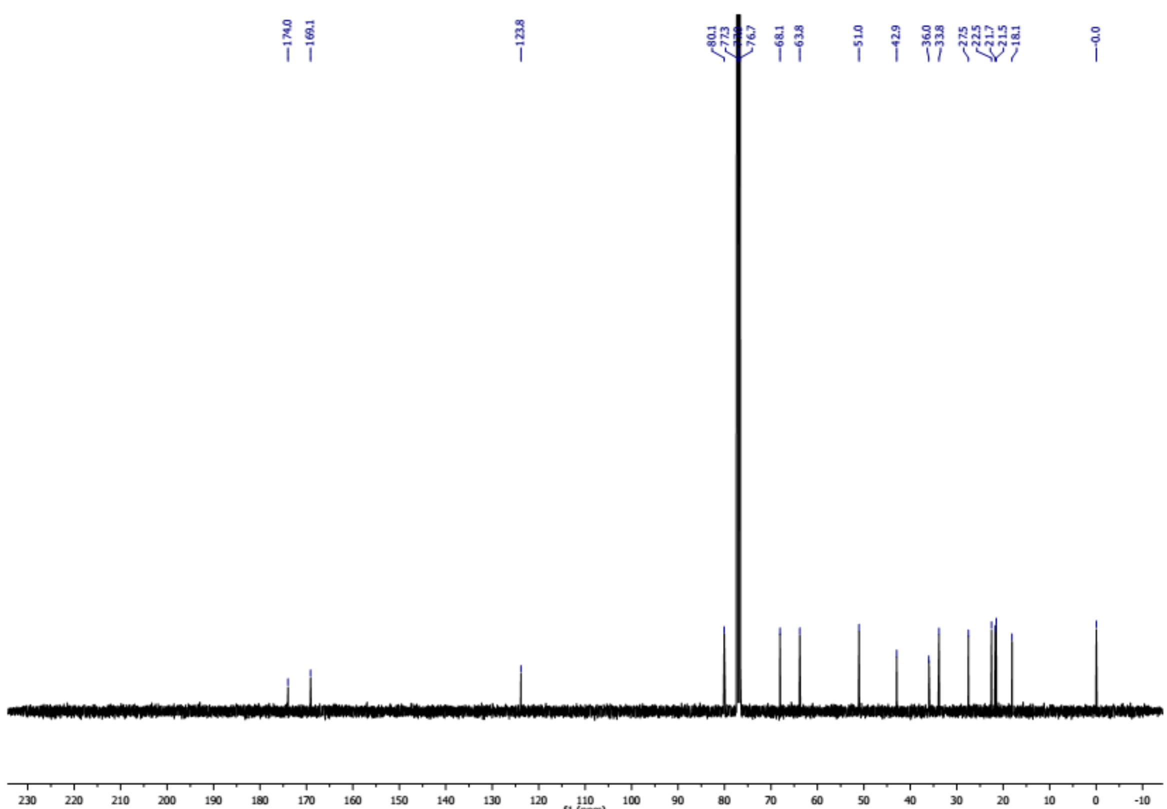
HRMS (ESI) Calcd. for $C_{15}H_{22}O_4$ $[M+Na]^+$: 289.1410, Found: 289.1413.

FTIR (neat): 3331, 2925, 2853, 1739, 1558, 1456, 1436, 1404, 1386, 1289, 1258, 1202, 1193, 1083, 1039, 1012, 991, 799, 731 cm^{-1}.

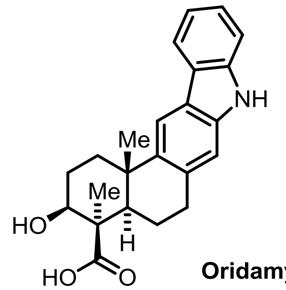
MP 190.4-194.3 ˚C ($CHCl_3$)

Optical Rotation $[\alpha]_D^{30}$ = -37.0˚ (*c* = 0.18, $CHCl_3$)





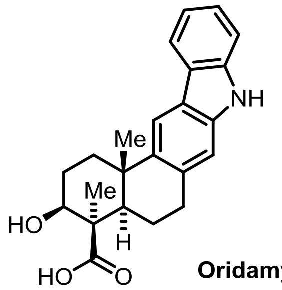
Comparison of NMR Data between Natural and Synthetic Oridamycin A, Triptoquinone C and Isoiresin



Oridamycin A

Table S1. ^{1}H NMR data of natural and synthetic oridamycin A

Natural[7]		Synthetic 1[8]		Synthetic 2[9]		Synthetic (This Work)	
δ_H (ppm)	multiplicity, *J*(Hz), integration	δ_H (ppm)	multiplicity, *J*(Hz), integration	δ_H (ppm)	multiplicity, *J*(Hz), integration	δ_H (ppm)	multiplicity, *J*(Hz), integration
7.93	d, 8.1, 1H	7.96	d, 8.0, 1 H	7.96	d, 8.1, 1H	7.96	dt, 8.1, 1.0, 1H
7.93	s, 1H	7.95	s, 1H	7.95	s, 1H	7.96	s, 1H
7.32	d, 8.1, 1H	7.34	d, 8.0, 1H	7.34	d, 8.1, 1H	7.34	dt, 8.1, 0.9, 1H
7.25	td, 8.1, 1.4, 1H	7.28	t, 7.6, 1H	7.28	t, 7.6, 1H	7.28	ddd, 8.1, 7.1, 1.2, 1H
7.05	td, 8.1, 1.4, 1H	7.08	t, 7.6, 1H	7.08	t, 7.6, 1H	7.08	ddd, 8.1, 7.1, 1.2, 1H
7.03	s, 1H	7.05	s, 1H	7.05	s, 1H	7.06	s, 1H
3.22	dd, 12.2, 4.6, 1H	3.24	dd, 12.1, 4.3, 1H	3.24	dd, 12.2, 4.6, 1H	3.25	dd, 12.2, 4.4, 1H
3.06	ddd, 16.3, 5.4, 2.3, 1H	3.08	dd, 16.5, 4.0, 1H	3.07	ddd, 16.9, 5.5, 1.9, 1H	3.08	ddd, 17.2, 5.6, 1.9, 1H
2.94	ddd, 16.7, 12.7, 2.3, 1H	3.01-2.89	m, 1H	3.00-2.92	m, 1H	2.97	dddd, 16.5, 12.7, 6.2, 1.3, 1H
2.57	dt, 13.6, 3.6, 1H	2.59	dd, 13.2, 2.0, 1H	2.58	dt, 13.2, 3.6, 1H	2.60	dt, 13.2, 3.7, 1H
2.30	qd, 13.6, 3.6, 1H	2.38-2.26	m, 1H	2.32	dq, 13.6, 3.6, 1H	2.32	qd, 13.3, 3.8, 1H
2.23	m, 1H	2.25-2.19	m, 1H	2.26-2.19	m, 1H	2.28-2.19	m, 1H
2.09	td, 12.7, 5.4, 1H	2.19-2.06	m, 1H	2.13	dt, 12.9, 5.4, 1H	2.20-2.07	m, 1H
1.90	dq, 13.6, 3.6, 1H	1.97-1.89	m, 1H	1.96-1.90	m, 1H	1.93	dq, 13.1, 3.9, 1H
1.58	td, 13.6, 4.1, 1H	1.59	ddd, 13.6, 13.6, 3.5, 1H	1.59	dt, 13.6, 3.9, 1H	1.61	td, 13.5, 3.9, 1H
1.51	dd, 12.2, 2.3, 1H	1.51	d, 12.9, 1H	1.52	d, 12.3, 1H	1.52	dd, 12.2, 2.1, 1H
1.48	s, 3H	1.49	s, 3H	1.49	s, 3H	1.50	s, 3H
1.26	s, 3H	1.27	s, 3H	1.27	s, 3H	1.28	s, 3H

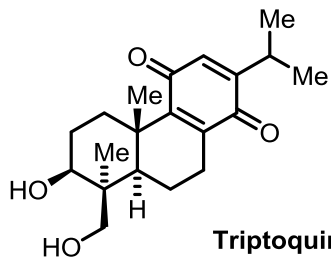


Oridamycin A

Table S2. ^{13}C NMR data of natural and synthetic oridamycin A

Natural[7] δ_C (ppm)	**Synthetic 1**[8] δ_C (ppm)	**Synthetic 2**[9] δ_C (ppm)	**Synthetic (This Work)** δ_C (ppm)
181.0	181.0	181.0	181.0
142.0	142.1	142.0	142.1
140.3	140.4	140.3	140.4
140.1	140.1	140.1	140.1
134.5	134.5	134.5	134.5
126.1	126.1	126.1	126.1
124.6	124.6	124.6	124.6
123.2	123.3	123.2	123.3
120.6	120.6	120.6	120.6
119.3	119.4	119.3	119.3
117.5	117.5	117.5	117.5
111.4	111.4	111.4	111.4
110.7	110.7	110.7	110.7
79.1	79.1	79.1	79.1
54.1	54.1	54.1	54.1
48.7*	49.8	48.7	49.8
40.0	40.0	40.0	40.0
39.6	39.6	39.6	39.6
34.0	34.0	34.0	34.0
30.3	30.3	30.3	30.3
24.8	24.8	24.8	24.8
24.6	24.6	24.6	24.6
22.5	22.5	22.5	22.5

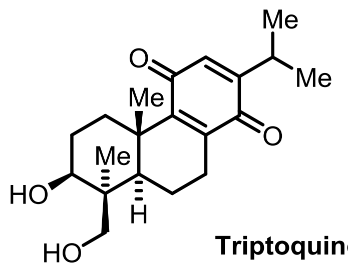
***** The peak at 48.7 ppm is buried under the solvent peaks, and there is no other evidence (such as HMBC data) to support this assignment. On the other hand, the peak at 49.8 ppm can be seen in ^{13}C NMR spectra of all three synthetic oridamycin A, but the author of synthetic 2 did not assign this peak to any carbon.



Triptoquinone C

Table S3. ^{1}H NMR data of natural and synthetic triptoquinone C

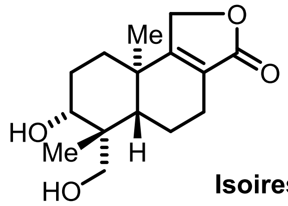
Natural[10]		**Synthetic**	
δ_H (ppm)	multiplicity, *J*(Hz), integration	δ_H (ppm)	multiplicity, *J*(Hz), integration
6.32	d, 1.0, 1H	6.32	d, 1.3, 1H
4.26	ABq, 11.2, 1H	4.26	d, 11.2, 1H
3.48	dd, 11.7, 4.9, 1H	3.49	d, 11.8, 1H
3.35	ABq, 11.2, 1H	3.35	t, 9.6, 1H
2.98	heptd, 6.8, 1.0, 1H	2.98	heptd, 6.9, 1.2, 1H
2.81	ddd, 13.7, 3.9, 3.9, 1H	2.81	dt, 13.6, 3.6, 1H
2.73	dd, 20.5, 4.9, 1H	2.73	ddd, 20.3, 5.7, 1.2, 1H
2.31	ddd, 20.5, 11.7, 7.3, 1H	2.31	ddd, 20.3, 11.6, 7.3, 1H
2.01-1.91	m, 2H	2.03-1.90	m, 2H
1.81	ddd, 13.7, 8.3, 3.9, 1H	1.81	dq, 13.5, 3.9, 1H
1.38	m, 1H	1.39	ddd, 25.0, 12.5, 5.6, 1H
1.29	s, 3H	1.29	s, 3H
1.25	m, 1H	1.28-1.19	m, 1H
1.23	s, 3H	1.23	s, 3H
1.19	d, 11.7, 1H	1.19	dd, 12.5, 1.5, 1H
1.10	d, 6.8, 3H	1.10	d, 6.8, 3H
1.09	d, 6.8, 3H	1.09	d, 6.9, 3H



Triptoquinone C

Table S4. ^{13}C NMR data of natural and synthetic triptoquinone C

Natural[10] δ_C (ppm)	**Synthetic** δ_C (ppm)
187.8	187.9
187.7	187.8
153.1	153.2
149.6	149.7
142.7	142.8
131.9	132.1
80.0	80.3
64.0	64.1
51.8	51.9
43.1	43.3
37.8	38.0
34.2	34.3
28.1	28.3
26.4	26.6
26.3	26.5
22.3	22.8
21.3	21.5
21.3	21.5
20.9	21.0
17.4	17.5



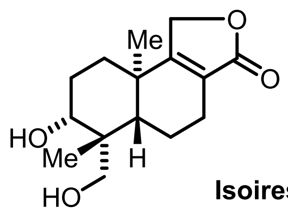
Isoiresin

Table S5. ^{1}H NMR data of synthetic isoiresin

Synthetic 1[11]		**Synthetic (This Work)**	
δ_H (ppm)	multiplicity, *J*(Hz), integration	δ_H (ppm)	multiplicity, *J*(Hz), integration
4.76-4.62	m, 2H	4.73	dt, 16.9, 2.8, 1H
		4.64	ddd, 16.9, 3.7, 1.6, 1H
4.23	dd, 11.2, 2.0, 1H	4.23	d, 11.2, 1H
3.54-3.49	m, 1H	3.52	dd, 11.3, 4.8, 1H
3.41	t, 9.6, 1H	3.41	d, 11.2, 1H
2.72*	dd, 8.8, 2.4, 1H	2.68*	br
2.61*	d, 4.0, 1H	2.59*	br
2.43	dt, 18.0, 2.8, 1H	2.49-2.36	m, 1H
2.17-2.10	m, 1H	2.15	ddddd, 17.8, 11.0, 6.7, 3.7, 2.8, 1H
2.03-1.86	m, 3H	2.06-1.85	m, 3H
1.73	ddd, 12.8, 3.2, 3.2, 1H	1.72	dt, 13.0, 3.5, 1H
1.53-1.45	m, 2H	1.58-1.43	m, 2H
		1.34-1.31#	m, 1H
1.31	s, 3H	1.31	s, 3H
1.14	s, 3H	1.14	s, 3H

***** Alcoholic protons

The authors in ref. 11 only identified 21 out of 22 protons in isoiresin. They did not indicate this proton, but it was seen in the spectrum they provided.



Isoiresin

Table S6. ^{13}C NMR data of synthetic isoiresin

Synthetic 1[11]	**Synthetic (This Work)**
δ_C (ppm)	δ_C (ppm)
174.0	174.0
169.2	169.1
123.8	123.8
80.1	80.1
68.1	68.1
63.8	63.8
51.0	51.0
42.9	42.9
36.0	36.0
33.8	33.8
27.5	27.5
22.6	22.5
21.7	21.7
21.5	21.5
18.1	18.1

Crystallographic Material for 5

X-ray Experimental for $C_{17}H_{15}NO_2$ (5)

Crystals grew as thin, colorless plates by slow evaporation from chloroform. The data crystal was cut from a larger crystal and had approximate dimensions: 0.29 x 0.20 x 0.03 mm. The data were collected on an Agilent Technologies SuperNova Dual Source diffractometer using a μ-focus Cu Kα radiation source (λ = 1.5418 Å) with collimating mirror monochromators. A total of 990 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 10 seconds per frame with a detector offset of +/- 40.6° and 40 seconds per frame with a detector offset of +/- 108.3°. The data were collected at 100 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using Agilent Technologies CrysAlisPro V 1.171.37.31.[12] The structure was solved by direct methods using SuperFlip[13] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-2013.[14] Structure analysis was aided by use of the programs PLATON98[15] and WinGX.[16] The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

For six of the eight molecules in the unit cell, the carbazole moiety was disordered by rotation about the bond from the carbazole to a methylene carbon atom. The rings were disordered to differing degrees but were all modeled in the same fashion. The site occupancy factors for one component of the disordered carbazole were assigned to the variable x. The site occupancy factors for the alternate component were set to (1-x). A common isotropic displacement parameter was refined for the non-H atoms of both components while refining x. The geometry of the two components were restrained to be equivalent throughout the refinement process. The non-H atoms of the two components were refined anisotropically with their displacement parameters restrained to be approximately isotropic.

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.01064*P)^2 + (1.475*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_W(F^2)$ refined to 0.212, with R(F) equal to 0.0786 and a goodness of fit, S, = 1.07. Definitions used for calculating R(F), $R_W(F^2)$ and the goodness of fit, S, are given below.[17] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[18] All figures were generated using SHELXTL/PC.[19] Tables of positional and thermal parameters, bond lengths and angles, torsion angles and figures are found elsewhere.

Table S7. Crystal data and structure refinement for **5**.

Empirical formula	$C_{23}H_{27}NO_2$	
Formula weight	349.45	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	triclinic	
Space group	P 1	
Unit cell dimensions	a = 11.7783(4) Å	α = 79.911(3)°.
	b = 14.8717(5) Å	β = 84.025(3)°.
	c = 21.5048(11) Å	γ = 89.908(3)°.
Volume	3687.9(3) $Å^3$	
Z	8	
Density (calculated)	1.259 Mg/m^3	
Absorption coefficient	0.622 mm^{-1}	
F(000)	1504	
Crystal size	0.29 x 0.20 x 0.03 mm	
Theta range for data collection	3.019 to 74.573°.	
Index ranges	-14<=h<=14, -18<=k<=16, -26<=l<=26	
Reflections collected	22860	
Independent reflections	22860 [R(int) = ?]	
Completeness to theta = 67.684°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00 and 0.851	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	22860 / 7267 / 2566	
Goodness-of-fit on F^2	1.068	
Final R indices [I>2sigma(I)]	R1 = 0.0786, wR2 = 0.2037	
R indices (all data)	R1 = 0.0843, wR2 = 0.2115	
Absolute structure parameter	0.10(16)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.658 and -0.502 e.$Å^{-3}$	

Table S8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O1	-1733(6)	9117(4)	8745(4)	45(1)
O2	-390(5)	11392(4)	8522(4)	40(1)
N1	2564(8)	9402(5)	4899(5)	32(2)
C12	872(13)	8579(6)	6500(18)	35(2)
C13	-12(12)	8243(6)	6245(7)	34(2)
C14	-96(10)	8268(6)	5587(6)	28(2)
C15	838(10)	8682(5)	5168(7)	24(2)
C16	1770(11)	9039(6)	5397(7)	27(2)
C17	1849(11)	9011(6)	6068(8)	31(2)
C18	1031(11)	8817(5)	4498(7)	26(2)
C19	521(10)	8654(6)	3983(8)	24(2)
C20	984(13)	8894(7)	3369(7)	28(2)
C21	2057(14)	9342(7)	3234(7)	33(2)
C22	2625(11)	9527(6)	3738(10)	30(2)
C23	2161(14)	9289(7)	4328(8)	31(2)
N1D	56(12)	8347(7)	4923(8)	29(2)
C12D	849(19)	8480(8)	6500(30)	35(2)
C13D	1884(15)	8918(8)	6388(12)	32(3)
C14D	2231(16)	9141(9)	5732(11)	28(3)
C15D	1620(20)	8951(10)	5248(12)	28(2)
C16D	552(19)	8486(9)	5448(12)	27(2)
C17D	110(20)	8230(10)	6060(12)	29(3)
C18D	1800(18)	9107(9)	4558(12)	28(2)
C19D	2628(19)	9503(10)	4102(14)	28(3)
C20D	2500(20)	9528(11)	3458(14)	30(3)
C21D	1510(30)	9147(13)	3283(11)	30(3)
C22D	669(18)	8747(10)	3709(15)	26(3)
C23D	785(18)	8717(9)	4348(13)	26(2)
C1	73(7)	9770(6)	8596(4)	32(2)

C2	-927(7)	9471(5)	9101(5)	37(2)
C3	-697(10)	8624(6)	9581(5)	50(2)
C4	-737(9)	7853(6)	9196(5)	46(2)
C5	-991(8)	8394(6)	8550(5)	43(2)
C6	123(8)	8914(6)	8239(5)	38(2)
C7	1203(10)	9924(9)	8842(6)	54(3)
C8	-258(7)	10652(5)	8171(5)	37(2)
C9	-1658(12)	7861(7)	8164(6)	66(3)
C10	278(9)	9145(7)	7512(5)	46(2)
C11	835(14)	8373(10)	7198(6)	74(3)
O3	4270(5)	5589(4)	8978(3)	31(1)
O4	1658(5)	6833(4)	8546(4)	41(1)
N2	4939(6)	8319(5)	5127(4)	34(1)
C24	3637(6)	7066(5)	8771(4)	28(1)
C25	3614(6)	6220(5)	9302(4)	28(1)
C26	4328(7)	6332(6)	9842(4)	37(2)
C27	5542(7)	6253(6)	9521(4)	37(2)
C28	5334(6)	6144(5)	8839(4)	29(1)
C29	4922(6)	7072(5)	8493(4)	25(1)
C30	3280(7)	7940(5)	9005(4)	37(2)
C31	2840(6)	6875(5)	8289(4)	32(1)
C32	6216(7)	5617(6)	8500(5)	39(2)
C33	5197(7)	7262(5)	7772(4)	29(1)
C34	4996(8)	8258(5)	7470(4)	33(2)
C35	5473(7)	8487(5)	6782(4)	30(1)
C36	6560(7)	8904(5)	6615(4)	32(2)
C37	7073(7)	9113(5)	6004(4)	29(1)
C38	6464(6)	8869(5)	5528(4)	28(1)
C39	5381(7)	8458(5)	5680(4)	30(2)
C40	4882(8)	8241(5)	6314(4)	32(2)
C41	6698(7)	9015(5)	4844(4)	28(1)
C42	7603(7)	9388(5)	4394(4)	31(2)
C43	7528(8)	9425(6)	3767(5)	37(2)
C44	6553(9)	9083(6)	3550(5)	39(2)
C45	5656(8)	8697(6)	3985(5)	36(2)

C46	5736(6)	8651(5)	4612(4)	26(1)
O5	1132(6)	4989(4)	8938(3)	32(1)
O6	3923(5)	3980(4)	8520(4)	40(1)
N3	2512(7)	4353(5)	5097(5)	30(2)
C58	914(11)	3448(6)	6664(12)	32(2)
C59	-31(10)	3079(6)	6432(6)	33(2)
C60	-104(9)	3143(5)	5792(6)	32(2)
C61	818(9)	3598(5)	5356(6)	27(2)
C62	1721(9)	3946(5)	5602(6)	28(2)
C63	1828(11)	3893(6)	6259(7)	32(2)
C64	1013(11)	3776(5)	4684(7)	30(2)
C65	482(10)	3621(6)	4170(8)	31(2)
C66	930(13)	3898(7)	3559(7)	32(2)
C67	1985(12)	4368(7)	3406(7)	31(2)
C68	2566(12)	4544(6)	3922(7)	31(2)
C69	2108(11)	4261(6)	4544(6)	31(2)
N1E	26(14)	3312(8)	5079(10)	31(2)
C12E	1060(20)	3478(9)	6610(20)	32(2)
C13E	2127(17)	3934(9)	6371(13)	29(3)
C14E	2395(18)	4141(10)	5738(11)	30(3)
C15E	1681(19)	3932(9)	5279(14)	28(2)
C16E	610(20)	3471(11)	5541(14)	29(2)
C17E	280(20)	3227(12)	6239(14)	32(3)
C18E	1700(20)	4046(11)	4606(13)	30(2)
C19E	2500(20)	4436(13)	4134(15)	28(3)
C20E	2280(20)	4446(14)	3505(16)	29(3)
C21E	1200(20)	4044(13)	3366(15)	30(3)
C22E	510(20)	3694(12)	3848(18)	30(3)
C23E	700(20)	3671(10)	4474(17)	30(2)
C47	1870(7)	3630(5)	8701(4)	33(1)
C48	1685(6)	4174(5)	9243(4)	29(1)
C49	752(7)	3763(6)	9770(4)	35(2)
C50	-340(7)	4020(6)	9447(4)	36(2)
C51	146(7)	4523(5)	8784(4)	31(2)
C52	692(7)	3791(5)	8410(4)	31(1)

C53	2117(8)	2617(6)	8915(5)	41(2)
C54	2865(6)	4074(5)	8239(4)	32(1)
C55	-616(7)	5230(6)	8454(4)	37(2)
C56	714(6)	4018(5)	7696(4)	29(1)
C57	984(8)	3200(5)	7358(4)	35(2)
O7	-2743(5)	11528(4)	8856(3)	31(1)
O8	-3998(6)	9407(4)	8605(4)	50(2)
N4	-5044(8)	13320(5)	4988(5)	27(2)
C81	-4370(14)	13384(7)	6610(15)	34(2)
C82	-3305(11)	13827(6)	6453(8)	36(2)
C83	-2824(10)	14097(6)	5827(6)	30(2)
C84	-3478(11)	13898(5)	5336(7)	25(2)
C85	-4556(11)	13446(6)	5525(7)	24(2)
C86	-5006(14)	13189(7)	6151(8)	32(2)
C87	-3316(12)	14047(6)	4667(7)	26(2)
C88	-2454(11)	14446(6)	4220(9)	27(2)
C89	-2551(12)	14488(6)	3575(9)	33(2)
C90	-3534(12)	14122(7)	3376(7)	32(2)
C91	-4397(11)	13723(6)	3817(8)	29(2)
C92	-4295(11)	13685(5)	4450(7)	26(2)
N1B	-2496(11)	14291(6)	5046(7)	26(2)
C12B	-2917(15)	14419(7)	3356(10)	30(3)
C13B	-4026(16)	13995(8)	3470(11)	32(3)
C14B	-4518(17)	13713(8)	4080(13)	29(3)
C15B	-3988(19)	13821(8)	4594(10)	26(2)
C16B	-2850(20)	14257(10)	4478(12)	26(2)
C17B	-2364(16)	14536(8)	3865(14)	28(3)
C18B	-4228(15)	13624(7)	5253(10)	26(2)
C19B	-5192(15)	13208(9)	5647(9)	28(2)
C20B	-5150(20)	13115(11)	6298(13)	32(3)
C21B	-4200(20)	13413(12)	6590(20)	34(2)
C22B	-3243(18)	13827(10)	6194(12)	30(3)
C23B	-3366(18)	13889(8)	5552(11)	26(2)
C70	-4498(7)	10981(6)	8674(4)	34(2)
C71	-3706(6)	10998(5)	9198(4)	33(1)

C72	-4135(8)	11591(6)	9686(4)	41(2)
C73	-3865(8)	12560(6)	9301(4)	40(2)
C74	-3375(7)	12347(5)	8659(4)	33(1)
C75	-4358(7)	12019(6)	8328(4)	36(2)
C76	-5748(9)	10712(8)	8908(7)	58(3)
C77	-4008(8)	10319(6)	8255(5)	42(2)
C78	-2551(9)	13085(6)	8284(5)	48(2)
C79	-4224(9)	12180(6)	7607(5)	43(2)
C80	-4555(12)	13147(8)	7309(5)	68(3)
O9	6692(5)	8488(4)	1278(3)	34(1)
O10	5335(6)	10634(4)	1493(4)	42(1)
C93	4877(7)	9035(5)	1431(4)	32(2)
C94	5880(7)	9013(6)	913(4)	36(2)
C95	5620(9)	8391(6)	445(4)	45(2)
C96	5761(9)	7426(6)	845(4)	43(2)
C97	5990(7)	7656(5)	1489(4)	33(1)
C98	4865(7)	8023(5)	1795(4)	32(1)
C99	3734(9)	9331(7)	1191(6)	50(2)
C100	5211(7)	9726(5)	1846(4)	36(2)
C101	6675(8)	6946(6)	1879(5)	45(2)
C102	4703(7)	7884(5)	2513(4)	33(2)
C103	4276(10)	6911(7)	2827(5)	52(2)
N5	2468(8)	6882(5)	5070(5)	30(2)
C104	4115(12)	6776(6)	3551(9)	29(2)
C105	5083(12)	6278(7)	3800(7)	33(2)
C106	5104(11)	6028(6)	4437(6)	27(2)
C107	4203(11)	6248(5)	4844(7)	26(2)
C108	3289(11)	6727(6)	4583(7)	28(2)
C109	3211(12)	6996(7)	3958(7)	29(2)
C110	3895(15)	6108(7)	5541(7)	29(2)
C111	4484(13)	5673(7)	6038(8)	33(2)
C112	3929(13)	5662(8)	6670(9)	35(2)
C113	2846(13)	6081(7)	6754(7)	32(2)
C114	2308(13)	6495(7)	6260(8)	31(2)
C115	2832(13)	6508(6)	5655(7)	29(2)

N1A	5062(13)	5832(7)	5114(8)	30(2)
C12A	4203(18)	6680(8)	3484(15)	29(2)
C13A	3107(17)	7013(8)	3769(11)	28(3)
C14A	2711(17)	6961(9)	4414(10)	30(3)
C15A	3450(18)	6546(9)	4828(12)	28(2)
C16A	4550(20)	6201(9)	4578(12)	27(2)
C17A	4870(20)	6272(11)	3953(12)	28(3)
C18A	3360(30)	6365(11)	5503(11)	30(2)
C19A	2500(20)	6542(10)	5993(15)	31(3)
C20A	2700(20)	6288(12)	6552(14)	36(3)
C21A	3680(20)	5846(12)	6748(14)	36(3)
C22A	4590(20)	5646(11)	6280(12)	33(3)
C23A	4390(20)	5925(10)	5645(12)	30(2)
O11	646(5)	5074(4)	1126(3)	28(1)
O12	3281(5)	6020(4)	1581(3)	38(1)
N6	59(7)	5838(6)	5038(4)	40(2)
C116	1312(6)	6413(5)	1375(4)	30(1)
C117	1343(7)	5869(5)	823(4)	30(1)
C118	607(7)	6302(6)	303(4)	35(2)
C119	-626(7)	6052(6)	613(4)	33(1)
C120	-386(7)	5569(5)	1281(4)	30(1)
C121	12(6)	6287(5)	1652(4)	26(1)
C122	1668(8)	7428(6)	1160(5)	44(2)
C123	2121(6)	5935(5)	1845(4)	30(1)
C124	-1280(7)	4860(5)	1611(4)	33(2)
C125	-269(6)	6077(5)	2371(4)	27(1)
C126	-58(7)	6890(5)	2693(4)	35(2)
C127	-507(7)	6743(5)	3377(4)	30(1)
C128	-1597(8)	7084(6)	3555(5)	38(2)
C129	-2042(8)	7001(6)	4163(5)	36(2)
C130	-1483(7)	6585(5)	4644(5)	34(2)
C131	-375(8)	6212(5)	4496(5)	36(2)
C132	111(8)	6288(5)	3858(4)	34(2)
C133	-1682(8)	6396(6)	5330(5)	38(2)
C134	-2581(9)	6583(6)	5783(6)	46(2)

C135	-2479(10)	6320(7)	6408(5)	52(2)
C136	-1504(10)	5851(8)	6615(5)	53(2)
C137	-597(9)	5660(7)	6178(5)	46(2)
C138	-712(9)	5954(6)	5555(5)	42(2)
O13	3832(5)	4395(4)	1124(3)	29(1)
O14	1027(5)	3239(4)	1558(4)	42(1)
N7	2458(7)	1851(4)	5026(5)	34(2)
C150	4027(10)	1727(5)	3448(10)	30(1)
C151	4994(9)	1251(5)	3702(6)	32(2)
C152	5069(9)	1018(5)	4337(6)	31(2)
C153	4199(9)	1234(4)	4757(6)	29(2)
C154	3211(9)	1713(5)	4517(6)	29(2)
C155	3153(10)	1935(5)	3898(6)	31(2)
C156	3970(12)	1098(5)	5457(6)	28(2)
C157	4567(10)	690(5)	5951(6)	30(2)
C158	4054(11)	690(6)	6586(6)	34(2)
C159	3002(13)	1085(7)	6690(7)	37(2)
C160	2413(10)	1484(6)	6207(8)	34(2)
C161	2903(12)	1495(5)	5567(7)	31(2)
N1F	5050(20)	850(11)	4990(13)	32(3)
C12F	3890(20)	1684(10)	3430(30)	30(1)
C13F	2970(20)	1929(10)	3751(17)	28(3)
C14F	2430(30)	1956(13)	4374(16)	34(3)
C15F	3360(30)	1526(13)	4730(20)	30(2)
C16F	4440(30)	1208(13)	4472(19)	30(3)
C17F	4690(30)	1293(15)	3820(20)	31(3)
C18F	3420(40)	1321(15)	5425(17)	29(2)
C19F	2590(30)	1504(14)	5860(30)	30(3)
C20F	2680(30)	1302(19)	6480(20)	32(3)
C21F	3600(40)	929(17)	6653(17)	33(3)
C22F	4610(30)	690(15)	6190(20)	31(3)
C23F	4370(30)	942(15)	5560(20)	30(3)
C139	3078(6)	2925(5)	1394(4)	30(1)
C140	3267(7)	3748(5)	833(4)	31(1)
C141	4171(7)	3562(6)	314(4)	37(2)

C142	5276(7)	3643(6)	624(4)	35(2)
C143	4813(7)	3834(5)	1286(4)	30(1)
C144	4265(6)	2935(5)	1681(4)	28(1)
C145	2809(7)	2028(6)	1191(5)	42(2)
C146	2080(7)	3177(6)	1842(4)	36(2)
C147	5609(7)	4367(6)	1591(5)	38(2)
C148	4241(7)	2814(5)	2396(4)	32(1)
C149	3944(8)	1822(6)	2746(5)	40(2)
O15	-2333(5)	859(4)	1120(3)	34(1)
O16	-977(6)	-1378(4)	1319(4)	54(2)
N8	44(8)	864(7)	4930(5)	29(2)
C173	-584(15)	1618(17)	3261(11)	28(2)
C174	-1676(10)	1996(8)	3421(7)	30(2)
C175	-2077(11)	1977(8)	4029(7)	30(2)
C176	-1505(12)	1577(9)	4536(7)	28(2)
C177	-386(12)	1199(8)	4360(7)	27(2)
C178	20(14)	1195(11)	3758(7)	29(2)
C179	-1675(11)	1426(8)	5190(7)	26(2)
C180	-2620(11)	1615(8)	5624(6)	29(2)
C181	-2505(12)	1377(10)	6271(7)	32(2)
C182	-1562(13)	953(10)	6493(6)	34(2)
C183	-618(13)	743(9)	6085(8)	34(2)
C184	-711(12)	1005(10)	5436(7)	31(2)
N1C	-2480(16)	1831(12)	4839(9)	32(2)
C12C	-760(30)	1660(40)	3310(20)	28(2)
C13C	200(30)	1260(20)	3618(14)	29(3)
C14C	210(20)	1059(14)	4218(11)	27(3)
C15C	-700(20)	1295(16)	4594(13)	30(2)
C16C	-1690(20)	1746(18)	4364(14)	28(2)
C17C	-1720(20)	1902(16)	3723(16)	28(3)
C18C	-970(30)	1163(18)	5308(13)	29(2)
C19C	-370(20)	833(16)	5821(15)	31(3)
C20C	-870(20)	849(17)	6444(13)	36(3)
C21C	-1950(30)	1224(19)	6523(13)	34(3)
C22C	-2560(20)	1545(18)	6042(16)	31(3)

C23C	-2070(30)	1514(16)	5392(14)	29(2)
C162	-536(7)	255(6)	1225(4)	36(2)
C163	-1384(7)	529(5)	742(4)	36(2)
C164	-1024(8)	1379(6)	249(4)	43(2)
C165	-1291(9)	2147(6)	641(4)	46(2)
C166	-1683(8)	1590(5)	1294(4)	39(2)
C167	-642(7)	1090(5)	1582(4)	37(2)
C168	668(9)	118(9)	955(7)	61(3)
C169	-950(8)	-639(6)	1667(5)	48(2)
C170	-2506(10)	2114(7)	1703(5)	55(2)
C171	-723(8)	888(6)	2305(4)	41(2)
C172	-260(10)	1656(7)	2589(5)	57(2)

Table S9. Bond lengths [Å] and angles [°] for **5**.

O1-C2	1.435(9)	C12D-C13D	1.36(3)
O1-C5	1.475(11)	C12D-C17D	1.44(6)
O2-C8	1.439(9)	C12D-C11	1.48(7)
O2-H2O	0.84	C13D-C14D	1.41(3)
N1-C16	1.379(18)	C13D-H13D	0.95
N1-C23	1.399(17)	C14D-C15D	1.39(3)
N1-H1	0.88	C14D-H14D	0.95
C12-C13	1.36(3)	C15D-C16D	1.43(4)
C12-C17	1.47(3)	C15D-C18D	1.45(4)
C12-C11	1.47(4)	C16D-C17D	1.35(4)
C13-C14	1.422(19)	C17D-H17D	0.95
C13-H13	0.95	C18D-C19D	1.36(3)
C14-C15	1.415(17)	C18D-C23D	1.48(3)
C14-H14	0.95	C19D-C20D	1.40(3)
C15-C16	1.391(17)	C19D-H19D	0.95
C15-C18	1.41(2)	C20D-C21D	1.40(4)
C16-C17	1.45(2)	C20D-H20D	0.95
C17-H17	0.95	C21D-C22D	1.34(4)
C18-C19	1.371(19)	C21D-H21D	0.95
C18-C23	1.49(2)	C22D-C23D	1.39(4)
C19-C20	1.362(19)	C22D-H22D	0.95
C19-H19	0.95	C1-C7	1.515(12)
C20-C21	1.408(19)	C1-C2	1.527(11)
C20-H20	0.95	C1-C8	1.537(11)
C21-C22	1.40(2)	C1-C6	1.596(11)
C21-H21	0.95	C2-C3	1.526(12)
C22-C23	1.32(2)	C2-H2	1.00
C22-H22	0.95	C3-C4	1.532(12)
N1D-C16D	1.37(3)	C3-H3A	0.99
N1D-C23D	1.45(3)	C3-H3B	0.99
N1D-H1D	0.88	C4-C5	1.537(12)

C4-H4A	0.99	C27-C28	1.547(11)
C4-H4B	0.99	C27-H27A	0.99
C5-C9	1.518(12)	C27-H27B	0.99
C5-C6	1.552(12)	C28-C32	1.505(11)
C6-C10	1.532(13)	C28-C29	1.551(10)
C6-H6	1.00	C29-C33	1.527(10)
C7-H7A	0.98	C29-H29	1.00
C7-H7B	0.98	C30-H30A	0.98
C7-H7C	0.98	C30-H30B	0.98
C8-H8A	0.99	C30-H30C	0.98
C8-H8B	0.99	C31-H31A	0.99
C9-H9A	0.98	C31-H31B	0.99
C9-H9B	0.98	C32-H32A	0.98
C9-H9C	0.98	C32-H32B	0.98
C10-C11	1.545(14)	C32-H32C	0.98
C10-H10F	0.99	C33-C34	1.539(10)
C10-H10G	0.99	C33-H33A	0.99
C11-H11E	0.99	C33-H33B	0.99
C11-H11F	0.99	C34-C35	1.507(11)
O3-C25	1.441(9)	C34-H34A	0.99
O3-C28	1.476(8)	C34-H34B	0.99
O4-C31	1.441(8)	C35-C40	1.381(11)
O4-H4O	0.84	C35-C36	1.408(11)
N2-C39	1.393(10)	C36-C37	1.370(11)
N2-C46	1.395(10)	C36-H36	0.95
N2-H2N	0.88	C37-C38	1.407(10)
C24-C30	1.519(10)	C37-H37	0.95
C24-C31	1.531(9)	C38-C39	1.397(9)
C24-C25	1.542(10)	C38-C41	1.446(11)
C24-C29	1.567(9)	C39-C40	1.410(12)
C25-C26	1.534(10)	C40-H40	0.95
C25-H25	1.00	C41-C42	1.408(11)
C26-C27	1.535(11)	C41-C46	1.428(10)
C26-H26A	0.99	C42-C43	1.351(12)
C26-H26B	0.99	C42-H42	0.95

C43-C44	1.410(12)	N1E-C23E	1.46(4)
C43-H43	0.95	N1E-H1EN	0.88
C44-C45	1.389(13)	C12E-C17E	1.38(5)
C44-H44	0.95	C12E-C13E	1.43(3)
C45-C46	1.352(12)	C12E-C57	1.58(5)
C45-H45	0.95	C13E-C14E	1.35(4)
O5-C51	1.450(9)	C13E-H13E	0.95
O5-C48	1.459(9)	C14E-C15E	1.43(3)
O6-C54	1.436(9)	C14E-H14E	0.95
O6-H6O	0.84	C15E-C18E	1.43(4)
N3-C69	1.355(15)	C15E-C16E	1.45(4)
N3-C62	1.405(14)	C16E-C17E	1.49(4)
N3-H3N	0.88	C17E-H17E	0.95
C58-C63	1.40(2)	C18E-C19E	1.36(4)
C58-C59	1.42(2)	C18E-C23E	1.39(4)
C58-C57	1.48(3)	C19E-C20E	1.40(4)
C59-C60	1.375(18)	C19E-H19E	0.95
C59-H59	0.95	C20E-C21E	1.48(4)
C60-C61	1.444(16)	C20E-H20E	0.95
C60-H60	0.95	C21E-C22E	1.29(4)
C61-C62	1.376(17)	C21E-H21E	0.95
C61-C64	1.417(18)	C22E-C23E	1.38(4)
C62-C63	1.419(19)	C22E-H22E	0.95
C63-H63	0.95	C47-C54	1.526(9)
C64-C65	1.378(17)	C47-C48	1.528(10)
C64-C69	1.457(18)	C47-C53	1.533(11)
C65-C66	1.36(2)	C47-C52	1.582(10)
C65-H65	0.95	C48-C49	1.538(9)
C66-C67	1.41(2)	C48-H48	1.00
C66-H66	0.95	C49-C50	1.541(11)
C67-C68	1.423(17)	C49-H49A	0.99
C67-H67	0.95	C49-H49B	0.99
C68-C69	1.382(18)	C50-C51	1.541(11)
C68-H68	0.95	C50-H50A	0.99
N1E-C16E	1.32(3)	C50-H50B	0.99

C51-C55	1.511(11)	C87-C88	1.381(18)
C51-C52	1.564(10)	C87-C92	1.428(19)
C52-C56	1.510(11)	C88-C89	1.39(2)
C52-H52	1.00	C88-H88	0.95
C53-H53A	0.98	C89-C90	1.42(2)
C53-H53B	0.98	C89-H89	0.95
C53-H53C	0.98	C90-C91	1.375(19)
C54-H54A	0.99	C90-H90	0.95
C54-H54B	0.99	C91-C92	1.37(2)
C55-H55A	0.98	C91-H91	0.95
C55-H55B	0.98	N1B-C16B	1.34(2)
C55-H55C	0.98	N1B-C23B	1.46(3)
C56-C57	1.540(10)	N1B-H1BN	0.88
C56-H56A	0.99	C12B-C17B	1.37(3)
C56-H56B	0.99	C12B-C13B	1.43(2)
C57-H57A	0.99	C12B-H12B	0.95
C57-H57B	0.99	C13B-C14B	1.37(3)
O7-C71	1.441(8)	C13B-H13B	0.95
O7-C74	1.449(9)	C14B-C15B	1.36(3)
O8-C77	1.432(11)	C14B-H14B	0.95
O8-H8O	0.84	C15B-C18B	1.40(3)
N4-C85	1.384(16)	C15B-C16B	1.47(3)
N4-C92	1.407(16)	C16B-C17B	1.37(3)
N4-H4N	0.88	C17B-H17B	0.95
C81-C86	1.37(3)	C18B-C23B	1.35(3)
C81-C82	1.40(2)	C18B-C19B	1.42(3)
C81-C80	1.48(3)	C19B-C20B	1.39(3)
C82-C83	1.39(2)	C19B-H19B	0.95
C82-H82	0.95	C20B-C21B	1.45(5)
C83-C84	1.441(18)	C20B-H20B	0.95
C83-H83	0.95	C21B-C22B	1.41(4)
C84-C87	1.410(19)	C21B-C80	1.53(5)
C84-C85	1.425(19)	C22B-C23B	1.39(3)
C85-C86	1.38(2)	C22B-H22B	0.95
C86-H86	0.95	C70-C77	1.522(11)

C70-C76	1.535(12)	C93-C98	1.569(10)
C70-C71	1.540(10)	C94-C95	1.536(11)
C70-C75	1.591(11)	C94-H94	1.00
C71-C72	1.532(11)	C95-C96	1.556(12)
C71-H71	1.00	C95-H95A	0.99
C72-C73	1.546(12)	C95-H95B	0.99
C72-H72A	0.99	C96-C97	1.534(11)
C72-H72B	0.99	C96-H96A	0.99
C73-C74	1.527(10)	C96-H96B	0.99
C73-H73A	0.99	C97-C101	1.515(11)
C73-H73B	0.99	C97-C98	1.559(11)
C74-C78	1.525(11)	C98-C102	1.513(11)
C74-C75	1.543(10)	C98-H98	1.00
C75-C79	1.519(11)	C99-H99A	0.98
C75-H75	1.00	C99-H99B	0.98
C76-H76A	0.98	C99-H99C	0.98
C76-H76B	0.98	C100-H10A	0.99
C76-H76C	0.98	C100-H10B	0.99
C77-H77A	0.99	C101-H10C	0.98
C77-H77B	0.99	C101-H10D	0.98
C78-H78A	0.98	C101-H10E	0.98
C78-H78B	0.98	C102-C103	1.547(11)
C78-H78C	0.98	C102-H10H	0.99
C79-C80	1.536(13)	C102-H10I	0.99
C79-H79A	0.99	C103-C12A	1.39(3)
C79-H79B	0.99	C103-C104	1.53(2)
C80-H80A	0.99	C103-H10J	0.99
C80-H80B	0.99	C103-H10K	0.99
O9-C94	1.442(10)	N5-C108	1.398(16)
O9-C97	1.466(8)	N5-C115	1.394(17)
O10-C100	1.429(10)	N5-H5N	0.88
O10-H10O	0.84	C104-C109	1.38(2)
C93-C99	1.528(13)	C104-C105	1.46(2)
C93-C94	1.541(11)	C105-C106	1.36(2)
C93-C100	1.551(10)	C105-H105	0.95

C106-C107	1.381(17)	C21A-C22A	1.45(3)
C106-H106	0.95	C21A-H21A	0.95
C107-C108	1.40(2)	C22A-C23A	1.40(3)
C107-C110	1.48(2)	C22A-H22A	0.95
C108-C109	1.35(2)	O11-C117	1.455(9)
C109-H109	0.95	O11-C120	1.460(10)
C110-C111	1.40(2)	O12-C123	1.421(8)
C110-C115	1.40(2)	O12-H12O	0.84
C111-C112	1.44(2)	N6-C131	1.352(12)
C111-H111	0.95	N6-C138	1.397(12)
C112-C113	1.43(2)	N6-H6N	0.88
C112-H112	0.95	C116-C123	1.543(10)
C113-C114	1.349(19)	C116-C122	1.545(10)
C113-H113	0.95	C116-C117	1.546(10)
C114-C115	1.38(2)	C116-C121	1.582(9)
C114-H114	0.95	C117-C118	1.538(10)
N1A-C23A	1.35(3)	C117-H117	1.00
N1A-C16A	1.38(3)	C118-C119	1.551(10)
N1A-H1AN	0.88	C118-H11A	0.99
C12A-C17A	1.40(4)	C118-H11B	0.99
C12A-C13A	1.49(3)	C119-C120	1.543(11)
C13A-C14A	1.41(3)	C119-H11C	0.99
C13A-H13A	0.95	C119-H11D	0.99
C14A-C15A	1.38(3)	C120-C124	1.519(9)
C14A-H14A	0.95	C120-C121	1.541(9)
C15A-C18A	1.42(3)	C121-C125	1.525(10)
C15A-C16A	1.47(3)	C121-H121	1.00
C16A-C17A	1.35(4)	C122-H12Q	0.98
C17A-H17A	0.95	C122-H12R	0.98
C18A-C23A	1.41(3)	C122-H12S	0.98
C18A-C19A	1.44(4)	C123-H12D	0.99
C19A-C20A	1.24(4)	C123-H12E	0.99
C19A-H19A	0.95	C124-H12F	0.98
C20A-C21A	1.40(4)	C124-H12G	0.98
C20A-H20A	0.95	C124-H12H	0.98

C125-C126	1.529(9)	C150-C149	1.51(2)
C125-H12I	0.99	C151-C152	1.360(17)
C125-H12J	0.99	C151-H151	0.95
C126-C127	1.488(11)	C152-C153	1.370(16)
C126-H12K	0.99	C152-H152	0.95
C126-H12L	0.99	C153-C154	1.457(15)
C127-C132	1.402(11)	C153-C156	1.478(17)
C127-C128	1.420(12)	C154-C155	1.324(17)
C128-C129	1.343(13)	C155-H155	0.95
C128-H128	0.95	C156-C157	1.386(16)
C129-C130	1.343(13)	C156-C161	1.403(17)
C129-H129	0.95	C157-C158	1.435(17)
C130-C131	1.443(13)	C157-H157	0.95
C130-C133	1.448(13)	C158-C159	1.385(18)
C131-C132	1.417(13)	C158-H158	0.95
C132-H132	0.95	C159-C160	1.357(17)
C133-C138	1.404(14)	C159-H159	0.95
C133-C134	1.423(14)	C160-C161	1.434(18)
C134-C135	1.350(15)	C160-H160	0.95
C134-H134	0.95	N1F-C16F	1.41(4)
C135-C136	1.414(17)	N1F-C23F	1.43(5)
C135-H135	0.95	N1F-H1FN	0.88
C136-C137	1.411(16)	C12F-C13F	1.31(5)
C136-H136	0.95	C12F-C17F	1.38(6)
C137-C138	1.356(15)	C12F-C149	1.44(6)
C137-H137	0.95	C13F-C14F	1.43(5)
O13-C140	1.436(8)	C13F-H13F	0.95
O13-C143	1.460(10)	C14F-C15F	1.49(5)
O14-C146	1.434(9)	C14F-H14F	0.95
O14-H14O	0.84	C15F-C16F	1.45(6)
N7-C161	1.349(14)	C15F-C18F	1.47(5)
N7-C154	1.379(14)	C16F-C17F	1.39(6)
N7-H7N	0.88	C17F-H17F	0.95
C150-C155	1.41(2)	C18F-C23F	1.29(5)
C150-C151	1.445(17)	C18F-C19F	1.34(6)

C19F-C20F	1.35(6)	C149-H201	0.99
C19F-H19F	0.95	C149-H202	0.99
C20F-C21F	1.27(6)	O15-C163	1.450(10)
C20F-H20F	0.95	O15-C166	1.454(9)
C21F-C22F	1.56(6)	O16-C169	1.434(12)
C21F-H21F	0.95	O16-H16O	0.84
C22F-C23F	1.38(5)	N8-C184	1.377(18)
C22F-H22F	0.95	N8-C177	1.387(16)
C139-C145	1.518(10)	N8-H8N	0.88
C139-C146	1.529(11)	C173-C178	1.40(3)
C139-C140	1.560(10)	C173-C174	1.44(2)
C139-C144	1.586(9)	C173-C172	1.45(2)
C140-C141	1.521(11)	C174-C175	1.338(17)
C140-H140	1.00	C174-H174	0.95
C141-C142	1.537(10)	C175-C176	1.385(17)
C141-H14T	0.99	C175-H175	0.95
C141-H14X	0.99	C176-C179	1.378(19)
C142-C143	1.545(11)	C176-C177	1.47(2)
C142-H14Y	0.99	C177-C178	1.33(2)
C142-H14Z	0.99	C178-H178	0.95
C143-C147	1.500(9)	C179-C184	1.402(18)
C143-C144	1.554(9)	C179-C180	1.436(16)
C144-C148	1.515(11)	C180-C181	1.395(18)
C144-H144	1.00	C180-H180	0.95
C145-H14Q	0.98	C181-C182	1.36(2)
C145-H14R	0.98	C181-H181	0.95
C145-H14S	0.98	C182-C183	1.41(2)
C146-H14H	0.99	C182-H182	0.95
C146-H14I	0.99	C183-C184	1.40(2)
C147-H14J	0.98	C183-H183	0.95
C147-H14K	0.98	N1C-C16C	1.33(3)
C147-H14L	0.98	N1C-C23C	1.34(3)
C148-C149	1.557(10)	N1C-H1CN	0.88
C148-H14M	0.99	C12C-C13C	1.45(6)
C148-H14N	0.99	C12C-C17C	1.45(5)

C12C-C172	1.60(5)	C163-H163	1.00
C13C-C14C	1.27(4)	C164-C165	1.548(12)
C13C-H13C	0.95	C164-H16A	0.99
C14C-C15C	1.36(3)	C164-H16B	0.99
C14C-H203	0.95	C165-C166	1.527(11)
C15C-C16C	1.43(4)	C165-H16C	0.99
C15C-C18C	1.51(4)	C165-H16D	0.99
C16C-C17C	1.36(4)	C166-C170	1.542(14)
C17C-H17C	0.95	C166-C167	1.559(12)
C18C-C19C	1.38(4)	C167-C171	1.524(11)
C18C-C23C	1.41(4)	C167-H167	1.00
C19C-C20C	1.41(4)	C168-H16E	0.98
C19C-H19C	0.95	C168-H16F	0.98
C20C-C21C	1.40(4)	C168-H16G	0.98
C20C-H20C	0.95	C169-H16H	0.99
C21C-C22C	1.34(4)	C169-H16I	0.99
C21C-H21C	0.95	C170-H17X	0.98
C22C-C23C	1.46(4)	C170-H17Y	0.98
C22C-H22C	0.95	C170-H17Z	0.98
C162-C168	1.504(14)	C171-C172	1.514(11)
C162-C163	1.519(11)	C171-H17G	0.99
C162-C169	1.540(11)	C171-H17H	0.99
C162-C167	1.569(11)	C172-H17I	0.99
C163-C164	1.530(11)	C172-H17J	0.99

C2-O1-C5	95.8(6)	C14-C13-H13	116.9
C8-O2-H2O	109.5	C15-C14-C13	115.8(12)
C16-N1-C23	109.0(12)	C15-C14-H14	122.1
C16-N1-H1	125.5	C13-C14-H14	122.1
C23-N1-H1	125.5	C16-C15-C18	108.4(12)
C13-C12-C17	118(3)	C16-C15-C14	121.1(13)
C13-C12-C11	116.3(17)	C18-C15-C14	130.5(11)
C17-C12-C11	125(2)	N1-C16-C15	110.2(12)
C12-C13-C14	126.1(19)	N1-C16-C17	127.1(12)
C12-C13-H13	116.9	C15-C16-C17	122.7(13)

C16-C17-C12	115.8(17)	C16D-C15D-C18D	109(2)
C16-C17-H17	122.1	C17D-C16D-N1D	126(2)
C12-C17-H17	122.1	C17D-C16D-C15D	125(2)
C19-C18-C15	140.5(13)	N1D-C16D-C15D	109(2)
C19-C18-C23	113.6(12)	C16D-C17D-C12D	112(3)
C15-C18-C23	105.8(12)	C16D-C17D-H17D	124.0
C20-C19-C18	124.4(12)	C12D-C17D-H17D	124.0
C20-C19-H19	117.8	C19D-C18D-C15D	136(2)
C18-C19-H19	117.8	C19D-C18D-C23D	118(2)
C19-C20-C21	119.7(12)	C15D-C18D-C23D	106(2)
C19-C20-H20	120.2	C18D-C19D-C20D	120(2)
C21-C20-H20	120.2	C18D-C19D-H19D	120.0
C22-C21-C20	118.9(13)	C20D-C19D-H19D	120.0
C22-C21-H21	120.6	C19D-C20D-C21D	120(2)
C20-C21-H21	120.6	C19D-C20D-H20D	120.0
C23-C22-C21	120.4(13)	C21D-C20D-H20D	120.0
C23-C22-H22	119.8	C22D-C21D-C20D	123(2)
C21-C22-H22	119.8	C22D-C21D-H21D	118.6
C22-C23-N1	130.5(16)	C20D-C21D-H21D	118.5
C22-C23-C18	123.0(13)	C21D-C22D-C23D	118(2)
N1-C23-C18	106.6(14)	C21D-C22D-H22D	121.1
C16D-N1D-C23D	110.3(18)	C23D-C22D-H22D	121.1
C16D-N1D-H1D	124.8	C22D-C23D-N1D	132(2)
C23D-N1D-H1D	124.8	C22D-C23D-C18D	122(2)
C13D-C12D-C17D	130(5)	N1D-C23D-C18D	106(2)
C13D-C12D-C11	93(3)	C7-C1-C2	115.9(8)
C17D-C12D-C11	136(3)	C7-C1-C8	109.1(7)
C12D-C13D-C14D	111(3)	C2-C1-C8	108.1(6)
C12D-C13D-H13D	124.5	C7-C1-C6	111.2(7)
C14D-C13D-H13D	124.6	C2-C1-C6	99.9(6)
C15D-C14D-C13D	126(2)	C8-C1-C6	112.5(6)
C15D-C14D-H14D	117.0	O1-C2-C3	101.4(6)
C13D-C14D-H14D	117.0	O1-C2-C1	102.4(7)
C14D-C15D-C16D	116(2)	C3-C2-C1	114.0(7)
C14D-C15D-C18D	136(2)	O1-C2-H2	112.7

C3-C2-H2	112.7	O2-C8-H8B	109.4
C1-C2-H2	112.7	C1-C8-H8B	109.4
C2-C3-C4	102.9(7)	H8A-C8-H8B	108.0
C2-C3-H3A	111.2	C5-C9-H9A	109.5
C4-C3-H3A	111.2	C5-C9-H9B	109.5
C2-C3-H3B	111.2	H9A-C9-H9B	109.5
C4-C3-H3B	111.2	C5-C9-H9C	109.5
H3A-C3-H3B	109.1	H9A-C9-H9C	109.5
C3-C4-C5	100.7(7)	H9B-C9-H9C	109.5
C3-C4-H4A	111.6	C6-C10-C11	113.3(8)
C5-C4-H4A	111.6	C6-C10-H10F	108.9
C3-C4-H4B	111.6	C11-C10-H10F	108.9
C5-C4-H4B	111.6	C6-C10-H10G	108.9
H4A-C4-H4B	109.4	C11-C10-H10G	108.9
O1-C5-C9	107.9(8)	H10F-C10-H10G	107.7
O1-C5-C4	101.5(7)	C12-C11-C10	111.9(9)
C9-C5-C4	114.4(7)	C12D-C11-C10	116.6(10)
O1-C5-C6	103.8(6)	C12-C11-H11E	109.2
C9-C5-C6	119.8(9)	C10-C11-H11E	109.2
C4-C5-C6	107.3(7)	C12-C11-H11F	109.2
C10-C6-C5	116.8(7)	C10-C11-H11F	109.2
C10-C6-C1	115.5(7)	H11E-C11-H11F	107.9
C5-C6-C1	100.7(6)	C25-O3-C28	96.3(5)
C10-C6-H6	107.8	C31-O4-H4O	109.5
C5-C6-H6	107.8	C39-N2-C46	108.1(6)
C1-C6-H6	107.8	C39-N2-H2N	125.9
C1-C7-H7A	109.5	C46-N2-H2N	125.9
C1-C7-H7B	109.5	C30-C24-C31	109.2(6)
H7A-C7-H7B	109.5	C30-C24-C25	114.0(6)
C1-C7-H7C	109.5	C31-C24-C25	108.2(5)
H7A-C7-H7C	109.5	C30-C24-C29	113.6(5)
H7B-C7-H7C	109.5	C31-C24-C29	111.6(6)
O2-C8-C1	111.1(7)	C25-C24-C29	100.1(5)
O2-C8-H8A	109.4	O3-C25-C26	103.0(6)
C1-C8-H8A	109.4	O3-C25-C24	101.0(5)

C26-C25-C24	114.1(5)	O4-C31-H31A	109.2
O3-C25-H25	112.6	C24-C31-H31A	109.2
C26-C25-H25	112.6	O4-C31-H31B	109.2
C24-C25-H25	112.6	C24-C31-H31B	109.2
C25-C26-C27	101.0(6)	H31A-C31-H31B	107.9
C25-C26-H26A	111.6	C28-C32-H32A	109.5
C27-C26-H26A	111.6	C28-C32-H32B	109.5
C25-C26-H26B	111.6	H32A-C32-H32B	109.5
C27-C26-H26B	111.6	C28-C32-H32C	109.5
H26A-C26-H26B	109.4	H32A-C32-H32C	109.5
C26-C27-C28	102.8(6)	H32B-C32-H32C	109.5
C26-C27-H27A	111.2	C29-C33-C34	113.3(6)
C28-C27-H27A	111.2	C29-C33-H33A	108.9
C26-C27-H27B	111.2	C34-C33-H33A	108.9
C28-C27-H27B	111.2	C29-C33-H33B	108.9
H27A-C27-H27B	109.1	C34-C33-H33B	108.9
O3-C28-C32	107.8(6)	H33A-C33-H33B	107.7
O3-C28-C27	99.9(6)	C35-C34-C33	113.1(6)
C32-C28-C27	116.0(7)	C35-C34-H34A	109.0
O3-C28-C29	102.5(5)	C33-C34-H34A	109.0
C32-C28-C29	119.8(7)	C35-C34-H34B	109.0
C27-C28-C29	108.1(6)	C33-C34-H34B	109.0
C33-C29-C28	115.7(6)	H34A-C34-H34B	107.8
C33-C29-C24	118.0(6)	C40-C35-C36	119.3(7)
C28-C29-C24	102.0(5)	C40-C35-C34	120.5(7)
C33-C29-H29	106.8	C36-C35-C34	120.1(7)
C28-C29-H29	106.8	C37-C36-C35	124.1(7)
C24-C29-H29	106.8	C37-C36-H36	118.0
C24-C30-H30A	109.5	C35-C36-H36	118.0
C24-C30-H30B	109.5	C36-C37-C38	116.4(7)
H30A-C30-H30B	109.5	C36-C37-H37	121.8
C24-C30-H30C	109.5	C38-C37-H37	121.8
H30A-C30-H30C	109.5	C39-C38-C37	120.8(7)
H30B-C30-H30C	109.5	C39-C38-C41	107.0(6)
O4-C31-C24	112.2(6)	C37-C38-C41	132.1(6)

N2-C39-C38	109.8(7)	C59-C60-C61	118.1(11)
N2-C39-C40	128.7(7)	C59-C60-H60	120.9
C38-C39-C40	121.4(7)	C61-C60-H60	120.9
C35-C40-C39	117.9(7)	C62-C61-C64	109.1(11)
C35-C40-H40	121.0	C62-C61-C60	118.4(11)
C39-C40-H40	121.0	C64-C61-C60	132.5(12)
C42-C41-C46	117.4(7)	C61-C62-N3	108.7(11)
C42-C41-C38	136.2(7)	C61-C62-C63	125.2(11)
C46-C41-C38	106.4(6)	N3-C62-C63	126.1(11)
C43-C42-C41	120.3(7)	C58-C63-C62	114.5(14)
C43-C42-H42	119.8	C58-C63-H63	122.7
C41-C42-H42	119.8	C62-C63-H63	122.7
C42-C43-C44	121.0(8)	C65-C64-C61	138.7(13)
C42-C43-H43	119.5	C65-C64-C69	116.6(11)
C44-C43-H43	119.5	C61-C64-C69	104.8(12)
C45-C44-C43	119.9(8)	C66-C65-C64	123.2(12)
C45-C44-H44	120.1	C66-C65-H65	118.4
C43-C44-H44	120.1	C64-C65-H65	118.4
C46-C45-C44	119.1(8)	C65-C66-C67	121.7(13)
C46-C45-H45	120.5	C65-C66-H66	119.1
C44-C45-H45	120.5	C67-C66-H66	119.1
C45-C46-N2	129.0(7)	C66-C67-C68	117.1(14)
C45-C46-C41	122.2(7)	C66-C67-H67	121.5
N2-C46-C41	108.6(7)	C68-C67-H67	121.5
C51-O5-C48	96.6(5)	C69-C68-C67	121.1(14)
C54-O6-H6O	109.5	C69-C68-H68	119.5
C69-N3-C62	108.5(10)	C67-C68-H68	119.5
C69-N3-H3N	125.8	N3-C69-C68	130.7(13)
C62-N3-H3N	125.8	N3-C69-C64	109.0(11)
C63-C58-C59	122.2(19)	C68-C69-C64	120.3(12)
C63-C58-C57	120.9(14)	C16E-N1E-C23E	108(2)
C59-C58-C57	116.0(11)	C16E-N1E-H1EN	126.1
C60-C59-C58	121.5(13)	C23E-N1E-H1EN	126.1
C60-C59-H59	119.2	C17E-C12E-C13E	124(4)
C58-C59-H59	119.2	C17E-C12E-C57	125(2)

C13E-C12E-C57	110(3)	C54-C47-C53	109.3(7)
C14E-C13E-C12E	119(3)	C48-C47-C53	114.3(7)
C14E-C13E-H13E	120.6	C54-C47-C52	111.7(6)
C12E-C13E-H13E	120.6	C48-C47-C52	100.2(6)
C13E-C14E-C15E	124(2)	C53-C47-C52	113.0(6)
C13E-C14E-H14E	117.8	O5-C48-C47	101.6(6)
C15E-C14E-H14E	117.9	O5-C48-C49	101.9(6)
C18E-C15E-C14E	138(2)	C47-C48-C49	113.6(6)
C18E-C15E-C16E	106(2)	O5-C48-H48	112.9
C14E-C15E-C16E	115(2)	C47-C48-H48	112.9
N1E-C16E-C15E	110(2)	C49-C48-H48	112.9
N1E-C16E-C17E	127(3)	C48-C49-C50	101.4(6)
C15E-C16E-C17E	122(3)	C48-C49-H49A	111.5
C12E-C17E-C16E	115(3)	C50-C49-H49A	111.5
C12E-C17E-H17E	122.7	C48-C49-H49B	111.5
C16E-C17E-H17E	122.7	C50-C49-H49B	111.5
C19E-C18E-C23E	121(3)	H49A-C49-H49B	109.3
C19E-C18E-C15E	131(3)	C51-C50-C49	102.2(6)
C23E-C18E-C15E	108(3)	C51-C50-H50A	111.3
C18E-C19E-C20E	118(3)	C49-C50-H50A	111.3
C18E-C19E-H19E	121.1	C51-C50-H50B	111.3
C20E-C19E-H19E	121.2	C49-C50-H50B	111.3
C19E-C20E-C21E	121(3)	H50A-C50-H50B	109.2
C19E-C20E-H20E	119.7	O5-C51-C55	108.3(6)
C21E-C20E-H20E	119.7	O5-C51-C50	101.1(6)
C22E-C21E-C20E	116(3)	C55-C51-C50	115.9(7)
C22E-C21E-H21E	121.8	O5-C51-C52	103.0(6)
C20E-C21E-H21E	121.8	C55-C51-C52	118.7(7)
C21E-C22E-C23E	125(3)	C50-C51-C52	107.5(6)
C21E-C22E-H22E	117.7	C56-C52-C51	115.7(6)
C23E-C22E-H22E	117.7	C56-C52-C47	118.1(6)
C18E-C23E-C22E	119(3)	C51-C52-C47	101.3(6)
C18E-C23E-N1E	108(3)	C56-C52-H52	107.0
C22E-C23E-N1E	133(3)	C51-C52-H52	107.0
C54-C47-C48	108.0(6)	C47-C52-H52	107.0

C47-C53-H53A	109.5	C92-N4-H4N	125.8
C47-C53-H53B	109.5	C86-C81-C82	121(2)
H53A-C53-H53B	109.5	C86-C81-C80	131.1(17)
C47-C53-H53C	109.5	C82-C81-C80	107(2)
H53A-C53-H53C	109.5	C83-C82-C81	122.8(19)
H53B-C53-H53C	109.5	C83-C82-H82	118.6
O6-C54-C47	111.6(6)	C81-C82-H82	118.6
O6-C54-H54A	109.3	C82-C83-C84	116.9(12)
C47-C54-H54A	109.3	C82-C83-H83	121.6
O6-C54-H54B	109.3	C84-C83-H83	121.6
C47-C54-H54B	109.3	C87-C84-C85	107.1(12)
H54A-C54-H54B	108.0	C87-C84-C83	135.0(12)
C51-C55-H55A	109.5	C85-C84-C83	117.8(12)
C51-C55-H55B	109.5	N4-C85-C86	127.4(14)
H55A-C55-H55B	109.5	N4-C85-C84	108.9(12)
C51-C55-H55C	109.5	C86-C85-C84	123.7(14)
H55A-C55-H55C	109.5	C81-C86-C85	117.4(18)
H55B-C55-H55C	109.5	C81-C86-H86	121.3
C52-C56-C57	114.0(6)	C85-C86-H86	121.3
C52-C56-H56A	108.8	C88-C87-C84	133.9(13)
C57-C56-H56A	108.7	C88-C87-C92	118.4(12)
C52-C56-H56B	108.8	C84-C87-C92	107.7(13)
C57-C56-H56B	108.8	C87-C88-C89	120.0(12)
H56A-C56-H56B	107.6	C87-C88-H88	120.0
C58-C57-C56	111.5(7)	C89-C88-H88	120.0
C56-C57-C12E	112.4(8)	C88-C89-C90	120.1(12)
C58-C57-H57A	109.3	C88-C89-H89	119.9
C56-C57-H57A	109.3	C90-C89-H89	119.9
C58-C57-H57B	109.3	C91-C90-C89	120.3(13)
C56-C57-H57B	109.3	C91-C90-H90	119.9
H57A-C57-H57B	108.0	C89-C90-H90	119.9
C71-O7-C74	96.0(5)	C92-C91-C90	119.2(13)
C77-O8-H8O	109.5	C92-C91-H91	120.4
C85-N4-C92	108.5(11)	C90-C91-H91	120.4
C85-N4-H4N	125.8	C91-C92-N4	130.3(14)

C91-C92-C87	122.0(12)	C23B-C22B-C21B	113(3)
N4-C92-C87	107.7(13)	C23B-C22B-H22B	123.5
C16B-N1B-C23B	110.0(18)	C21B-C22B-H22B	123.5
C16B-N1B-H1BN	125.0	C18B-C23B-C22B	131(2)
C23B-N1B-H1BN	125.0	C18B-C23B-N1B	105.5(19)
C17B-C12B-C13B	118.7(19)	C22B-C23B-N1B	123(2)
C17B-C12B-H12B	120.6	C77-C70-C76	109.1(7)
C13B-C12B-H12B	120.6	C77-C70-C71	107.9(6)
C14B-C13B-C12B	120(2)	C76-C70-C71	115.4(8)
C14B-C13B-H13B	119.9	C77-C70-C75	112.6(7)
C12B-C13B-H13B	119.9	C76-C70-C75	112.3(7)
C15B-C14B-C13B	122(2)	C71-C70-C75	99.2(6)
C15B-C14B-H14B	118.9	O7-C71-C72	101.9(6)
C13B-C14B-H14B	118.9	O7-C71-C70	102.1(6)
C14B-C15B-C18B	137(2)	C72-C71-C70	114.0(7)
C14B-C15B-C16B	117.6(19)	O7-C71-H71	112.6
C18B-C15B-C16B	105(2)	C72-C71-H71	112.6
N1B-C16B-C17B	133(2)	C70-C71-H71	112.6
N1B-C16B-C15B	107(2)	C71-C72-C73	101.1(6)
C17B-C16B-C15B	119.7(18)	C71-C72-H72A	111.6
C12B-C17B-C16B	121.5(19)	C73-C72-H72A	111.6
C12B-C17B-H17B	119.2	C71-C72-H72B	111.6
C16B-C17B-H17B	119.2	C73-C72-H72B	111.5
C23B-C18B-C15B	111.9(19)	H72A-C72-H72B	109.4
C23B-C18B-C19B	116(2)	C74-C73-C72	101.6(6)
C15B-C18B-C19B	131.6(18)	C74-C73-H73A	111.5
C20B-C19B-C18B	116.4(19)	C72-C73-H73A	111.5
C20B-C19B-H19B	121.8	C74-C73-H73B	111.5
C18B-C19B-H19B	121.8	C72-C73-H73B	111.5
C19B-C20B-C21B	125(3)	H73A-C73-H73B	109.3
C19B-C20B-H20B	117.6	O7-C74-C78	109.1(7)
C21B-C20B-H20B	117.6	O7-C74-C73	100.9(6)
C22B-C21B-C20B	118(4)	C78-C74-C73	113.2(7)
C22B-C21B-C80	138(3)	O7-C74-C75	104.5(5)
C20B-C21B-C80	104(2)	C78-C74-C75	118.8(7)

C73-C74-C75	108.5(7)	C81-C80-H80B	108.2
C79-C75-C74	117.4(7)	C79-C80-H80B	108.2
C79-C75-C70	116.0(7)	H80A-C80-H80B	107.3
C74-C75-C70	101.0(6)	C94-O9-C97	97.5(5)
C79-C75-H75	107.2	C100-O10-H10O	109.5
C74-C75-H75	107.2	C99-C93-C94	115.6(8)
C70-C75-H75	107.2	C99-C93-C100	107.4(7)
C70-C76-H76A	109.5	C94-C93-C100	107.1(6)
C70-C76-H76B	109.5	C99-C93-C98	113.5(6)
H76A-C76-H76B	109.5	C94-C93-C98	100.9(6)
C70-C76-H76C	109.5	C100-C93-C98	112.2(6)
H76A-C76-H76C	109.5	O9-C94-C95	102.4(7)
H76B-C76-H76C	109.5	O9-C94-C93	100.7(6)
O8-C77-C70	111.3(7)	C95-C94-C93	112.0(6)
O8-C77-H77A	109.4	O9-C94-H94	113.5
C70-C77-H77A	109.4	C95-C94-H94	113.5
O8-C77-H77B	109.4	C93-C94-H94	113.5
C70-C77-H77B	109.4	C94-C95-C96	101.6(6)
H77A-C77-H77B	108.0	C94-C95-H95A	111.5
C74-C78-H78A	109.5	C96-C95-H95A	111.5
C74-C78-H78B	109.5	C94-C95-H95B	111.5
H78A-C78-H78B	109.5	C96-C95-H95B	111.5
C74-C78-H78C	109.5	H95A-C95-H95B	109.3
H78A-C78-H78C	109.5	C97-C96-C95	102.1(6)
H78B-C78-H78C	109.5	C97-C96-H96A	111.4
C75-C79-C80	113.1(7)	C95-C96-H96A	111.4
C75-C79-H79A	109.0	C97-C96-H96B	111.4
C80-C79-H79A	109.0	C95-C96-H96B	111.4
C75-C79-H79B	109.0	H96A-C96-H96B	109.2
C80-C79-H79B	109.0	O9-C97-C101	109.0(6)
H79A-C79-H79B	107.8	O9-C97-C96	100.2(6)
C81-C80-C79	116.6(8)	C101-C97-C96	114.1(7)
C21B-C80-C79	114.9(9)	O9-C97-C98	102.7(6)
C81-C80-H80A	108.1	C101-C97-C98	120.4(7)
C79-C80-H80A	108.1	C96-C97-C98	108.0(6)

C102-C98-C97	116.5(6)	C102-C103-H10K	109.0
C102-C98-C93	116.7(6)	H10J-C103-H10K	107.8
C97-C98-C93	101.6(6)	C108-N5-C115	109.3(12)
C102-C98-H98	107.1	C108-N5-H5N	125.3
C97-C98-H98	107.1	C115-N5-H5N	125.3
C93-C98-H98	107.1	C109-C104-C105	120.6(16)
C93-C99-H99A	109.5	C109-C104-C103	130.4(15)
C93-C99-H99B	109.5	C105-C104-C103	108.9(13)
H99A-C99-H99B	109.5	C106-C105-C104	120.0(14)
C93-C99-H99C	109.5	C106-C105-H105	120.0
H99A-C99-H99C	109.5	C104-C105-H105	120.0
H99B-C99-H99C	109.5	C105-C106-C107	119.5(13)
O10-C100-C93	111.8(7)	C105-C106-H106	120.2
O10-C100-H10A	109.2	C107-C106-H106	120.2
C93-C100-H10A	109.2	C106-C107-C108	118.6(14)
O10-C100-H10B	109.2	C106-C107-C110	136.4(13)
C93-C100-H10B	109.2	C108-C107-C110	105.0(13)
H10A-C100-H10B	107.9	C109-C108-N5	125.1(14)
C97-C101-H10C	109.5	C109-C108-C107	125.1(13)
C97-C101-H10D	109.5	N5-C108-C107	109.9(14)
H10C-C101-H10D	109.5	C108-C109-C104	116.3(15)
C97-C101-H10E	109.5	C108-C109-H109	121.9
H10C-C101-H10E	109.5	C104-C109-H109	121.8
H10D-C101-H10E	109.5	C111-C110-C115	121.7(13)
C98-C102-C103	113.3(6)	C111-C110-C107	130.4(16)
C98-C102-H10H	108.9	C115-C110-C107	107.9(15)
C103-C102-H10H	108.9	C110-C111-C112	115.8(15)
C98-C102-H10I	108.9	C110-C111-H111	122.1
C103-C102-H10I	108.9	C112-C111-H111	122.1
H10H-C102-H10I	107.7	C113-C112-C111	119.8(16)
C12A-C103-C102	118.2(8)	C113-C112-H112	120.1
C104-C103-C102	113.0(7)	C111-C112-H112	120.1
C104-C103-H10J	109.0	C114-C113-C112	122.4(15)
C102-C103-H10J	109.0	C114-C113-H113	118.8
C104-C103-H10K	109.0	C112-C113-H113	118.8

C113-C114-C115	118.2(14)	C21A-C20A-H20A	117.2
C113-C114-H114	120.9	C20A-C21A-C22A	120(3)
C115-C114-H114	120.9	C20A-C21A-H21A	119.9
C114-C115-N5	129.9(15)	C22A-C21A-H21A	119.9
C114-C115-C110	122.2(13)	C23A-C22A-C21A	115(2)
N5-C115-C110	108.0(14)	C23A-C22A-H22A	122.3
C23A-N1A-C16A	111(2)	C21A-C22A-H22A	122.3
C23A-N1A-H1AN	124.7	N1A-C23A-C22A	129(2)
C16A-N1A-H1AN	124.7	N1A-C23A-C18A	112(2)
C103-C12A-C17A	139(2)	C22A-C23A-C18A	119(2)
C103-C12A-C13A	109.7(19)	C117-O11-C120	96.7(5)
C17A-C12A-C13A	112(2)	C123-O12-H12O	109.5
C14A-C13A-C12A	129(2)	C131-N6-C138	108.9(8)
C14A-C13A-H13A	115.7	C131-N6-H6N	125.5
C12A-C13A-H13A	115.7	C138-N6-H6N	125.5
C15A-C14A-C13A	114(2)	C123-C116-C122	110.0(6)
C15A-C14A-H14A	122.8	C123-C116-C117	107.1(6)
C13A-C14A-H14A	122.8	C122-C116-C117	113.6(6)
C14A-C15A-C18A	131(2)	C123-C116-C121	112.5(6)
C14A-C15A-C16A	120(2)	C122-C116-C121	112.4(6)
C18A-C15A-C16A	109(2)	C117-C116-C121	100.9(5)
C17A-C16A-N1A	133(3)	O11-C117-C118	101.2(5)
C17A-C16A-C15A	123(2)	O11-C117-C116	100.6(5)
N1A-C16A-C15A	105(2)	C118-C117-C116	112.4(6)
C16A-C17A-C12A	123(3)	O11-C117-H117	113.8
C16A-C17A-H17A	118.5	C118-C117-H117	113.8
C12A-C17A-H17A	118.5	C116-C117-H117	113.8
C23A-C18A-C15A	104(2)	C117-C118-C119	102.8(6)
C23A-C18A-C19A	122(2)	C117-C118-H11A	111.2
C15A-C18A-C19A	134(3)	C119-C118-H11A	111.2
C20A-C19A-C18A	117(2)	C117-C118-H11B	111.2
C20A-C19A-H19A	121.4	C119-C118-H11B	111.2
C18A-C19A-H19A	121.4	H11A-C118-H11B	109.1
C19A-C20A-C21A	126(3)	C120-C119-C118	100.7(6)
C19A-C20A-H20A	117.2	C120-C119-H11C	111.6

C118-C119-H11C	111.6	C121-C125-H12I	108.9
C120-C119-H11D	111.6	C126-C125-H12I	108.9
C118-C119-H11D	111.6	C121-C125-H12J	109.0
H11C-C119-H11D	109.4	C126-C125-H12J	108.9
O11-C120-C124	106.9(6)	H12I-C125-H12J	107.8
O11-C120-C121	104.4(5)	C127-C126-C125	113.5(6)
C124-C120-C121	118.7(6)	C127-C126-H12K	108.9
O11-C120-C119	100.7(6)	C125-C126-H12K	108.9
C124-C120-C119	115.0(6)	C127-C126-H12L	108.9
C121-C120-C119	108.9(6)	C125-C126-H12L	108.9
C125-C121-C120	116.2(5)	H12K-C126-H12L	107.7
C125-C121-C116	118.1(6)	C132-C127-C128	118.3(8)
C120-C121-C116	100.9(5)	C132-C127-C126	122.6(8)
C125-C121-H121	107.0	C128-C127-C126	119.1(8)
C120-C121-H121	107.0	C129-C128-C127	122.5(9)
C116-C121-H121	107.0	C129-C128-H128	118.8
C116-C122-H12Q	109.5	C127-C128-H128	118.8
C116-C122-H12R	109.5	C128-C129-C130	121.9(9)
H12Q-C122-H12R	109.5	C128-C129-H129	119.1
C116-C122-H12S	109.5	C130-C129-H129	119.1
H12Q-C122-H12S	109.5	C129-C130-C131	118.5(8)
H12R-C122-H12S	109.5	C129-C130-C133	136.5(8)
O12-C123-C116	111.9(6)	C131-C130-C133	105.0(8)
O12-C123-H12D	109.2	N6-C131-C132	129.4(9)
C116-C123-H12D	109.2	N6-C131-C130	109.9(8)
O12-C123-H12E	109.2	C132-C131-C130	120.7(8)
C116-C123-H12E	109.2	C127-C132-C131	118.2(8)
H12D-C123-H12E	107.9	C127-C132-H132	120.9
C120-C124-H12F	109.5	C131-C132-H132	120.9
C120-C124-H12G	109.5	C138-C133-C134	118.2(9)
H12F-C124-H12G	109.5	C138-C133-C130	107.2(8)
C120-C124-H12H	109.5	C134-C133-C130	134.6(9)
H12F-C124-H12H	109.5	C135-C134-C133	119.2(10)
H12G-C124-H12H	109.5	C135-C134-H134	120.4
C121-C125-C126	113.1(6)	C133-C134-H134	120.4

C134-C135-C136	120.9(10)	C157-C156-C161	121.8(11)
C134-C135-H135	119.5	C157-C156-C153	133.6(13)
C136-C135-H135	119.5	C161-C156-C153	104.5(12)
C137-C136-C135	121.3(10)	C156-C157-C158	117.4(11)
C137-C136-H136	119.4	C156-C157-H157	121.3
C135-C136-H136	119.4	C158-C157-H157	121.3
C138-C137-C136	116.3(10)	C159-C158-C157	120.3(11)
C138-C137-H137	121.8	C159-C158-H158	119.8
C136-C137-H137	121.8	C157-C158-H158	119.8
C137-C138-N6	126.9(10)	C160-C159-C158	122.5(12)
C137-C138-C133	124.1(9)	C160-C159-H159	118.8
N6-C138-C133	109.0(9)	C158-C159-H159	118.7
C140-O13-C143	96.9(5)	C159-C160-C161	118.5(12)
C146-O14-H14O	109.5	C159-C160-H160	120.7
C161-N7-C154	108.6(11)	C161-C160-H160	120.7
C161-N7-H7N	125.7	N7-C161-C156	112.9(14)
C154-N7-H7N	125.7	N7-C161-C160	127.7(14)
C155-C150-C151	116.1(14)	C156-C161-C160	119.4(10)
C155-C150-C149	125.1(10)	C16F-N1F-C23F	109(3)
C151-C150-C149	118.0(12)	C16F-N1F-H1FN	125.7
C152-C151-C150	122.5(12)	C23F-N1F-H1FN	125.7
C152-C151-H151	118.8	C13F-C12F-C17F	112(5)
C150-C151-H151	118.8	C13F-C12F-C149	119(3)
C151-C152-C153	119.5(11)	C17F-C12F-C149	129(3)
C151-C152-H152	120.3	C12F-C13F-C14F	144(3)
C153-C152-H152	120.3	C12F-C13F-H13F	107.9
C152-C153-C154	119.4(11)	C14F-C13F-H13F	107.8
C152-C153-C156	135.3(11)	C13F-C14F-C15F	98(3)
C154-C153-C156	105.3(10)	C13F-C14F-H14F	131.2
C155-C154-N7	131.0(11)	C15F-C14F-H14F	131.2
C155-C154-C153	120.2(11)	C16F-C15F-C18F	104(4)
N7-C154-C153	108.8(11)	C16F-C15F-C14F	127(4)
C154-C155-C150	122.2(12)	C18F-C15F-C14F	129(4)
C154-C155-H155	118.9	C17F-C16F-N1F	133(4)
C150-C155-H155	118.9	C17F-C16F-C15F	120(4)

N1F-C16F-C15F	107(3)	C140-C141-H14T	111.4
C16F-C17F-C12F	119(4)	C142-C141-H14T	111.4
C16F-C17F-H17F	120.5	C140-C141-H14X	111.4
C12F-C17F-H17F	120.5	C142-C141-H14X	111.4
C23F-C18F-C19F	124(4)	H14T-C141-H14X	109.3
C23F-C18F-C15F	112(4)	C141-C142-C143	102.0(6)
C19F-C18F-C15F	124(4)	C141-C142-H14Y	111.4
C18F-C19F-C20F	122(4)	C143-C142-H14Y	111.4
C18F-C19F-H19F	119.1	C141-C142-H14Z	111.4
C20F-C19F-H19F	119.1	C143-C142-H14Z	111.4
C21F-C20F-C19F	117(4)	H14Y-C142-H14Z	109.2
C21F-C20F-H20F	121.3	O13-C143-C147	109.1(6)
C19F-C20F-H20F	121.3	O13-C143-C142	100.4(6)
C20F-C21F-C22F	125(4)	C147-C143-C142	114.9(6)
C20F-C21F-H21F	117.7	O13-C143-C144	103.7(5)
C22F-C21F-H21F	117.7	C147-C143-C144	118.4(7)
C23F-C22F-C21F	111(3)	C142-C143-C144	108.1(6)
C23F-C22F-H22F	124.7	C148-C144-C143	117.0(6)
C21F-C22F-H22F	124.6	C148-C144-C139	117.6(6)
C18F-C23F-C22F	122(4)	C143-C144-C139	100.8(6)
C18F-C23F-N1F	109(4)	C148-C144-H144	106.9
C22F-C23F-N1F	129(4)	C143-C144-H144	106.9
C145-C139-C146	109.6(6)	C139-C144-H144	106.9
C145-C139-C140	113.9(7)	C139-C145-H14Q	109.5
C146-C139-C140	106.3(6)	C139-C145-H14R	109.5
C145-C139-C144	113.6(6)	H14Q-C145-H14R	109.5
C146-C139-C144	112.7(6)	C139-C145-H14S	109.5
C140-C139-C144	100.3(5)	H14Q-C145-H14S	109.5
O13-C140-C141	102.7(6)	H14R-C145-H14S	109.5
O13-C140-C139	101.1(6)	O14-C146-C139	112.2(7)
C141-C140-C139	112.5(6)	O14-C146-H14H	109.2
O13-C140-H140	113.1	C139-C146-H14H	109.2
C141-C140-H140	113.1	O14-C146-H14I	109.2
C139-C140-H140	113.1	C139-C146-H14I	109.2
C140-C141-C142	101.7(6)	H14H-C146-H14I	107.9

C143-C147-H14J	109.5	C175-C176-C177	115.0(12)
C143-C147-H14K	109.5	C178-C177-N8	132.0(15)
H14J-C147-H14K	109.5	C178-C177-C176	122.6(13)
C143-C147-H14L	109.5	N8-C177-C176	105.4(12)
H14J-C147-H14L	109.5	C177-C178-C173	120.2(16)
H14K-C147-H14L	109.5	C177-C178-H178	119.9
C144-C148-C149	113.8(6)	C173-C178-H178	119.9
C144-C148-H14M	108.8	C176-C179-C184	109.3(13)
C149-C148-H14M	108.8	C176-C179-C180	131.8(12)
C144-C148-H14N	108.8	C184-C179-C180	118.9(12)
C149-C148-H14N	108.8	C181-C180-C179	117.0(12)
H14M-C148-H14N	107.7	C181-C180-H180	121.5
C12F-C149-C148	115.4(8)	C179-C180-H180	121.5
C150-C149-C148	111.3(6)	C182-C181-C180	122.4(13)
C150-C149-H201	109.4	C182-C181-H181	118.8
C148-C149-H201	109.4	C180-C181-H181	118.8
C150-C149-H202	109.4	C181-C182-C183	122.6(12)
C148-C149-H202	109.4	C181-C182-H182	118.7
H201-C149-H202	108.0	C183-C182-H182	118.7
C163-O15-C166	95.2(5)	C184-C183-C182	115.3(13)
C169-O16-H16O	109.5	C184-C183-H183	122.3
C184-N8-C177	110.7(11)	C182-C183-H183	122.3
C184-N8-H8N	124.7	N8-C184-C183	128.6(13)
C177-N8-H8N	124.7	N8-C184-C179	107.6(12)
C178-C173-C174	118.1(17)	C183-C184-C179	123.7(14)
C178-C173-C172	126.4(16)	C16C-N1C-C23C	109(3)
C174-C173-C172	115.4(18)	C16C-N1C-H1CN	125.4
C175-C174-C173	120.6(15)	C23C-N1C-H1CN	125.4
C175-C174-H174	119.7	C13C-C12C-C17C	116(4)
C173-C174-H174	119.7	C13C-C12C-C172	98(3)
C174-C175-C176	123.4(13)	C17C-C12C-C172	146(4)
C174-C175-H175	118.3	C14C-C13C-C12C	124(3)
C176-C175-H175	118.3	C14C-C13C-H13C	118.0
C179-C176-C175	138.0(13)	C12C-C13C-H13C	118.0
C179-C176-C177	107.0(12)	C13C-C14C-C15C	118(3)

C13C-C14C-H203	120.9	O15-C163-C162	102.4(6)
C15C-C14C-H203	120.9	O15-C163-C164	102.2(6)
C14C-C15C-C16C	125(3)	C162-C163-C164	114.1(6)
C14C-C15C-C18C	132(2)	O15-C163-H163	112.4
C16C-C15C-C18C	103(2)	C162-C163-H163	112.4
N1C-C16C-C17C	131(3)	C164-C163-H163	112.4
N1C-C16C-C15C	111(3)	C163-C164-C165	101.3(6)
C17C-C16C-C15C	116(3)	C163-C164-H16A	111.5
C16C-C17C-C12C	120(3)	C165-C164-H16A	111.5
C16C-C17C-H17C	119.9	C163-C164-H16B	111.5
C12C-C17C-H17C	119.9	C165-C164-H16B	111.5
C19C-C18C-C23C	121(3)	H16A-C164-H16B	109.3
C19C-C18C-C15C	135(3)	C166-C165-C164	101.1(7)
C23C-C18C-C15C	104(2)	C166-C165-H16C	111.5
C18C-C19C-C20C	120(3)	C164-C165-H16C	111.5
C18C-C19C-H19C	119.9	C166-C165-H16D	111.5
C20C-C19C-H19C	119.9	C164-C165-H16D	111.5
C21C-C20C-C19C	118(3)	H16C-C165-H16D	109.4
C21C-C20C-H20C	121.0	O15-C166-C165	100.9(6)
C19C-C20C-H20C	121.0	O15-C166-C170	107.7(7)
C22C-C21C-C20C	124(3)	C165-C166-C170	113.0(8)
C22C-C21C-H21C	118.1	O15-C166-C167	103.4(6)
C20C-C21C-H21C	118.1	C165-C166-C167	109.5(7)
C21C-C22C-C23C	119(3)	C170-C166-C167	120.0(7)
C21C-C22C-H22C	120.4	C171-C167-C166	115.5(7)
C23C-C22C-H22C	120.4	C171-C167-C162	117.5(7)
N1C-C23C-C18C	112(3)	C166-C167-C162	101.2(6)
N1C-C23C-C22C	130(3)	C171-C167-H167	107.3
C18C-C23C-C22C	117(2)	C166-C167-H167	107.3
C168-C162-C163	115.8(8)	C162-C167-H167	107.3
C168-C162-C169	107.7(8)	C162-C168-H16E	109.5
C163-C162-C169	109.4(6)	C162-C168-H16F	109.5
C168-C162-C167	112.6(7)	H16E-C168-H16F	109.5
C163-C162-C167	99.8(7)	C162-C168-H16G	109.5
C169-C162-C167	111.5(7)	H16E-C168-H16G	109.5

H16F-C168-H16G	109.5	C172-C171-C167	113.4(7)
O16-C169-C162	111.0(8)	C172-C171-H17G	108.9
O16-C169-H16H	109.4	C167-C171-H17G	108.9
C162-C169-H16H	109.4	C172-C171-H17H	108.9
O16-C169-H16I	109.4	C167-C171-H17H	108.9
C162-C169-H16I	109.4	H17G-C171-H17H	107.7
H16H-C169-H16I	108.0	C173-C172-C171	115.7(12)
C166-C170-H17X	109.5	C171-C172-C12C	114.2(19)
C166-C170-H17Y	109.5	C173-C172-H17I	108.4
H17X-C170-H17Y	109.5	C171-C172-H17I	108.4
C166-C170-H17Z	109.5	C173-C172-H17J	108.4
H17X-C170-H17Z	109.5	C171-C172-H17J	108.4
H17Y-C170-H17Z	109.5	H17I-C172-H17J	107.4

Table S10. Anisotropic displacement parameters ($Å^2$x 10^3) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[h^2 $a^{*2}U^{11}$ + ... + 2 h k a* b* U^{12}]

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	33(3)	28(3)	77(4)	-12(2)	-19(2)	-4(2)
O2	28(2)	30(3)	65(4)	-17(2)	-2(2)	-3(2)
N1	23(3)	32(4)	40(4)	-6(3)	-5(3)	-4(3)
C12	36(3)	28(3)	43(3)	-10(3)	-9(2)	2(2)
C13	34(4)	27(4)	43(5)	-11(3)	-3(4)	5(3)
C14	23(4)	24(4)	40(4)	-8(3)	-4(3)	1(3)
C15	19(3)	19(3)	38(3)	-11(3)	-5(3)	1(3)
C16	24(4)	22(4)	37(4)	-11(3)	-6(3)	0(3)
C17	32(4)	24(4)	42(4)	-10(3)	-12(3)	3(3)
C18	22(4)	20(3)	38(4)	-14(3)	-3(3)	1(3)
C19	18(4)	22(4)	35(4)	-13(3)	-4(3)	7(3)
C20	27(4)	21(4)	37(4)	-14(3)	0(4)	-1(4)
C21	31(4)	26(4)	44(4)	-11(3)	0(4)	0(4)
C22	25(4)	26(4)	42(4)	-11(3)	-3(4)	3(3)
C23	26(4)	27(4)	41(4)	-9(3)	-4(3)	1(3)
N1D	24(4)	26(5)	37(4)	-10(4)	0(3)	-9(4)
C12D	36(3)	28(3)	43(3)	-10(3)	-9(2)	2(2)
C13D	37(5)	24(5)	39(5)	-14(4)	-11(4)	7(4)
C14D	28(5)	21(5)	38(5)	-14(4)	-8(4)	1(4)
C15D	25(4)	23(4)	37(4)	-9(3)	-5(3)	-4(4)
C16D	24(4)	21(4)	38(4)	-9(3)	-3(3)	0(4)
C17D	28(5)	24(5)	37(5)	-12(4)	-2(4)	-2(4)
C18D	22(4)	25(4)	37(4)	-10(3)	-1(3)	-1(4)
C19D	22(5)	23(5)	37(5)	-5(4)	-1(4)	2(4)
C20D	24(5)	27(5)	40(5)	-9(4)	-1(4)	6(4)
C21D	24(5)	29(5)	37(5)	-11(4)	-1(4)	5(4)
C22D	24(5)	22(5)	35(5)	-13(4)	0(4)	3(4)
C23D	20(4)	23(4)	36(4)	-11(3)	-2(3)	4(4)
C1	26(3)	32(3)	42(4)	-13(3)	-9(3)	4(2)

C2	31(3)	30(3)	54(4)	-14(3)	-11(3)	1(3)
C3	69(6)	35(4)	52(4)	-14(3)	-23(4)	2(4)
C4	51(4)	32(4)	58(4)	-11(3)	-19(3)	5(3)
C5	48(4)	31(3)	56(4)	-13(3)	-21(3)	1(3)
C6	33(3)	33(3)	55(4)	-17(3)	-20(3)	11(3)
C7	42(4)	65(6)	63(6)	-22(5)	-23(4)	6(4)
C8	35(4)	26(3)	52(4)	-12(3)	-7(3)	3(3)
C9	90(7)	33(4)	83(7)	-10(4)	-48(6)	-12(5)
C10	42(4)	50(4)	53(4)	-21(3)	-17(3)	23(4)
C11	103(7)	70(6)	56(4)	-22(4)	-15(4)	45(5)
O3	24(2)	25(2)	46(3)	-10(2)	-2(2)	-5(2)
O4	21(2)	35(3)	66(4)	-6(2)	-8(2)	-2(2)
N2	29(3)	37(3)	36(3)	-8(2)	-6(2)	-13(3)
C24	24(3)	22(3)	38(3)	-9(2)	-1(2)	-6(2)
C25	22(3)	26(3)	35(3)	-6(2)	-1(2)	-6(2)
C26	38(3)	39(4)	34(3)	-7(3)	-7(3)	-7(3)
C27	30(3)	35(4)	45(4)	-2(3)	-11(3)	-12(3)
C28	18(3)	25(3)	46(3)	-5(2)	-10(2)	0(2)
C29	19(3)	22(3)	36(3)	-9(2)	-2(2)	-7(2)
C30	32(3)	28(3)	52(4)	-14(3)	2(3)	-1(3)
C31	24(3)	32(3)	41(3)	-8(3)	-7(2)	-9(3)
C32	29(3)	28(3)	59(5)	-6(3)	2(3)	-6(3)
C33	26(3)	20(3)	41(3)	-9(2)	-3(2)	1(2)
C34	43(4)	20(3)	38(3)	-7(2)	-3(3)	0(3)
C35	30(3)	20(3)	42(3)	-9(2)	-6(2)	5(2)
C36	29(3)	29(4)	38(3)	-7(3)	-7(2)	3(3)
C37	29(3)	22(3)	38(3)	-7(2)	-11(2)	-7(3)
C38	15(3)	23(3)	47(3)	-9(2)	-8(2)	-9(2)
C39	22(3)	29(3)	42(3)	-9(3)	-9(2)	-8(3)
C40	33(3)	25(3)	41(3)	-12(3)	-8(3)	-4(3)
C41	29(3)	21(3)	36(3)	-5(2)	-10(2)	-3(2)
C42	25(3)	23(3)	45(3)	-5(3)	-7(3)	-4(3)
C43	35(3)	26(3)	46(4)	0(3)	2(3)	-7(3)
C44	43(4)	35(4)	41(4)	-7(3)	-12(3)	1(3)
C45	30(3)	26(4)	53(4)	-8(3)	-13(3)	0(3)

C46	18(3)	16(3)	44(3)	-4(2)	-5(2)	0(2)
O5	27(2)	28(3)	42(3)	-10(2)	-8(2)	-4(2)
O6	21(2)	43(3)	61(4)	-22(3)	-10(2)	2(2)
N3	26(3)	30(3)	34(3)	-5(3)	-6(3)	-10(3)
C58	27(3)	32(3)	40(3)	-13(2)	-4(3)	-1(2)
C59	27(4)	31(4)	41(4)	-11(3)	-2(3)	-2(3)
C60	25(4)	28(4)	44(4)	-8(3)	-3(3)	-10(3)
C61	25(3)	21(3)	38(4)	-8(3)	-10(3)	-9(3)
C62	25(3)	25(3)	37(3)	-11(3)	-3(3)	-8(3)
C63	33(4)	28(4)	38(4)	-11(3)	-8(3)	-2(3)
C64	30(3)	26(3)	37(3)	-10(3)	-9(3)	-2(3)
C65	35(4)	24(4)	35(4)	-7(3)	-13(3)	5(3)
C66	37(4)	27(4)	35(4)	-6(3)	-14(3)	7(4)
C67	37(4)	24(4)	34(4)	-11(3)	-12(3)	6(3)
C68	37(4)	22(4)	34(4)	-2(3)	-14(4)	1(3)
C69	37(4)	23(4)	34(3)	-6(3)	-10(3)	-2(3)
N1E	27(4)	29(5)	40(4)	-7(4)	-12(4)	-8(4)
C12E	27(3)	32(3)	40(3)	-13(2)	-4(3)	-1(2)
C13E	23(5)	29(5)	37(5)	-7(4)	-8(4)	4(4)
C14E	28(5)	27(5)	37(4)	-9(4)	-10(4)	0(4)
C15E	27(4)	24(4)	36(4)	-5(4)	-10(3)	-3(4)
C16E	22(4)	29(4)	37(4)	-10(3)	-7(3)	-1(4)
C17E	25(5)	34(5)	39(4)	-10(4)	-3(4)	-2(4)
C18E	30(4)	27(4)	34(4)	-6(3)	-9(3)	-3(4)
C19E	31(5)	18(5)	36(5)	-1(4)	-11(4)	3(4)
C20E	28(5)	23(5)	36(5)	-1(4)	-11(4)	10(5)
C21E	29(5)	25(5)	37(5)	-8(4)	-5(4)	7(5)
C22E	31(5)	23(5)	37(5)	-6(4)	-9(4)	4(4)
C23E	29(4)	25(4)	37(4)	-6(4)	-11(4)	2(4)
C47	27(3)	36(3)	37(3)	-9(3)	-5(2)	-5(2)
C48	20(3)	32(3)	37(3)	-9(2)	-3(2)	-4(2)
C49	25(3)	45(4)	34(3)	-4(3)	1(2)	-8(3)
C50	28(3)	43(4)	40(3)	-13(3)	0(3)	-5(3)
C51	24(3)	33(3)	40(3)	-13(3)	-5(2)	-5(2)
C52	23(3)	27(3)	44(3)	-15(3)	-4(2)	-6(2)

C53	42(4)	34(4)	48(4)	-5(3)	-11(3)	-5(3)
C54	20(3)	38(4)	40(3)	-14(3)	-3(2)	-1(2)
C55	30(3)	37(4)	47(4)	-14(3)	-9(3)	2(3)
C56	21(3)	26(3)	43(3)	-14(3)	-6(2)	5(2)
C57	41(4)	23(3)	42(3)	-14(3)	-2(3)	1(3)
O7	22(2)	31(2)	40(3)	-6(2)	-5(2)	-4(2)
O8	34(3)	32(3)	90(5)	-21(3)	-17(3)	4(2)
N4	20(3)	29(4)	32(3)	-7(3)	-5(3)	-12(3)
C81	34(4)	31(3)	38(3)	-7(2)	-8(3)	5(3)
C82	40(4)	32(4)	36(4)	-6(4)	-10(4)	6(4)
C83	27(4)	25(4)	37(4)	-2(3)	-9(3)	-3(3)
C84	23(3)	19(3)	35(4)	-5(3)	-6(3)	-4(3)
C85	20(4)	20(4)	34(4)	-6(3)	-4(3)	-2(3)
C86	33(4)	27(4)	36(4)	-2(3)	-4(4)	0(4)
C87	24(3)	21(3)	33(3)	-4(3)	-3(3)	-7(3)
C88	25(4)	22(4)	31(4)	-4(3)	-2(3)	-7(3)
C89	32(4)	33(4)	35(4)	-7(4)	-3(4)	-9(4)
C90	33(4)	35(4)	29(4)	-10(3)	0(4)	-7(4)
C91	29(4)	28(4)	33(4)	-10(3)	1(4)	-5(3)
C92	26(4)	21(4)	33(4)	-8(3)	-4(3)	-7(3)
N1B	18(4)	24(4)	36(4)	-4(3)	-6(3)	-5(3)
C12B	32(5)	31(5)	31(5)	-10(4)	-6(4)	-5(4)
C13B	31(5)	35(5)	33(5)	-11(4)	-4(4)	-8(5)
C14B	30(4)	28(5)	32(5)	-11(4)	-5(4)	-6(4)
C15B	23(4)	24(4)	34(4)	-11(3)	-5(3)	-1(4)
C16B	22(4)	24(4)	33(4)	-8(3)	-5(3)	-2(4)
C17B	27(4)	27(5)	32(5)	-8(4)	-6(4)	-6(4)
C18B	20(4)	25(4)	35(4)	-5(3)	-5(3)	-5(3)
C19B	22(4)	26(5)	36(4)	-6(4)	-6(4)	-2(4)
C20B	29(5)	29(5)	36(5)	-4(4)	-4(4)	3(4)
C21B	34(4)	31(3)	38(3)	-7(2)	-8(3)	5(3)
C22B	32(4)	25(5)	34(4)	-9(4)	-6(4)	6(4)
C23B	24(4)	22(4)	33(4)	-5(3)	-7(3)	-3(4)
C70	27(3)	31(3)	49(4)	-11(3)	-12(3)	5(2)
C71	22(3)	35(3)	43(3)	-9(3)	0(2)	-2(2)

C72	45(4)	40(4)	39(4)	-10(3)	-3(3)	4(3)
C73	46(4)	40(4)	38(3)	-18(3)	-4(3)	8(3)
C74	38(3)	25(3)	36(3)	-10(2)	-5(2)	-1(3)
C75	31(3)	36(3)	44(3)	-11(3)	-12(3)	9(3)
C76	28(4)	49(6)	94(7)	-10(5)	-5(4)	4(4)
C77	43(4)	32(3)	61(4)	-22(3)	-22(3)	6(3)
C78	56(5)	34(4)	52(5)	-5(3)	-2(4)	-10(4)
C79	47(4)	42(4)	44(4)	-11(3)	-13(3)	15(3)
C80	97(7)	63(5)	42(4)	-8(3)	-11(4)	43(5)
O9	30(2)	31(2)	45(3)	-10(2)	-10(2)	-9(2)
O10	32(3)	25(2)	70(4)	-13(2)	1(2)	-1(2)
C93	25(3)	25(3)	46(4)	-6(2)	-7(3)	-8(2)
C94	35(3)	34(3)	40(3)	-4(3)	-8(3)	-6(3)
C95	62(5)	40(4)	35(4)	-7(3)	-12(3)	-11(3)
C96	54(4)	39(4)	38(4)	-11(3)	-10(3)	-9(3)
C97	33(3)	26(3)	41(3)	-8(2)	-11(2)	-5(2)
C98	25(3)	27(3)	45(3)	-9(2)	-8(2)	-8(2)
C99	34(4)	38(5)	78(6)	-3(4)	-15(4)	-10(3)
C100	30(3)	26(3)	54(4)	-14(3)	-2(3)	-6(3)
C101	48(4)	32(4)	58(5)	-11(3)	-17(4)	4(3)
C102	27(3)	27(3)	48(4)	-11(3)	-7(3)	-11(3)
C103	69(5)	43(4)	44(4)	-8(3)	-8(3)	-23(4)
N5	20(3)	35(4)	34(3)	-7(3)	-3(3)	6(3)
C104	31(3)	22(3)	38(3)	-11(2)	-6(2)	-10(2)
C105	30(4)	28(4)	40(5)	-6(3)	-4(3)	-8(3)
C106	21(4)	23(4)	37(4)	-10(3)	0(3)	-3(3)
C107	22(3)	23(3)	36(3)	-9(3)	-7(3)	-4(3)
C108	22(3)	28(4)	36(3)	-8(3)	-4(3)	-2(3)
C109	27(4)	23(4)	38(4)	-10(3)	-8(3)	-4(3)
C110	27(4)	24(4)	36(3)	-5(3)	-5(3)	-1(3)
C111	34(4)	28(4)	38(4)	-7(4)	-9(4)	0(3)
C112	38(5)	29(4)	39(4)	-5(4)	-7(4)	1(4)
C113	37(4)	26(4)	31(4)	2(3)	-5(3)	0(4)
C114	36(4)	27(4)	32(4)	-5(3)	-6(4)	4(4)
C115	32(4)	25(4)	32(4)	-6(3)	-4(3)	0(3)

N1A	26(4)	28(4)	37(4)	-6(4)	-8(3)	-1(4)
C12A	31(3)	22(3)	38(3)	-11(2)	-6(2)	-10(2)
C13A	29(5)	23(5)	35(5)	-11(4)	-10(4)	-7(4)
C14A	29(5)	26(5)	37(5)	-7(4)	-10(4)	-1(4)
C15A	25(4)	25(4)	36(4)	-8(3)	-6(3)	0(3)
C16A	22(4)	25(4)	36(4)	-9(3)	-6(3)	-6(4)
C17A	25(5)	25(5)	36(4)	-11(4)	-3(4)	-10(4)
C18A	28(4)	28(4)	37(4)	-6(3)	-7(3)	-2(4)
C19A	32(4)	26(4)	37(4)	-8(4)	-4(4)	-1(4)
C20A	37(5)	31(5)	40(5)	-6(4)	-7(4)	-2(4)
C21A	37(5)	32(5)	38(5)	-7(4)	-3(4)	-1(4)
C22A	34(5)	27(5)	38(5)	-6(4)	-6(4)	-2(4)
C23A	28(4)	25(5)	37(4)	-8(4)	-6(4)	-7(4)
O11	23(2)	27(2)	36(2)	-10(2)	-4(2)	-2(2)
O12	17(2)	38(3)	65(3)	-26(2)	-4(2)	-1(2)
N6	34(3)	42(4)	48(3)	-13(3)	-8(2)	1(3)
C116	25(3)	27(3)	38(3)	-10(2)	-3(2)	-6(2)
C117	26(3)	32(3)	34(3)	-9(2)	-4(2)	-3(2)
C118	33(3)	41(4)	30(3)	-4(3)	-6(2)	4(3)
C119	27(3)	39(4)	36(3)	-14(3)	-9(2)	7(3)
C120	25(3)	28(3)	38(3)	-12(2)	-3(2)	-7(2)
C121	22(3)	20(3)	37(3)	-10(2)	-5(2)	0(2)
C122	32(4)	38(4)	59(5)	-9(3)	2(3)	-13(3)
C123	20(3)	34(3)	40(3)	-16(3)	-5(2)	-4(2)
C124	24(3)	29(3)	49(4)	-16(3)	2(3)	-7(3)
C125	24(3)	23(3)	37(3)	-13(2)	-6(2)	-4(2)
C126	38(4)	30(3)	39(3)	-13(3)	-4(3)	-8(3)
C127	31(3)	21(3)	39(3)	-11(2)	-5(2)	-6(2)
C128	38(3)	30(4)	50(4)	-17(3)	-8(3)	-5(3)
C129	29(3)	36(4)	46(4)	-12(3)	-7(3)	-4(3)
C130	26(3)	25(3)	54(4)	-18(3)	-6(2)	-8(3)
C131	37(3)	22(3)	50(3)	-9(3)	-13(3)	-12(3)
C132	41(4)	20(3)	45(4)	-12(3)	-16(3)	3(3)
C133	43(4)	28(3)	45(3)	-10(3)	-10(3)	-14(3)
C134	45(4)	31(4)	63(4)	-12(3)	-5(3)	-2(3)

C135	55(5)	48(5)	56(4)	-25(4)	8(3)	-2(4)
C136	63(5)	54(5)	43(4)	-13(4)	-7(3)	-2(4)
C137	43(4)	47(5)	53(4)	-14(3)	-16(3)	2(3)
C138	41(4)	35(4)	52(4)	-17(3)	-2(3)	-3(3)
O13	23(2)	19(2)	46(3)	-7(2)	-6(2)	-7(2)
O14	22(2)	35(3)	65(4)	1(3)	-8(2)	-4(2)
N7	26(3)	34(3)	42(3)	-8(3)	-9(3)	8(3)
C150	26(3)	24(3)	43(3)	-8(2)	-8(2)	-9(2)
C151	22(4)	29(4)	46(4)	-4(3)	-5(3)	-7(3)
C152	21(3)	26(4)	47(4)	-8(3)	-7(3)	-2(3)
C153	21(3)	22(3)	45(3)	-7(3)	-9(3)	-1(3)
C154	21(3)	21(3)	45(3)	-5(3)	-6(3)	0(3)
C155	25(3)	22(3)	48(4)	-8(3)	-13(3)	-3(3)
C156	21(3)	22(3)	42(3)	-4(3)	-13(3)	2(3)
C157	27(3)	29(4)	38(4)	-8(3)	-15(3)	1(3)
C158	38(4)	29(4)	38(4)	-5(3)	-16(3)	6(3)
C159	36(4)	30(4)	45(4)	-5(3)	-11(3)	2(3)
C160	29(4)	32(4)	40(4)	-7(3)	-9(3)	4(3)
C161	27(3)	28(3)	40(3)	-6(3)	-9(3)	5(3)
N1F	24(5)	30(5)	42(5)	-7(4)	-9(4)	-2(4)
C12F	26(3)	24(3)	43(3)	-8(2)	-8(2)	-9(2)
C13F	27(5)	20(5)	42(5)	-13(4)	-11(4)	-10(4)
C14F	30(5)	30(5)	43(5)	-9(4)	-10(4)	1(5)
C15F	24(4)	27(4)	41(4)	-7(4)	-9(3)	-1(4)
C16F	23(4)	24(5)	42(4)	-6(4)	-6(4)	-6(4)
C17F	23(5)	29(5)	42(5)	-7(4)	-6(4)	-9(5)
C18F	24(4)	25(4)	40(4)	-6(4)	-8(3)	-1(4)
C19F	27(5)	27(5)	39(5)	-8(4)	-11(4)	1(4)
C20F	29(5)	29(5)	39(5)	-8(4)	-12(4)	0(5)
C21F	29(5)	33(5)	37(5)	-3(4)	-10(4)	3(5)
C22F	29(5)	27(5)	39(5)	-6(5)	-11(4)	3(5)
C23F	25(4)	27(5)	39(4)	-6(4)	-9(4)	-3(4)
C139	21(3)	22(3)	48(4)	-9(2)	-7(2)	-3(2)
C140	31(3)	23(3)	43(3)	-12(2)	-10(2)	-6(2)
C141	34(3)	40(4)	38(3)	-11(3)	-3(3)	-1(3)

C142	27(3)	34(4)	44(4)	-9(3)	-6(3)	-1(3)
C143	26(3)	25(3)	41(3)	-8(2)	-8(2)	-7(2)
C144	19(3)	18(3)	47(3)	-6(2)	-6(2)	-5(2)
C145	33(4)	29(3)	70(5)	-18(3)	-16(3)	-2(3)
C146	24(3)	32(4)	53(4)	-6(3)	-7(3)	-3(3)
C147	30(3)	31(4)	55(4)	-11(3)	-11(3)	-13(3)
C148	19(3)	26(3)	52(4)	-8(3)	-8(3)	-3(2)
C149	43(4)	26(3)	51(4)	-4(3)	-14(3)	-4(3)
O15	29(2)	34(2)	39(3)	-7(2)	-5(2)	-9(2)
O16	28(3)	31(3)	105(5)	-12(3)	-13(3)	-4(2)
N8	25(3)	27(3)	37(3)	-8(3)	-6(3)	-1(3)
C173	30(4)	21(3)	36(3)	-7(2)	-8(3)	-10(3)
C174	34(4)	22(4)	34(4)	-8(3)	-8(3)	-7(3)
C175	29(4)	29(4)	34(4)	-9(3)	-6(3)	-8(3)
C176	26(3)	22(4)	36(3)	-8(3)	-5(3)	-5(3)
C177	27(4)	21(3)	34(3)	-10(3)	-2(3)	-7(3)
C178	30(4)	23(4)	35(4)	-11(3)	-5(3)	-7(4)
C179	22(3)	21(3)	37(3)	-9(3)	-2(3)	-5(3)
C180	31(4)	24(4)	34(4)	-8(3)	-2(3)	-2(3)
C181	32(4)	31(4)	33(4)	-6(4)	-2(4)	-1(3)
C182	34(4)	33(4)	33(4)	-4(3)	-6(4)	2(4)
C183	35(4)	32(4)	35(4)	-3(4)	-4(3)	-1(4)
C184	28(4)	29(4)	37(4)	-5(3)	-4(3)	-6(3)
N1C	28(4)	30(5)	39(4)	-9(4)	-5(3)	-2(4)
C12C	30(4)	21(3)	36(3)	-7(2)	-8(3)	-10(3)
C13C	27(5)	24(5)	35(5)	-6(4)	-4(4)	-9(5)
C14C	25(5)	21(5)	35(5)	-7(4)	-3(4)	-5(4)
C15C	27(4)	25(4)	37(4)	-7(3)	-3(3)	-6(4)
C16C	27(4)	22(4)	35(4)	-7(4)	-5(3)	-6(4)
C17C	30(5)	20(5)	36(5)	-7(4)	-7(4)	-6(4)
C18C	27(4)	24(4)	35(4)	-4(4)	-3(3)	-2(4)
C19C	30(5)	28(5)	36(5)	-6(4)	-3(4)	-2(4)
C20C	36(5)	33(6)	38(5)	-2(5)	0(5)	1(5)
C21C	33(5)	31(6)	34(5)	-1(5)	0(4)	0(5)
C22C	30(5)	26(5)	36(5)	-5(4)	-2(4)	0(4)

C23C	27(4)	23(4)	36(4)	-6(4)	-3(3)	-6(4)
C162	31(3)	32(3)	50(4)	-9(3)	-14(3)	-12(3)
C163	34(3)	34(3)	42(3)	-11(3)	-6(3)	-5(3)
C164	45(4)	42(4)	40(4)	-5(3)	-7(3)	-11(3)
C165	57(4)	41(4)	40(4)	-1(3)	-10(3)	-14(3)
C166	44(4)	31(3)	40(3)	-4(3)	-7(3)	-14(3)
C167	31(3)	32(3)	50(4)	-8(3)	-11(3)	-16(3)
C168	34(4)	66(6)	87(7)	-18(5)	-9(4)	-6(4)
C169	38(4)	35(4)	72(5)	-5(3)	-21(3)	-7(3)
C170	67(5)	38(4)	61(5)	-16(4)	-5(4)	-8(4)
C171	41(4)	39(4)	44(4)	-8(3)	-10(3)	-21(3)
C172	75(5)	51(5)	46(4)	-11(3)	-4(3)	-31(4)

Table S11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for **5**.

	x	y	z	U(eq)
H2O	-1086	11457	8633	60
H1	3218	9663	4935	38
H13	-631	7967	6533	41
H14	-739	8021	5439	34
H17	2490	9255	6220	37
H19	-204	8353	4059	29
H20	585	8761	3033	33
H21	2389	9515	2807	40
H22	3351	9826	3654	36
H1D	-609	8072	4933	35
H13D	2314	9054	6711	39
H14D	2946	9450	5608	34
H17D	-606	7921	6186	34
H19D	3293	9762	4220	33
H20D	3075	9802	3141	36
H21D	1443	9173	2845	36
H22D	13	8493	3578	31
H2	-1239	9985	9308	45
H3A	-1291	8534	9949	60
H3B	61	8668	9736	60
H4A	-1352	7401	9380	55
H4B	2	7537	9165	55
H6	782	8533	8378	46
H7A	1155	10463	9048	81
H7B	1808	10022	8487	81
H7C	1377	9388	9151	81
H8A	340	10817	7810	44
H8B	-984	10548	7998	44
H9A	-2372	7626	8415	98

H9B	-1202	7349	8054	98
H9C	-1831	8263	7775	98
H10F	757	9706	7380	55
H10G	-478	9276	7357	55
H11E	1622	8283	7317	89
H11F	398	7796	7359	89
H4O	1451	6285	8669	61
H2N	4273	8065	5107	40
H25	2822	5989	9460	33
H26A	4153	5842	10215	44
H26B	4209	6934	9972	44
H27A	5930	5714	9737	44
H27B	6005	6809	9518	44
H29	5318	7560	8660	30
H30A	3245	8435	8641	56
H30B	3837	8103	9277	56
H30C	2526	7848	9250	56
H31A	2946	7362	7909	38
H31B	3046	6288	8154	38
H32A	6267	5000	8746	59
H32B	6960	5930	8456	59
H32C	5997	5578	8078	59
H33A	4719	6853	7586	34
H33B	6006	7114	7666	34
H34A	4165	8371	7503	40
H34B	5353	8670	7711	40
H36	6963	9050	6945	38
H37	7802	9407	5908	34
H40	4161	7936	6416	38
H42	8269	9616	4531	37
H43	8141	9684	3468	44
H44	6511	9116	3108	47
H45	4994	8468	3843	43
H6O	4043	4445	8677	60
H3N	3161	4622	5134	36

H59	-629	2780	6726	39
H60	-743	2895	5641	39
H63	2468	4140	6410	38
H65	-233	3305	4247	37
H66	521	3771	3225	39
H67	2296	4560	2978	37
H68	3281	4861	3837	37
H1EN	-647	3038	5126	37
H13E	2635	4089	6652	35
H14E	3104	4445	5584	36
H17E	-424	2923	6412	39
H19E	3190	4693	4227	34
H20E	2822	4712	3165	35
H21E	1034	4045	2943	36
H22E	-189	3432	3769	36
H48	2412	4316	9410	35
H49A	774	4041	10154	42
H49B	823	3093	9885	42
H50A	-789	3470	9417	44
H50B	-826	4425	9677	44
H52	227	3216	8554	37
H53A	2766	2560	9170	62
H53B	2300	2329	8542	62
H53C	1443	2313	9172	62
H54A	2931	3787	7855	38
H54B	2712	4731	8107	38
H55A	-852	5664	8735	55
H55B	-1293	4928	8351	55
H55C	-197	5557	8063	55
H56A	1293	4506	7531	35
H56B	-38	4260	7590	35
H57A	1762	2984	7433	42
H57B	438	2693	7538	42
H8O	-3320	9251	8647	75
H4N	-5711	13056	4984	32

H82	-2892	13948	6786	43
H83	-2100	14397	5730	36
H86	-5729	12889	6259	39
H88	-1794	14692	4352	32
H89	-1956	14762	3268	40
H90	-3597	14152	2936	39
H91	-5057	13477	3685	35
H1BN	-1837	14522	5106	31
H12B	-2572	14615	2935	36
H13B	-4424	13908	3122	39
H14B	-5255	13433	4145	35
H17B	-1627	14817	3794	34
H19B	-5835	13003	5474	34
H20B	-5785	12837	6569	38
H22B	-2585	14042	6348	36
H71	-3491	10373	9400	40
H72A	-3715	11470	10067	49
H72B	-4963	11496	9816	49
H73A	-4564	12928	9264	48
H73B	-3299	12886	9494	48
H75	-5061	12342	8465	43
H76A	-5790	10076	9129	86
H76B	-6197	10777	8544	86
H76C	-6053	11111	9200	86
H77A	-4471	10331	7895	51
H77B	-3219	10514	8079	51
H78A	-1932	13178	8539	72
H78B	-2961	13657	8183	72
H78C	-2231	12894	7889	72
H79A	-4706	11730	7462	52
H79B	-3420	12077	7455	52
H80A	-5373	13228	7442	81
H80B	-4116	13590	7488	81
H10O	6027	10747	1365	64
H94	6173	9634	701	44

H95A	6171	8499	58	54
H95B	4835	8477	322	54
H96A	5057	7049	883	51
H96B	6410	7102	659	51
H98	4217	7697	1658	38
H99A	3784	9977	993	75
H99B	3133	9243	1548	75
H99C	3553	8962	877	75
H10A	4616	9715	2209	43
H10B	5939	9541	2020	43
H10C	7415	6875	1640	67
H10D	6257	6360	1969	67
H10E	6795	7142	2279	67
H10H	4148	8332	2645	40
H10I	5439	8007	2669	40
H10J	3540	6788	2671	62
H10K	4832	6462	2695	62
H5N	1824	7170	5015	36
H105	5700	6128	3517	39
H106	5735	5704	4601	32
H109	2571	7319	3805	35
H111	5205	5401	5964	39
H112	4284	5377	7029	42
H113	2492	6069	7173	38
H114	1587	6769	6327	38
H1AN	5734	5574	5109	36
H13A	2610	7297	3475	34
H14A	1995	7193	4553	36
H17A	5593	6035	3823	33
H19A	1815	6840	5892	38
H20A	2132	6404	6875	43
H21A	3765	5676	7188	43
H22A	5266	5347	6397	39
H12O	3472	5545	1437	57
H6N	721	5565	5061	48

H117	2131	5717	657	36
H11A	776	6039	-88	42
H11B	728	6972	198	42
H11C	-1091	6604	633	39
H11D	-1012	5638	386	39
H121	-375	6870	1496	31
H12Q	1728	7712	1534	66
H12R	1093	7745	908	66
H12S	2408	7471	902	66
H12D	2033	6206	2236	36
H12E	1906	5279	1964	36
H12F	-1365	4400	1343	50
H12G	-2012	5159	1684	50
H12H	-1039	4564	2019	50
H12I	201	5561	2547	32
H12J	-1081	5883	2472	32
H12K	774	7018	2654	42
H12L	-422	7435	2468	42
H128	-2028	7382	3231	45
H129	-2774	7245	4256	44
H132	835	6037	3758	41
H134	-3244	6889	5647	55
H135	-3070	6452	6712	63
H136	-1460	5660	7057	63
H137	60	5342	6313	55
H14O	875	3791	1441	63
H7N	1798	2126	5001	40
H151	5600	1095	3415	39
H152	5719	707	4487	37
H155	2499	2245	3751	37
H157	5289	422	5872	36
H158	4438	417	6937	41
H159	2679	1076	7114	44
H160	1692	1750	6292	40
H1FN	5736	611	4963	38

H13F	2464	2189	3455	34
H14F	1712	2178	4514	40
H17F	5394	1086	3642	37
H19F	1912	1785	5717	36
H20F	2091	1433	6788	38
H21F	3671	785	7095	40
H22F	5294	410	6311	38
H140	2540	3981	667	38
H14T	4155	4022	-78	44
H14X	4073	2944	213	44
H14Y	5770	4153	384	42
H14Z	5710	3069	656	42
H144	4718	2421	1544	33
H14Q	2125	2095	965	63
H14R	2673	1548	1567	63
H14S	3455	1862	910	63
H14H	1994	2711	2234	44
H14I	2252	3771	1962	44
H14J	5829	4942	1305	57
H14K	6293	4009	1676	57
H14L	5226	4498	1991	57
H14M	3673	3233	2555	38
H14N	4998	2992	2502	38
H201	3159	1658	2675	48
H202	4474	1393	2567	48
H16O	-1647	-1459	1237	81
H8N	708	597	4964	35
H174	-2118	2262	3094	36
H175	-2790	2253	4118	36
H178	720	903	3666	34
H180	-3293	1891	5476	35
H181	-3106	1514	6568	38
H182	-1539	793	6939	40
H183	36	445	6242	41
H1CN	-3163	2059	4796	38

H13C	867	1144	3357	34
H203	841	751	4398	32
H17C	-2377	2168	3548	34
H19C	369	597	5752	37
H20C	-476	611	6800	44
H21C	-2275	1253	6942	40
H22C	-3292	1790	6121	37
H163	-1610	6	543	43
H16A	-1478	1439	-118	51
H16B	-202	1372	97	51
H16C	-603	2523	651	55
H16D	-1903	2546	473	55
H167	46	1492	1429	45
H16E	951	676	667	92
H16F	684	-391	720	92
H16G	1155	-19	1301	92
H16H	-433	-791	2003	57
H16I	-1725	-555	1874	57
H17X	-3118	2374	1455	82
H17Y	-2087	2606	1833	82
H17Z	-2837	1694	2081	82
H17G	-1532	768	2475	50
H17H	-294	326	2439	50
H17I	584	1658	2515	69
H17J	-521	2243	2358	69

Table S12. Torsion angles [°] for **5**.

C17-C12-C13-C14	-0.15(18)	C17D-C12D-C13D-C14D	0.02(19)
C11-C12-C13-C14	-172.5(7)	C11-C12D-C13D-C14D	179.2(7)
C12-C13-C14-C15	0.1(3)	C12D-C13D-C14D-C15D	0.0(3)
C13-C14-C15-C16	-0.1(4)	C13D-C14D-C15D-C16D	0.0(4)
C13-C14-C15-C18	179.8(2)	C13D-C14D-C15D-C18D	-179.9(3)
C23-N1-C16-C15	0.0(3)	C23D-N1D-C16D-C17D	-180.0(3)
C23-N1-C16-C17	179.9(2)	C23D-N1D-C16D-C15D	0.0(3)
C18-C15-C16-N1	0.0(3)	C14D-C15D-C16D-C17D	0.0(4)
C14-C15-C16-N1	179.9(2)	C18D-C15D-C16D-C17D	179.9(3)
C18-C15-C16-C17	-179.8(2)	C14D-C15D-C16D-N1D	-180.0(2)
C14-C15-C16-C17	0.1(4)	C18D-C15D-C16D-N1D	0.0(3)
N1-C16-C17-C12	-179.95(19)	N1D-C16D-C17D-C12D	179.9(2)
C15-C16-C17-C12	-0.2(4)	C15D-C16D-C17D-C12D	0.0(4)
C13-C12-C17-C16	0.2(2)	C13D-C12D-C17D-C16D	0.0(3)
C11-C12-C17-C16	171.9(8)	C11-C12D-C17D-C16D	-178.9(10)
C16-C15-C18-C19	179.9(3)	C14D-C15D-C18D-C19D	-0.2(6)
C14-C15-C18-C19	0.0(6)	C16D-C15D-C18D-C19D	179.9(3)
C16-C15-C18-C23	-0.1(3)	C14D-C15D-C18D-C23D	180.0(3)
C14-C15-C18-C23	-180.0(3)	C16D-C15D-C18D-C23D	0.1(3)
C15-C18-C19-C20	180.0(3)	C15D-C18D-C19D-C20D	-179.9(3)
C23-C18-C19-C20	-0.1(4)	C23D-C18D-C19D-C20D	-0.1(4)
C18-C19-C20-C21	0.0(4)	C18D-C19D-C20D-C21D	0.2(4)
C19-C20-C21-C22	0.2(4)	C19D-C20D-C21D-C22D	-0.2(4)
C20-C21-C22-C23	-0.2(4)	C20D-C21D-C22D-C23D	0.2(4)
C21-C22-C23-N1	-180.0(2)	C21D-C22D-C23D-N1D	180.0(3)
C21-C22-C23-C18	0.1(4)	C21D-C22D-C23D-C18D	-0.1(4)
C16-N1-C23-C22	-180.0(3)	C16D-N1D-C23D-C22D	-180.0(3)
C16-N1-C23-C18	-0.1(2)	C16D-N1D-C23D-C18D	0.1(3)
C19-C18-C23-C22	0.0(4)	C19D-C18D-C23D-C22D	0.1(4)
C15-C18-C23-C22	180.0(3)	C15D-C18D-C23D-C22D	180.0(3)
C19-C18-C23-N1	-179.9(2)	C19D-C18D-C23D-N1D	-180.0(2)
C15-C18-C23-N1	0.1(3)	C15D-C18D-C23D-N1D	-0.1(3)

C5-O1-C2-C3	-57.4(8)	C17-C12-C11-C12D	-122(12)
C5-O1-C2-C1	60.5(7)	C13-C12-C11-C10	-94.0(12)
C7-C1-C2-O1	-162.9(7)	C17-C12-C11-C10	94.2(12)
C8-C1-C2-O1	74.3(7)	C13D-C12D-C11-C12	53(12)
C6-C1-C2-O1	-43.4(7)	C17D-C12D-C11-C12	-128(12)
C7-C1-C2-C3	-54.3(10)	C13D-C12D-C11-C10	90.8(13)
C8-C1-C2-C3	-177.1(6)	C17D-C12D-C11-C10	-90.0(15)
C6-C1-C2-C3	65.2(8)	C6-C10-C11-C12	174.6(11)
O1-C2-C3-C4	36.0(9)	C6-C10-C11-C12D	170.7(14)
C1-C2-C3-C4	-73.2(9)	C28-O3-C25-C26	-57.3(6)
C2-C3-C4-C5	-0.1(9)	C28-O3-C25-C24	60.8(6)
C2-O1-C5-C9	178.4(8)	C30-C24-C25-O3	-164.9(6)
C2-O1-C5-C4	57.7(7)	C31-C24-C25-O3	73.5(6)
C2-O1-C5-C6	-53.6(7)	C29-C24-C25-O3	-43.3(6)
C3-C4-C5-O1	-34.7(8)	C30-C24-C25-C26	-55.2(8)
C3-C4-C5-C9	-150.6(10)	C31-C24-C25-C26	-176.8(6)
C3-C4-C5-C6	73.9(9)	C29-C24-C25-C26	66.4(7)
O1-C5-C6-C10	-99.4(8)	O3-C25-C26-C27	33.9(7)
C9-C5-C6-C10	20.9(12)	C24-C25-C26-C27	-74.6(7)
C4-C5-C6-C10	153.6(7)	C25-C26-C27-C28	2.1(7)
O1-C5-C6-C1	26.5(7)	C25-O3-C28-C32	178.7(6)
C9-C5-C6-C1	146.8(8)	C25-O3-C28-C27	57.1(6)
C4-C5-C6-C1	-80.6(8)	C25-O3-C28-C29	-54.1(6)
C7-C1-C6-C10	-101.3(9)	C26-C27-C28-O3	-36.1(7)
C2-C1-C6-C10	135.9(7)	C26-C27-C28-C32	-151.6(6)
C8-C1-C6-C10	21.5(10)	C26-C27-C28-C29	70.6(7)
C7-C1-C6-C5	131.9(8)	O3-C28-C29-C33	-102.8(6)
C2-C1-C6-C5	9.1(7)	C32-C28-C29-C33	16.4(9)
C8-C1-C6-C5	-105.3(7)	C27-C28-C29-C33	152.3(6)
C7-C1-C8-O2	-61.0(9)	O3-C28-C29-C24	26.6(7)
C2-C1-C8-O2	65.9(8)	C32-C28-C29-C24	145.8(6)
C6-C1-C8-O2	175.1(6)	C27-C28-C29-C24	-78.3(6)
C5-C6-C10-C11	-87.1(11)	C30-C24-C29-C33	-101.0(7)
C1-C6-C10-C11	154.7(10)	C31-C24-C29-C33	22.9(8)
C13-C12-C11-C12D	50(12)	C25-C24-C29-C33	137.1(6)

C30-C24-C29-C28	131.0(7)	C44-C45-C46-N2	178.1(8)
C31-C24-C29-C28	-105.1(6)	C44-C45-C46-C41	2.2(12)
C25-C24-C29-C28	9.1(6)	C39-N2-C46-C45	-176.9(8)
C30-C24-C31-O4	-55.8(7)	C39-N2-C46-C41	-0.5(9)
C25-C24-C31-O4	68.7(7)	C42-C41-C46-C45	-3.2(11)
C29-C24-C31-O4	177.8(5)	C38-C41-C46-C45	178.1(7)
C28-C29-C33-C34	-168.1(6)	C42-C41-C46-N2	-179.8(7)
C24-C29-C33-C34	70.7(8)	C38-C41-C46-N2	1.4(8)
C29-C33-C34-C35	169.1(6)	C63-C58-C59-C60	-0.33(18)
C33-C34-C35-C40	80.0(9)	C57-C58-C59-C60	-170.1(6)
C33-C34-C35-C36	-96.2(8)	C58-C59-C60-C61	0.2(3)
C40-C35-C36-C37	2.0(12)	C59-C60-C61-C62	-0.1(4)
C34-C35-C36-C37	178.2(7)	C59-C60-C61-C64	179.7(2)
C35-C36-C37-C38	-1.4(12)	C64-C61-C62-N3	0.0(3)
C36-C37-C38-C39	1.7(11)	C60-C61-C62-N3	179.9(2)
C36-C37-C38-C41	176.5(8)	C64-C61-C62-C63	-179.7(2)
C46-N2-C39-C38	-0.7(9)	C60-C61-C62-C63	0.1(4)
C46-N2-C39-C40	179.5(8)	C69-N3-C62-C61	0.0(2)
C37-C38-C39-N2	177.5(7)	C69-N3-C62-C63	179.8(2)
C41-C38-C39-N2	1.6(9)	C59-C58-C63-C62	0.3(3)
C37-C38-C39-C40	-2.6(12)	C57-C58-C63-C62	169.6(6)
C41-C38-C39-C40	-178.6(7)	C61-C62-C63-C58	-0.2(4)
C36-C35-C40-C39	-2.7(11)	N3-C62-C63-C58	-179.92(18)
C34-C35-C40-C39	-178.8(7)	C62-C61-C64-C65	179.8(3)
N2-C39-C40-C35	-177.1(8)	C60-C61-C64-C65	0.0(6)
C38-C39-C40-C35	3.1(12)	C62-C61-C64-C69	-0.1(3)
C39-C38-C41-C42	179.8(9)	C60-C61-C64-C69	-179.9(3)
C37-C38-C41-C42	4.5(16)	C61-C64-C65-C66	-180.0(3)
C39-C38-C41-C46	-1.8(8)	C69-C64-C65-C66	-0.1(4)
C37-C38-C41-C46	-177.1(8)	C64-C65-C66-C67	0.1(4)
C46-C41-C42-C43	2.3(11)	C65-C66-C67-C68	-0.1(4)
C38-C41-C42-C43	-179.4(9)	C66-C67-C68-C69	0.1(4)
C41-C42-C43-C44	-0.7(13)	C62-N3-C69-C68	-180.0(3)
C42-C43-C44-C45	-0.4(13)	C62-N3-C69-C64	-0.1(2)
C43-C44-C45-C46	-0.4(13)	C67-C68-C69-N3	179.8(2)

C67-C68-C69-C64	-0.1(4)	C16E-N1E-C23E-C18E	0.1(2)
C65-C64-C69-N3	-179.8(2)	C16E-N1E-C23E-C22E	179.9(3)
C61-C64-C69-N3	0.1(3)	C51-O5-C48-C47	60.4(6)
C65-C64-C69-C68	0.1(4)	C51-O5-C48-C49	-57.1(6)
C61-C64-C69-C68	-180.0(2)	C54-C47-C48-O5	75.2(7)
C17E-C12E-C13E-C14E	0.06(18)	C53-C47-C48-O5	-162.9(6)
C57-C12E-C13E-C14E	173.4(8)	C52-C47-C48-O5	-41.8(6)
C12E-C13E-C14E-C15E	-0.1(3)	C54-C47-C48-C49	-176.2(6)
C13E-C14E-C15E-C18E	-179.9(3)	C53-C47-C48-C49	-54.3(8)
C13E-C14E-C15E-C16E	0.1(4)	C52-C47-C48-C49	66.8(7)
C23E-N1E-C16E-C15E	-0.1(2)	O5-C48-C49-C50	34.0(7)
C23E-N1E-C16E-C17E	-179.9(2)	C47-C48-C49-C50	-74.4(8)
C18E-C15E-C16E-N1E	0.0(3)	C48-C49-C50-C51	0.8(8)
C14E-C15E-C16E-N1E	-179.9(2)	C48-O5-C51-C55	179.6(6)
C18E-C15E-C16E-C17E	179.9(2)	C48-O5-C51-C50	57.3(7)
C14E-C15E-C16E-C17E	-0.1(4)	C48-O5-C51-C52	-53.9(6)
C13E-C12E-C17E-C16E	-0.1(3)	C49-C50-C51-O5	-35.6(7)
C57-C12E-C17E-C16E	-172.5(9)	C49-C50-C51-C55	-152.5(7)
N1E-C16E-C17E-C12E	179.9(2)	C49-C50-C51-C52	72.0(7)
C15E-C16E-C17E-C12E	0.1(4)	O5-C51-C52-C56	-101.4(7)
C14E-C15E-C18E-C19E	-0.1(6)	C55-C51-C52-C56	18.2(9)
C16E-C15E-C18E-C19E	180.0(3)	C50-C51-C52-C56	152.3(6)
C14E-C15E-C18E-C23E	180.0(3)	O5-C51-C52-C47	27.6(7)
C16E-C15E-C18E-C23E	0.0(3)	C55-C51-C52-C47	147.2(6)
C23E-C18E-C19E-C20E	-0.1(4)	C50-C51-C52-C47	-78.7(7)
C15E-C18E-C19E-C20E	180.0(3)	C54-C47-C52-C56	21.7(9)
C18E-C19E-C20E-C21E	0.1(4)	C48-C47-C52-C56	135.8(6)
C19E-C20E-C21E-C22E	-0.1(4)	C53-C47-C52-C56	-102.1(8)
C20E-C21E-C22E-C23E	0.1(4)	C54-C47-C52-C51	-105.8(7)
C19E-C18E-C23E-C22E	0.1(4)	C48-C47-C52-C51	8.3(7)
C15E-C18E-C23E-C22E	-179.9(2)	C53-C47-C52-C51	130.4(7)
C19E-C18E-C23E-N1E	180.0(2)	C48-C47-C54-O6	66.9(8)
C15E-C18E-C23E-N1E	-0.1(3)	C53-C47-C54-O6	-58.0(8)
C21E-C22E-C23E-C18E	-0.1(4)	C52-C47-C54-O6	176.1(6)
C21E-C22E-C23E-N1E	-179.9(2)	C51-C52-C56-C57	-166.1(6)

C47-C52-C56-C57	73.6(8)	C90-C91-C92-N4	180.0(2)
C63-C58-C57-C56	81.8(7)	C90-C91-C92-C87	0.0(4)
C59-C58-C57-C56	-108.3(6)	C85-N4-C92-C91	180.0(3)
C63-C58-C57-C12E	-17(7)	C85-N4-C92-C87	-0.1(2)
C59-C58-C57-C12E	153(8)	C88-C87-C92-C91	0.0(4)
C52-C56-C57-C58	176.0(8)	C84-C87-C92-C91	-179.9(3)
C52-C56-C57-C12E	-176.9(12)	C88-C87-C92-N4	-180.0(2)
C17E-C12E-C57-C58	-26(7)	C84-C87-C92-N4	0.1(3)
C13E-C12E-C57-C58	161(8)	C17B-C12B-C13B-C14B	-0.05(18)
C17E-C12E-C57-C56	-109.7(9)	C12B-C13B-C14B-C15B	0.1(3)
C13E-C12E-C57-C56	76.9(9)	C13B-C14B-C15B-C18B	180.0(3)
C86-C81-C82-C83	0.02(19)	C13B-C14B-C15B-C16B	-0.1(4)
C80-C81-C82-C83	176.0(6)	C23B-N1B-C16B-C17B	180.0(3)
C81-C82-C83-C84	-0.1(3)	C23B-N1B-C16B-C15B	0.0(2)
C82-C83-C84-C87	180.0(3)	C14B-C15B-C16B-N1B	-180.0(2)
C82-C83-C84-C85	0.1(3)	C18B-C15B-C16B-N1B	0.0(3)
C92-N4-C85-C86	180.0(3)	C14B-C15B-C16B-C17B	0.0(4)
C92-N4-C85-C84	0.0(2)	C18B-C15B-C16B-C17B	-180.0(2)
C87-C84-C85-N4	0.0(3)	C13B-C12B-C17B-C16B	0.0(3)
C83-C84-C85-N4	179.9(2)	N1B-C16B-C17B-C12B	-180.0(2)
C87-C84-C85-C86	-179.9(3)	C15B-C16B-C17B-C12B	0.0(4)
C83-C84-C85-C86	0.0(4)	C14B-C15B-C18B-C23B	180.0(3)
C82-C81-C86-C85	0.1(3)	C16B-C15B-C18B-C23B	0.0(3)
C80-C81-C86-C85	-174.8(8)	C14B-C15B-C18B-C19B	-0.1(6)
N4-C85-C86-C81	-179.99(19)	C16B-C15B-C18B-C19B	179.9(3)
C84-C85-C86-C81	-0.1(4)	C23B-C18B-C19B-C20B	-0.1(4)
C85-C84-C87-C88	-180.0(3)	C15B-C18B-C19B-C20B	180.0(3)
C83-C84-C87-C88	0.1(6)	C18B-C19B-C20B-C21B	0.1(4)
C85-C84-C87-C92	0.0(3)	C19B-C20B-C21B-C22B	0.0(4)
C83-C84-C87-C92	-180.0(3)	C19B-C20B-C21B-C80	-179.2(7)
C84-C87-C88-C89	179.9(3)	C20B-C21B-C22B-C23B	0.0(4)
C92-C87-C88-C89	0.0(4)	C80-C21B-C22B-C23B	178.9(10)
C87-C88-C89-C90	0.0(4)	C15B-C18B-C23B-C22B	-180.0(3)
C88-C89-C90-C91	-0.1(4)	C19B-C18B-C23B-C22B	0.1(4)
C89-C90-C91-C92	0.1(4)	C15B-C18B-C23B-N1B	0.0(3)

C19B-C18B-C23B-N1B	-179.9(2)	C71-C70-C77-O8	63.2(8)
C21B-C22B-C23B-C18B	0.0(4)	C75-C70-C77-O8	171.7(6)
C21B-C22B-C23B-N1B	-180.0(2)	C74-C75-C79-C80	-82.8(10)
C16B-N1B-C23B-C18B	0.0(2)	C70-C75-C79-C80	157.6(9)
C16B-N1B-C23B-C22B	180.0(2)	C86-C81-C80-C21B	160(6)
C74-O7-C71-C72	-57.9(6)	C82-C81-C80-C21B	-16(6)
C74-O7-C71-C70	60.1(6)	C86-C81-C80-C79	80.4(13)
C77-C70-C71-O7	74.8(7)	C82-C81-C80-C79	-95.1(12)
C76-C70-C71-O7	-162.9(7)	C22B-C21B-C80-C81	166(7)
C75-C70-C71-O7	-42.7(7)	C20B-C21B-C80-C81	-15(6)
C77-C70-C71-C72	-176.2(7)	C22B-C21B-C80-C79	-90.1(15)
C76-C70-C71-C72	-53.9(10)	C20B-C21B-C80-C79	88.9(12)
C75-C70-C71-C72	66.4(8)	C75-C79-C80-C81	175.7(12)
O7-C71-C72-C73	34.0(8)	C75-C79-C80-C21B	167.4(17)
C70-C71-C72-C73	-75.2(8)	C97-O9-C94-C95	-55.9(7)
C71-C72-C73-C74	2.0(8)	C97-O9-C94-C93	59.7(6)
C71-O7-C74-C78	178.4(6)	C99-C93-C94-O9	-165.1(6)
C71-O7-C74-C73	58.9(6)	C100-C93-C94-O9	75.2(7)
C71-O7-C74-C75	-53.6(6)	C98-C93-C94-O9	-42.3(6)
C72-C73-C74-O7	-37.1(7)	C99-C93-C94-C95	-56.9(9)
C72-C73-C74-C78	-153.5(7)	C100-C93-C94-C95	-176.6(7)
C72-C73-C74-C75	72.4(8)	C98-C93-C94-C95	65.9(8)
O7-C74-C75-C79	-100.5(7)	O9-C94-C95-C96	31.4(8)
C78-C74-C75-C79	21.4(11)	C93-C94-C95-C96	-75.7(9)
C73-C74-C75-C79	152.5(7)	C94-C95-C96-C97	3.9(8)
O7-C74-C75-C70	26.7(7)	C94-O9-C97-C101	178.0(6)
C78-C74-C75-C70	148.5(7)	C94-O9-C97-C96	57.9(7)
C73-C74-C75-C70	-80.3(7)	C94-O9-C97-C98	-53.3(7)
C77-C70-C75-C79	23.3(9)	C95-C96-C97-O9	-37.2(8)
C76-C70-C75-C79	-100.3(9)	C95-C96-C97-C101	-153.5(7)
C71-C70-C75-C79	137.2(7)	C95-C96-C97-C98	69.8(8)
C77-C70-C75-C74	-104.8(7)	O9-C97-C98-C102	-102.0(6)
C76-C70-C75-C74	131.6(8)	C101-C97-C98-C102	19.2(9)
C71-C70-C75-C74	9.1(7)	C96-C97-C98-C102	152.7(6)
C76-C70-C77-O8	-62.9(9)	O9-C97-C98-C93	26.0(7)

C101-C97-C98-C93	147.2(7)	C108-C107-C110-C111	180.0(3)
C96-C97-C98-C93	-79.3(7)	C106-C107-C110-C115	180.0(3)
C99-C93-C98-C102	-98.7(9)	C108-C107-C110-C115	-0.1(3)
C94-C93-C98-C102	137.0(6)	C115-C110-C111-C112	0.0(4)
C100-C93-C98-C102	23.4(9)	C107-C110-C111-C112	180.0(3)
C99-C93-C98-C97	133.5(8)	C110-C111-C112-C113	0.0(4)
C94-C93-C98-C97	9.2(7)	C111-C112-C113-C114	0.0(4)
C100-C93-C98-C97	-104.5(7)	C112-C113-C114-C115	-0.1(4)
C99-C93-C100-O10	-60.6(8)	C113-C114-C115-N5	179.9(2)
C94-C93-C100-O10	64.2(8)	C113-C114-C115-C110	0.1(4)
C98-C93-C100-O10	174.0(6)	C108-N5-C115-C114	-179.9(3)
C97-C98-C102-C103	-80.4(9)	C108-N5-C115-C110	-0.1(2)
C93-C98-C102-C103	159.4(7)	C111-C110-C115-C114	-0.1(4)
C98-C102-C103-C12A	173.9(13)	C107-C110-C115-C114	180.0(2)
C98-C102-C103-C104	180.0(9)	C111-C110-C115-N5	-179.9(2)
C12A-C103-C104-C109	-143(8)	C107-C110-C115-N5	0.1(3)
C102-C103-C104-C109	82.7(9)	C104-C103-C12A-C17A	-141(8)
C12A-C103-C104-C105	33(8)	C102-C103-C12A-C17A	-92.9(13)
C102-C103-C104-C105	-101.8(8)	C104-C103-C12A-C13A	35(8)
C109-C104-C105-C106	-0.16(18)	C102-C103-C12A-C13A	82.9(11)
C103-C104-C105-C106	-176.3(5)	C103-C12A-C13A-C14A	-177.0(7)
C104-C105-C106-C107	0.1(3)	C17A-C12A-C13A-C14A	0.00(17)
C105-C106-C107-C108	-0.1(4)	C12A-C13A-C14A-C15A	0.0(3)
C105-C106-C107-C110	179.8(3)	C13A-C14A-C15A-C18A	-180.0(2)
C115-N5-C108-C109	179.9(2)	C13A-C14A-C15A-C16A	0.0(3)
C115-N5-C108-C107	0.0(2)	C23A-N1A-C16A-C17A	-179.9(3)
C106-C107-C108-C109	0.1(4)	C23A-N1A-C16A-C15A	-0.1(2)
C110-C107-C108-C109	-179.8(3)	C14A-C15A-C16A-C17A	0.0(4)
C106-C107-C108-N5	180.0(2)	C18A-C15A-C16A-C17A	179.9(2)
C110-C107-C108-N5	0.0(3)	C14A-C15A-C16A-N1A	-179.9(2)
N5-C108-C109-C104	-179.97(18)	C18A-C15A-C16A-N1A	0.0(3)
C107-C108-C109-C104	-0.1(4)	N1A-C16A-C17A-C12A	179.92(19)
C105-C104-C109-C108	0.2(3)	C15A-C16A-C17A-C12A	0.1(4)
C103-C104-C109-C108	175.3(6)	C103-C12A-C17A-C16A	175.7(10)
C106-C107-C110-C111	0.1(5)	C13A-C12A-C17A-C16A	0.0(3)

C14A-C15A-C18A-C23A	180.0(3)	C124-C120-C121-C125	16.4(10)
C16A-C15A-C18A-C23A	0.0(3)	C119-C120-C121-C125	150.5(6)
C14A-C15A-C18A-C19A	-0.1(5)	O11-C120-C121-C116	26.4(7)
C16A-C15A-C18A-C19A	180.0(3)	C124-C120-C121-C116	145.4(7)
C23A-C18A-C19A-C20A	-0.1(4)	C119-C120-C121-C116	-80.5(7)
C15A-C18A-C19A-C20A	179.9(3)	C123-C116-C121-C125	23.2(8)
C18A-C19A-C20A-C21A	0.1(4)	C122-C116-C121-C125	-101.7(7)
C19A-C20A-C21A-C22A	-0.1(4)	C117-C116-C121-C125	137.0(6)
C20A-C21A-C22A-C23A	0.0(3)	C123-C116-C121-C120	-104.5(6)
C16A-N1A-C23A-C22A	180.0(2)	C122-C116-C121-C120	130.6(7)
C16A-N1A-C23A-C18A	0.1(2)	C117-C116-C121-C120	9.3(7)
C21A-C22A-C23A-N1A	-179.9(2)	C122-C116-C123-O12	-56.4(8)
C21A-C22A-C23A-C18A	0.0(3)	C117-C116-C123-O12	67.6(7)
C15A-C18A-C23A-N1A	0.0(3)	C121-C116-C123-O12	177.5(5)
C19A-C18A-C23A-N1A	180.0(2)	C120-C121-C125-C126	-168.9(6)
C15A-C18A-C23A-C22A	-180.0(2)	C116-C121-C125-C126	71.1(8)
C19A-C18A-C23A-C22A	0.0(4)	C121-C125-C126-C127	170.5(6)
C120-O11-C117-C118	-56.5(6)	C125-C126-C127-C132	84.3(9)
C120-O11-C117-C116	59.1(6)	C125-C126-C127-C128	-96.5(8)
C123-C116-C117-O11	75.6(6)	C132-C127-C128-C129	1.3(11)
C122-C116-C117-O11	-162.7(6)	C126-C127-C128-C129	-177.9(7)
C121-C116-C117-O11	-42.2(6)	C127-C128-C129-C130	-0.3(12)
C123-C116-C117-C118	-177.5(6)	C128-C129-C130-C131	-0.4(11)
C122-C116-C117-C118	-55.8(8)	C128-C129-C130-C133	178.3(8)
C121-C116-C117-C118	64.7(7)	C138-N6-C131-C132	177.7(7)
O11-C117-C118-C119	32.1(7)	C138-N6-C131-C130	-0.9(9)
C116-C117-C118-C119	-74.4(7)	C129-C130-C131-N6	178.9(7)
C117-C118-C119-C120	3.5(7)	C133-C130-C131-N6	-0.2(8)
C117-O11-C120-C124	179.7(6)	C129-C130-C131-C132	0.2(10)
C117-O11-C120-C121	-53.6(6)	C133-C130-C131-C132	-178.9(6)
C117-O11-C120-C119	59.3(6)	C128-C127-C132-C131	-1.5(10)
C118-C119-C120-O11	-38.0(6)	C126-C127-C132-C131	177.7(6)
C118-C119-C120-C124	-152.6(6)	N6-C131-C132-C127	-177.6(7)
C118-C119-C120-C121	71.4(7)	C130-C131-C132-C127	0.8(10)
O11-C120-C121-C125	-102.5(7)	C129-C130-C133-C138	-177.7(9)

C131-C130-C133-C138	1.2(8)	C161-C156-C157-C158	-0.1(4)
C129-C130-C133-C134	0.1(15)	C153-C156-C157-C158	-179.9(3)
C131-C130-C133-C134	179.0(8)	C156-C157-C158-C159	0.0(4)
C138-C133-C134-C135	-0.9(12)	C157-C158-C159-C160	0.0(4)
C130-C133-C134-C135	-178.5(8)	C158-C159-C160-C161	0.0(4)
C133-C134-C135-C136	-1.1(14)	C154-N7-C161-C156	-0.1(2)
C134-C135-C136-C137	1.4(16)	C154-N7-C161-C160	180.0(2)
C135-C136-C137-C138	0.3(15)	C157-C156-C161-N7	-179.8(2)
C136-C137-C138-N6	-179.2(9)	C153-C156-C161-N7	0.1(3)
C136-C137-C138-C133	-2.4(14)	C157-C156-C161-C160	0.2(4)
C131-N6-C138-C137	178.8(8)	C153-C156-C161-C160	-180.0(2)
C131-N6-C138-C133	1.7(9)	C159-C160-C161-N7	179.8(2)
C134-C133-C138-C137	2.8(13)	C159-C160-C161-C156	-0.1(4)
C130-C133-C138-C137	-179.0(8)	C17F-C12F-C13F-C14F	0.1(2)
C134-C133-C138-N6	-180.0(7)	C149-C12F-C13F-C14F	178.5(9)
C130-C133-C138-N6	-1.8(9)	C12F-C13F-C14F-C15F	0.0(3)
C155-C150-C151-C152	-0.36(17)	C13F-C14F-C15F-C16F	0.0(3)
C149-C150-C151-C152	-170.6(5)	C13F-C14F-C15F-C18F	-179.9(3)
C150-C151-C152-C153	0.3(3)	C23F-N1F-C16F-C17F	-179.9(3)
C151-C152-C153-C154	-0.2(3)	C23F-N1F-C16F-C15F	-0.1(3)
C151-C152-C153-C156	179.6(2)	C18F-C15F-C16F-C17F	179.9(3)
C161-N7-C154-C155	179.7(3)	C14F-C15F-C16F-C17F	0.0(4)
C161-N7-C154-C153	0.1(2)	C18F-C15F-C16F-N1F	0.0(3)
C152-C153-C154-C155	0.2(4)	C14F-C15F-C16F-N1F	-179.9(2)
C156-C153-C154-C155	-179.7(2)	N1F-C16F-C17F-C12F	179.9(2)
C152-C153-C154-N7	179.9(2)	C15F-C16F-C17F-C12F	0.1(4)
C156-C153-C154-N7	0.0(3)	C13F-C12F-C17F-C16F	-0.1(3)
N7-C154-C155-C150	-179.85(19)	C149-C12F-C17F-C16F	-178.3(10)
C153-C154-C155-C150	-0.3(4)	C16F-C15F-C18F-C23F	0.0(3)
C151-C150-C155-C154	0.4(3)	C14F-C15F-C18F-C23F	179.9(3)
C149-C150-C155-C154	169.9(5)	C16F-C15F-C18F-C19F	180.0(3)
C152-C153-C156-C157	-0.1(5)	C14F-C15F-C18F-C19F	-0.1(5)
C154-C153-C156-C157	179.8(3)	C23F-C18F-C19F-C20F	0.0(5)
C152-C153-C156-C161	-179.9(3)	C15F-C18F-C19F-C20F	-180.0(3)
C154-C153-C156-C161	0.0(3)	C18F-C19F-C20F-C21F	0.0(4)

C19F-C20F-C21F-C22F	-0.1(4)
C20F-C21F-C22F-C23F	0.1(4)
C19F-C18F-C23F-C22F	0.0(5)
C15F-C18F-C23F-C22F	180.0(3)
C19F-C18F-C23F-N1F	180.0(2)
C15F-C18F-C23F-N1F	-0.1(3)
C21F-C22F-C23F-C18F	0.0(4)
C21F-C22F-C23F-N1F	-180.0(2)
C16F-N1F-C23F-C18F	0.1(3)
C16F-N1F-C23F-C22F	-180.0(3)
C143-O13-C140-C141	-56.8(7)
C143-O13-C140-C139	59.6(6)
C145-C139-C140-O13	-163.1(6)
C146-C139-C140-O13	76.2(7)
C144-C139-C140-O13	-41.3(7)
C145-C139-C140-C141	-54.1(8)
C146-C139-C140-C141	-174.9(6)
C144-C139-C140-C141	67.6(7)
O13-C140-C141-C142	33.5(7)
C139-C140-C141-C142	-74.4(7)
C140-C141-C142-C143	1.7(8)
C140-O13-C143-C147	178.0(6)
C140-O13-C143-C142	56.9(6)
C140-O13-C143-C144	-54.9(6)
C141-C142-C143-O13	-35.6(7)
C141-C142-C143-C147	-152.5(7)
C141-C142-C143-C144	72.7(7)
O13-C143-C144-C148	-101.0(7)
C147-C143-C144-C148	20.0(10)
C142-C143-C144-C148	153.0(6)
O13-C143-C144-C139	27.8(7)
C147-C143-C144-C139	148.8(7)
C142-C143-C144-C139	-78.2(7)
C145-C139-C144-C148	-102.2(8)
C146-C139-C144-C148	23.2(8)

C140-C139-C144-C148	135.9(6)
C145-C139-C144-C143	129.4(7)
C146-C139-C144-C143	-105.2(7)
C140-C139-C144-C143	7.5(7)
C145-C139-C146-O14	-57.2(8)
C140-C139-C146-O14	66.3(7)
C144-C139-C146-O14	175.2(5)
C143-C144-C148-C149	-166.7(6)
C139-C144-C148-C149	73.0(8)
C13F-C12F-C149-C150	133(10)
C17F-C12F-C149-C150	-49(10)
C13F-C12F-C149-C148	79.5(11)
C17F-C12F-C149-C148	-102.4(10)
C155-C150-C149-C12F	-44(10)
C151-C150-C149-C12F	125(10)
C155-C150-C149-C148	84.8(7)
C151-C150-C149-C148	-105.8(6)
C144-C148-C149-C12F	-178.8(15)
C144-C148-C149-C150	175.4(8)
C178-C173-C174-C175	3(3)
C172-C173-C174-C175	178.3(13)
C173-C174-C175-C176	-2.2(19)
C174-C175-C176-C179	-178.3(13)
C174-C175-C176-C177	2.4(16)
C184-N8-C177-C178	-178.0(14)
C184-N8-C177-C176	-0.1(12)
C179-C176-C177-C178	176.8(11)
C175-C176-C177-C178	-3.7(17)
C179-C176-C177-N8	-1.3(12)
C175-C176-C177-N8	178.2(9)
N8-C177-C178-C173	-177.8(15)
C176-C177-C178-C173	5(2)
C174-C173-C178-C177	-4(3)
C172-C173-C178-C177	-179.0(16)
C175-C176-C179-C184	-177.1(14)

C177-C176-C179-C184	2.2(13)	C23C-C18C-C19C-C20C	0(4)
C175-C176-C179-C180	3(2)	C15C-C18C-C19C-C20C	176(3)
C177-C176-C179-C180	-177.5(11)	C18C-C19C-C20C-C21C	-2(4)
C176-C179-C180-C181	-179.4(12)	C19C-C20C-C21C-C22C	2(4)
C184-C179-C180-C181	1.0(16)	C20C-C21C-C22C-C23C	0(4)
C179-C180-C181-C182	-2.2(18)	C16C-N1C-C23C-C18C	-2(3)
C180-C181-C182-C183	2(2)	C16C-N1C-C23C-C22C	173(2)
C181-C182-C183-C184	0.4(19)	C19C-C18C-C23C-N1C	177(2)
C177-N8-C184-C183	177.7(12)	C15C-C18C-C23C-N1C	0(3)
C177-N8-C184-C179	1.4(13)	C19C-C18C-C23C-C22C	2(4)
C182-C183-C184-N8	-177.4(12)	C15C-C18C-C23C-C22C	-176(2)
C182-C183-C184-C179	-1.6(19)	C21C-C22C-C23C-N1C	-176(2)
C176-C179-C184-N8	-2.2(14)	C21C-C22C-C23C-C18C	-1(4)
C180-C179-C184-N8	177.5(9)	C166-O15-C163-C162	60.8(6)
C176-C179-C184-C183	-178.8(12)	C166-O15-C163-C164	-57.6(6)
C180-C179-C184-C183	1.0(18)	C168-C162-C163-O15	-164.1(7)
C17C-C12C-C13C-C14C	5(6)	C169-C162-C163-O15	74.0(8)
C172-C12C-C13C-C14C	-175(3)	C167-C162-C163-O15	-43.1(6)
C12C-C13C-C14C-C15C	-5(5)	C168-C162-C163-C164	-54.5(10)
C13C-C14C-C15C-C16C	1(4)	C169-C162-C163-C164	-176.4(7)
C13C-C14C-C15C-C18C	180(3)	C167-C162-C163-C164	66.6(8)
C23C-N1C-C16C-C17C	174(3)	O15-C163-C164-C165	33.1(7)
C23C-N1C-C16C-C15C	4(3)	C162-C163-C164-C165	-76.6(9)
C14C-C15C-C16C-N1C	175(2)	C163-C164-C165-C166	3.4(8)
C18C-C15C-C16C-N1C	-4(3)	C163-O15-C166-C165	59.7(7)
C14C-C15C-C16C-C17C	3(4)	C163-O15-C166-C170	178.3(7)
C18C-C15C-C16C-C17C	-176(2)	C163-O15-C166-C167	-53.6(7)
N1C-C16C-C17C-C12C	-174(3)	C164-C165-C166-O15	-38.7(8)
C15C-C16C-C17C-C12C	-4(4)	C164-C165-C166-C170	-153.5(7)
C13C-C12C-C17C-C16C	0(5)	C164-C165-C166-C167	69.9(8)
C172-C12C-C17C-C16C	-180(5)	O15-C166-C167-C171	-100.6(7)
C14C-C15C-C18C-C19C	6(5)	C165-C166-C167-C171	152.5(6)
C16C-C15C-C18C-C19C	-175(3)	C170-C166-C167-C171	19.4(9)
C14C-C15C-C18C-C23C	-177(2)	O15-C166-C167-C162	27.4(7)
C16C-C15C-C18C-C23C	2(3)	C165-C166-C167-C162	-79.5(7)

C170-C166-C167-C162	147.4(7)	C162-C167-C171-C172	152.1(8)
C168-C162-C167-C171	-101.1(9)	C178-C173-C172-C171	91(2)
C163-C162-C167-C171	135.6(7)	C174-C173-C172-C171	-84(2)
C169-C162-C167-C171	20.1(9)	C178-C173-C172-C12C	170(25)
C168-C162-C167-C166	132.2(8)	C174-C173-C172-C12C	-5(22)
C163-C162-C167-C166	8.8(7)	C167-C171-C172-C173	164.0(12)
C169-C162-C167-C166	-106.7(7)	C167-C171-C172-C12C	157(2)
C168-C162-C169-O16	-62.8(9)	C13C-C12C-C172-C173	-2(21)
C163-C162-C169-O16	63.8(9)	C17C-C12C-C172-C173	177(29)
C167-C162-C169-O16	173.2(6)	C13C-C12C-C172-C171	102(3)
C166-C167-C171-C172	-88.5(9)	C17C-C12C-C172-C171	-79(7)

Table S13. Hydrogen bonds for **5** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O2-H2O...O7	0.84	1.97	2.805(7)	175.3
O4-H4O...O5	0.84	1.94	2.776(8)	174.3
O6-H6O...O3	0.84	1.95	2.789(8)	175.0
O8-H8O...O1	0.84	1.91	2.740(9)	170.0
O10-H10O...O15#1	0.84	1.95	2.784(7)	173.1
O12-H12O...O13	0.84	1.98	2.814(8)	176.5
O14-H14O...O11	0.84	1.94	2.776(8)	175.1
O16-H16O...O9#2	0.84	1.95	2.765(8)	163.2

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y+1,z #2 x-1,y-1,z

Figure S1. View of molecule 2 of **5** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level.

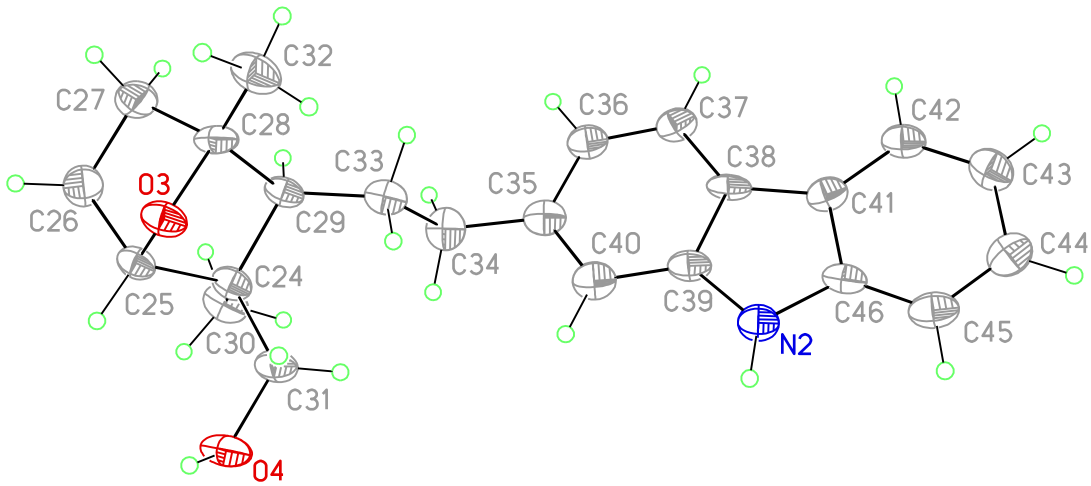
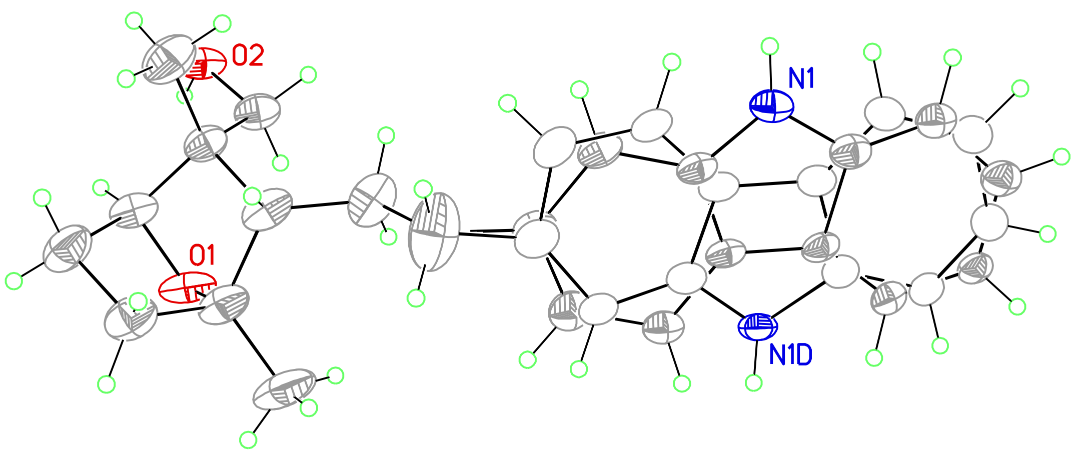


Figure S2. View of molecule 1 of **5** showing the partial atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The disorder of the carbazole group can be seen in the figure.



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(17) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection. $R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$. $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

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