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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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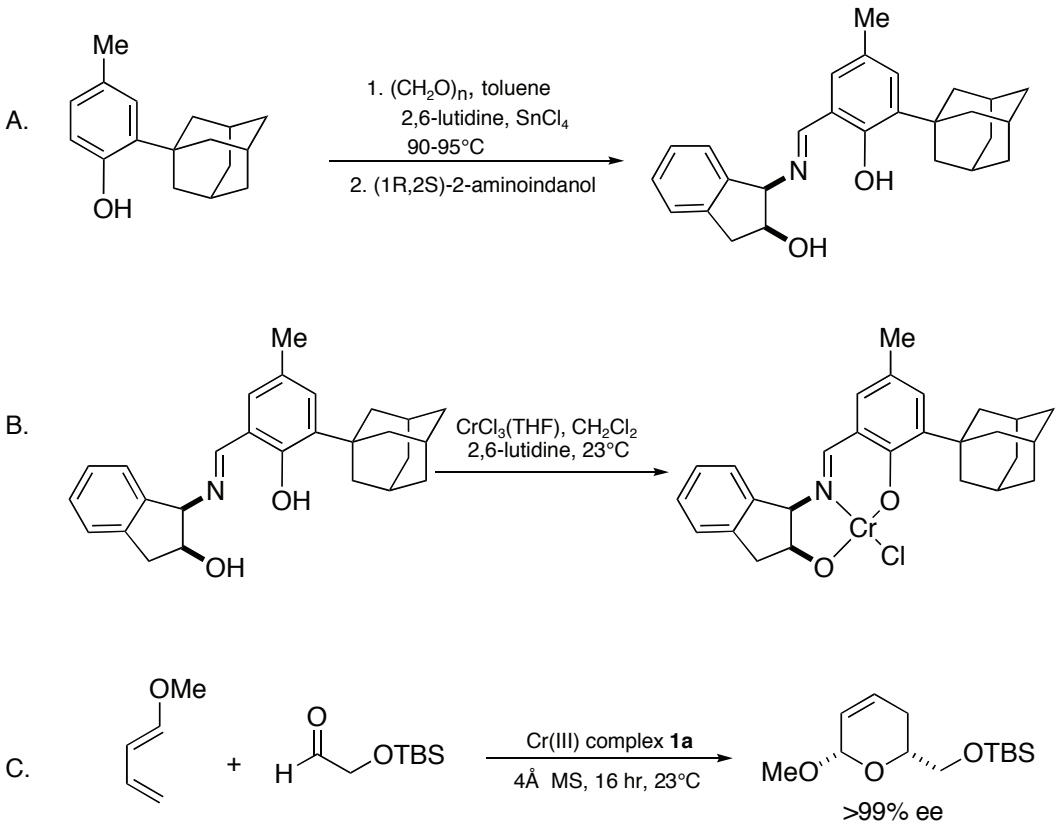
AN EFFICIENT, HIGHLY DIASTEREO- AND ENANTIOSELECTIVE HETERO-DIELS-ALDER CATALYST. PREPARATION OF (2*S*,6*R*)-6-(*tert*-BUTYLDIMETHYL-SILYLOXYMETHYL)-2-METHOXY-2,5-DIHYDROPYRAN (Silane, [[(2*R*,6*S*)-3,6-dihydro-6-methoxy-2H-pyran-2-yl]methoxy]-(1,1-dimethylethyl)dimethyl)-



Submitted by David E. Chavez and Eric N. Jacobsen.[1]
Checked by E. J. J. Grabowski and Michele Kubryk.

1. Procedure

A. (1R,2S)-1-[3-Adamantyl)-2-hydroxy-5-methylbenzyliden-amino]indan-2-ol. An oven-dried, 300-mL, three-necked, round-bottomed flask is equipped with a magnetic stirbar, fitted with a reflux condenser and thermometer, and purged with a nitrogen atmosphere by means of an inlet fitted to the condenser. The flask is charged with 2-adamantyl-4-methylphenol (12.1 g, 50.0 mmol, 1 eq) (Note 1), freshly distilled toluene (110 mL) (Note 2), and 2,6-lutidine (4.28 g, 4.67 mL, 40.00 mmol, 0.8 eq);

the open neck of the flask is capped with a septum. Neat stannic chloride ($SnCl_4$) (2.60 g, 1.17 mL, 10.00 mmol, 0.2 eq) is added by syringe over 10 min (Note 3). The solution turns pale yellow in color, and a pale yellow precipitate is also observable. The mixture is allowed to stir at room temperature for 20 min, then the septum is removed and solid paraformaldehyde (6.00 g, 200 mmol) is added in one portion against a gentle nitrogen counterflow (Note 4). The mixture is stirred an additional 10 min, the nitrogen inlet is replaced with a nitrogen balloon, the reaction flask is placed in a 90-95 °C bath, and heating is maintained at this temperature for 6 h. The reaction mixture is then allowed to cool to room temperature and filtered through a pad of premixed Celite® and silica gel (1:1, 12 g). The filter pad is washed with ethyl acetate (200 mL), and the combined organic filtrates are washed with water (350 mL), 1N HCl (350 mL), and brine (350 mL), and then dried over anhydrous Na_2SO_4. Concentration is effected by rotary evaporation, followed by removal of trace solvent on a high vacuum pump (0.5 mm) (13.4 g crude, 99.5%) (Note 5). Absolute ethanol (200 mL) is added and the mixture is heated gently until complete dissolution occurs (Note 6). (1*R*,2*S*)-1-Amino-2-indanol (7.83 g, 52.50 mmol, 1.05 equiv. Note 2) is added in one portion. The reaction mixture is then heated at 80 °C for 45 min, cooled to room temperature, and allowed to stand for 3-5 hours. The yellow solid product is isolated filtration, washed with cold ethanol (50 mL), and dried in the air (15.1 g, 75.2% over 2 steps) (Note 7).

*B. Chromium(III) Cl complex (**1a**)*. To a 200-mL round-bottomed flask is added chromium(III) chloride-tetrahydrofuran complex (1:3) (2.80 g, 7.48 mmol, 1 equiv.) and (1*R,*2*S*)-1-[3-adamantyl)-2-hydroxy-5-methylbenzyliden-amino]indan-2-ol (3.00 g, 7.48 mmol, 1 equiv.). The reaction mixture is placed under a nitrogen atmosphere, and dichloromethane (CH_2Cl_2, 60 mL) is added followed by dropwise addition of 2,6-lutidine (1.74 mL, 14.96 mmol, 2 equiv). The solution is stirred for 3 h, diluted with CH_2Cl_2 (300 mL), and washed with water (3 x 180 mL), then brine (180 mL) (Note 8). The organic phase is dried over anhydrous Na_2SO_4, filtered, and concentrated by rotary evaporation. The resulting solid is triturated with acetone (10 mL), filtered, washed with an additional portion of acetone (10 mL), and air-dried to give the chromium complex (**1a**) as a brown solid (2.3 g). Water (2 mL) is added to the filtrate (Note 9) and the solution allowed to stand uncovered at 23 °C overnight. The resulting precipitate is filtered and washed with cold acetone to give an

additional 600-800 mg of the chromium complex (**1a**) (combined yield 2.9-3.1 g, 80 – 85%) (Notes 10, 11).

C. (2S,6R)-6-(tert-Butyldimethylsilyloxymethyl)-2-methoxy-2,5-dihydro-pyran. 1-Methoxybutadiene (2.40 g, 2.89 mL, 28.7 mmol, 1.11 equiv.) is added dropwise to a stirring mixture of (*tert*-butyldimethylsilyloxy)acetaldehyde (90%, 5.00 g, 5.46 mL, 25.8 mmol, 1 equiv.), (1*R*, 2*S*) chromium(III) chloride complex (**1a**) (200 mg, 0.19 mmol, 1.5 mol% (Note 12) and 4Å molecular sieves (Note 13) under N_2 at 0 °C. The reaction mixture is allowed to stir at 0 °C for 1 h and then warmed to room temperature and allowed to stir for an additional 16 h. Distillation of this mixture (Kügelrohr, 110 °C, 0.5 mm) affords the cycloadduct (6.0 g, 90%) as a colorless oil (Note 14) in >99% ee (Note 15).

2. Notes

1. The purity of the 2-adamantyl-4-methylphenol is important; in particular, the material should be free of 2,6-diadamantyl-4-methylphenol.

2. All reagents were obtained from commercial suppliers (Acros, Aldrich Chemical Company, Inc., or Strem Chemicals, Inc.). Toluene was distilled from sodium, and dichloromethane was distilled from calcium hydride. All other reagents were used as received without further purification.

3. The use of a syringe containing a teflon plunger prevents clogging during the addition of $SnCl_4$.

4. Caution must be taken to prevent the fluffy solid paraformaldehyde from dispersing outside of the flask during this addition process.

5. This procedure for the synthesis of 2-adamantyl-5-methylsalicylaldehyde is a modification of the method reported by Casiraghi.[2] The aldehyde can be recrystallized from hexanes, but purification is not essential for successful formation of the Schiff base. The purified aldehyde has the following spectral and physical properties: mp 151.5-152 °C; IR (KBr) 3200-2500, 1649, 1607, 1524, 1447, 1416, 1356, 1312, 1244, 1221, 1163, 1105, 1084, 1040, 963, 864 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.78 (s, 6H), 2.08 (s, 3H), 2.12 (s, 6H), 2.31 (s, 3H), 7.14 (d, J = 1.5 Hz, 1H), 7.26 (d, J = 1.5 Hz, 1H), 9.8 (s, 1H), 11.65 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 28.9, 36.9, 40.1, 120.3, 128.2, 131.2,

135.4, 138.1, 159.3, 197.1; Calcd for $C_{18}H_{22}O_2$: C, 79.96; H, 8.20. Found: C, 79.70; H, 8.16.

6. The aldehyde is observed to dissolve completely between 60-70 °C.

7. The product exhibits the following physical and spectroscopic properties: mp 219-221 °C; $[\alpha]^{26}_D$ +70.0 (c .100, THF); IR (KBr disk) 3584, 2905, 2849, 1624, 1597cm^{-1}; ^{1}H NMR (500 MHz, DMSO- d_6-) δ 1.69 (m, 6H), 1.99 (m, 3H), 2.05 (m, 6H), 2.23 (s, 3H), 2.95 (dd, *J* = 6.0, 15.5 Hz, 1H), 3.11 (dd, *J* = 6.1, 15.5 Hz, 1H), 4.54 ('q', *J* = 5.7 Hz, 1H), 4.73, (d, *J* = 5.5 hz, 1H), 5.23, (d, *J* = 4.9 hz, 1H), 7.01 (s, 1H), 7.09 (s,1H), 7.18-7.31 (m, 4H), 8.61 (s, 1H), 10.94 (s, 1H); ^{13}C-NMR (ppm): 20.2, 28.3, 36.2, 36.5, 39.0, 39.7, 73.9 (2 carbons), 118.2, 124.7, 125.0, 125.7, 126.6, 127.4, 127.9, 129.6, 130.0, 136.4, 141.0, 142.0, 158.5, 166.5; HRMS (*m/z*) (Cl NH_3) calcd for $C_{27}H_{35}NO_2(M)^+$ 401.2355, found 401.2341.

8. The water washes should be carried out with gentle shaking in order to avoid formation of intractable emulsions.

9. If partial concentration occurs during filtration, the filtrate should be diluted with acetone prior to addition of water such that the total volume is 20 mL. Upon addition of water, a small amount of precipitate may form. This should be redissolved by gently warming the solution or by addition of a minimal amount of acetone.

10. X-Ray quality crystals are obtained by recrystallization from acetone/water. The solid state structure of complex **1** is that of a dimer bearing a bridging water molecule and one terminal water molecule on each metal center.[3] This dimeric complex exhibits the following spectral properties: IR (KBr): 3414, 2903, 2847, 1618, 1537, 1433, 1340, 1305, 1228, 1168, 1078 cm^{-1}. LRMS (FAB): calcd for dimer $C_{54}H_{68}Cl_2N_2O_7Cr_2$, $(M\text{-}2Cl\text{-}2H_2O)^+$, 920, found 919. A dehydrated sample suitable for elemental analysis was prepared as follows: Chlorotrimethylsilane (39.0 μL, 0.31 mmol) was added to a solution of Cr(III)Cl complex (50.0 mg, 0.048 mmol) in dry *tert*-butyl methyl ether (2 mL). The mixture was stirred for 2 h under nitrogen to give a green precipitate. The mixture was concentrated *in vacuo*, suspended in dry *tert*-butyl methyl ether (2 mL), filtered and the residue washed with dry *tert*-butyl methyl ether. The residue was then dried under high vacuum (0.5 mm). Anal. Calcd for $[C_{27}H_{29}ClCrNO_2\text{+}2HCl]$: C, 57.92; H, 5.58; Cr, 9.29; N 2.50. Found: C, 57.49; H, 5.73; Cr, 9.00; N, 2.48.

11. For certain applications (see, for example, the first entry in Table 1), superior results in HDA reactions are obtained with catalyst **1b**, wherein the chloride counterion of **1a** is replaced with SbF_6. Preparation of catalyst **1b** is achieved as follows: A flame-dried, 50-mL, foil wrapped round-bottomed flask equipped with a stirbar was charged with complex **1a** (100 mg, 0.97 mmol, 1 equiv) and silver hexafluoroantimonate (66.8 mg, 0.19 mmol, 2 equiv). The flask was placed under a nitrogen atmosphere, *tert*-butyl methyl ether (30 mL) was added, and the mixture allowed to stir for 3 h. The reaction mixture was then filtered through Celite® and the isolated solids are washed with *tert*-butyl methyl ether (20 mL). The filtrates were combined and concentrated by rotary evaporation to afford the desired SbF_6 complex **1b** as a brown solid (165 mg). IR (KBr) 3378, 2973, 2905, 1615, 1538, 1229, 1069 cm^{-1}. LRMS (m/z) (FAB) mass calcd for $C_{27}H_{35}CrNO_2$, $(M)^+$ 451; found 451; calcd for $2[C_{27}H_{35}CrNO_2,]$, $(2M)^+$, 902; found 902; calcd for $2\ C_{27}H_{35}CrNO_2, + H_2O]$, $(2M + H_2O)^+$, 920; found 921.

12. The catalyst loading was calculated based on the number of equivalents of chromium relative to the limiting aldehyde substrate.

13. The molecular sieves (1.6 mm pellets) are powdered with a mortar and pestle and activated in a vacuum oven (130 °C) overnight before use. Alternatively, commercially available finely powdered 4Å molecular sieves (<5 micron) may be used.

14. The product has the following spectral and physical properties: $[\alpha]_D^{26}$ +55.3 (c 1.14, $CDCl_3$); R_f = 0.70 (1:1 ether/hexanes); IR (thin film) 2955, 2934, 2888, 2858, 1471, 1400, 1339, 1255, 1204, 1129, 1112, 1080, 1057 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.07 (s, 6H), 0.89 (s, 9H), 2.08 (m, 2H), 3.47 (s, 3H), 3.65 (dd, *J* = 6.5, 10.4 Hz, 1H), 3.76 (dd, *J* = 5.6, 10.4 Hz, 1H), 3.85 ('q', *J* = 6.3 Hz, 1H), 5.02 (m, 1H), 5.65 ('dq', *J* = 3.7, 10.2 Hz, 1H), 5.97 ('dq', *J* = 5.3, 10.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 5.2, 5.3, 18.4, 25.9, 26.8, 55.2, 65.5, 72.6, 97.7, 127.0 128.5; HRMS (*m/z*) (Cl) calc. for $C_{13}H_{30}NO_3Si$ $(M+NH_4)^+$ 276.1995, found 276.2003.

15. Enantiomeric excess was determined by GC analysis following conversion to (*R*)-6-(*tert*-butyldimethylsilyoxymethyl)-5,6-dihydropyran-2-one, according to the following procedure: Pyridinium dichromate (1.04 g, 2.75 mmol) was added to a solution of the acetal (256 mg, 1.38 mmmol) and acetic acid (3 mL) in CH_2Cl_2 (20 mL) at 23 °C. The mixture was stirred overnight, diluted with 1:1 ether/hexanes (20 mL), and filtered through a pad

of $MgSO_4$. The residue remaining in the reaction flask was washed thoroughly with 1:1 ether/hexanes (4 x 20 mL) and the extracts were filtered. The combined filtrates were filtered once more through a fresh pad of $MgSO_4$ and concentrated *in vacuo*. Kügelrohr distillation (210-220 °C, 10 mm) afforded the product lactone (267 mg, 57.0%). GC analysis using a commercial chiral column (Cyclodex β. 135 °C, isothermal) revealed the product to be in >99% ee (t_R(major) = 50.23 min). $[\alpha]_D^{26}$ +79 (c 1.00, $CDCl_3$). R_f = 0.17 (10% ether/hexanes). IR (thin film) 2955, 2930, 2859, 1732, 1471, 1407, 251, 1136, 1093, 1043 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 0.06 (s, 6H), 0.87 (s, 9H), 2.40 ('dt', *J* = 4.6, 18.6 Hz, 1H), 2.51 (ddd, *J* = 2.6, 11.1, 18.6 Hz, 1H), 3.78 (dd, *J* = 5.4, 10.9 Hz, 1H), 3.80 (dd, *J* = 4.64, 10.9 Hz, 1H), 4.45 (dddd, *J* = 4.4, 4.6, 5.4, 11.1 Hz, 1H), 5.99 (d, *J* = 9.7 Hz, 1H), 6.89 (ddd, *J* = 2.6, 5.8, 9.7 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ -5.4, 18.3, 25.8, 64.2, 77.8, 121.1, 145.0, 163.9. HRMS (*m/z*) (Cl) Calcd for $C_{12}H_{26}NO_3Si$ $(M+NH_4)^+$ 260.1682. Found 260.1679.

Waste Disposal Information

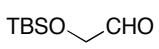
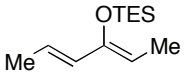
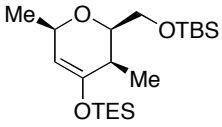
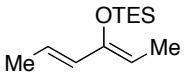
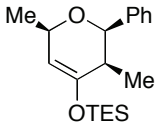
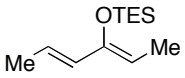
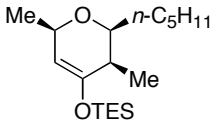
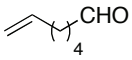
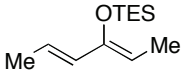
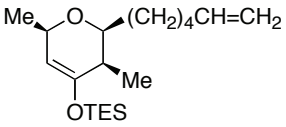
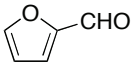
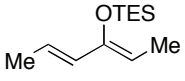
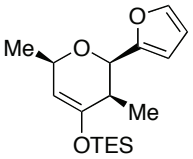
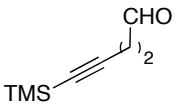
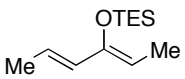
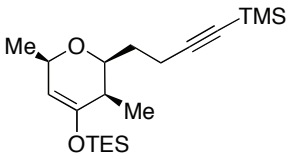
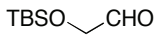
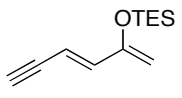
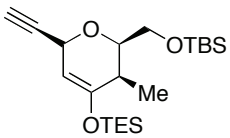
All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

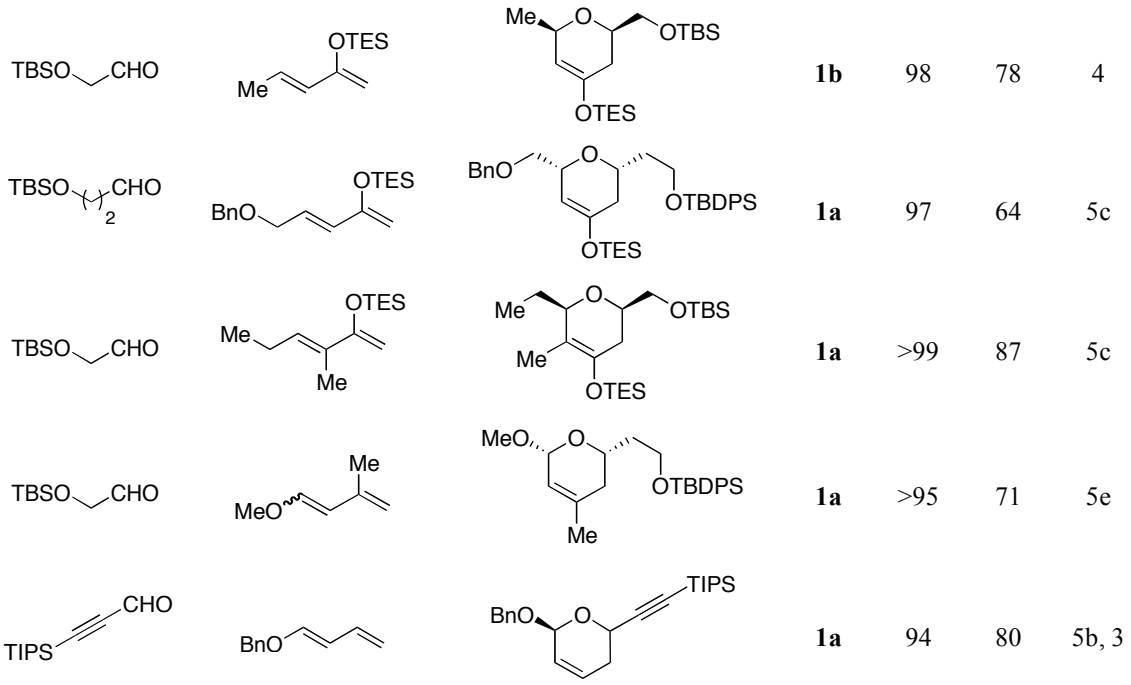
3. Discussion

This procedure describes a practical synthesis of the chiral tridentate Schiff base complex **1a**, and the use of this complex to catalyze an efficient, highly diastereo- and enantioselective hetero-Diels-Alder (HDA) reaction. The unique characteristic of this catalyst, and the derived SbF_6 complex **1b** (see Note 11), lies in their demonstrated ability to promote asymmetric hetero-Diels-Alder reactions between aldehydes and dienes bearing a single oxygen substituent.[4] Reactions proceed generally with excellent diastereo- and enantioselectivity, and provide access to enantiomerically enriched dihydropyran derivatives from simple achiral starting materials (Table 1). This HDA methodology has already been showcased in several natural product syntheses.[5] More recently, the same catalyst system has been applied to highly enantioselective inverse demand hetero-Diels-Alder reactions between conjugated aldehydes and ethyl vinyl ether.[3]

The method for the synthesis of complex **1a** described herein represents a significant improvement over the procedure first reported in 1999.[4] The use of air- and moisture-sensitive $CrCl_2$ is now avoided, and the necessity of conducting the metal-insertion step in a glove box is thereby precluded. Instead, the use of the $(Cr(III)Cl_3 \bullet [C_4H_8O]_3)$ complex allows the reaction to be conducted in a fume hood. Additionally, the procedure for the formylation of 2-adamantyl-4-methylphenol has been adapted such that purification of the resulting aldehyde by recrystallization is no longer necessary. Finally, and perhaps most important, catalysts prepared by the new procedure displays measurably higher enantioselectivity in a variety of HDA reactions.[3]

Table 1

Aldehyde	Diene	Product	Cat	ee (%)	Yield (%)	Ref
			**1a**	99	90	4
			1b	>99	97	4
			**1b**	90	72	4
			**1b**	98	85	4
			**1b**	98	85	4
			**1b**	95	77	4
			**1b**	95	92	5a
			**1b**	98	61	5a



The hetero-Diels-Alder reaction illustrated in this procedure utilizes commercially available 1-methoxy-1,3-butadiene and (*t*-butyldimethyl-silyloxy)acetaldehyde. The reaction is carried out with 1.5 mol% catalyst under solvent-free conditions. The dihydropyran is isolated in 90% yield, >97:3 dr, and >99% ee by direct distillation of the reaction mixture. The product can be oxidized to the corresponding lactone readily and in one step,providing efficient access to a substructure that occurs in several interesting natural products (i.e., fostriecin[5b], callystatin A[6a], ratjadone[6b]).

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Appendix
Chemical Abstracts Nomenclature (Registry Number)

2-(1-Adamantyl)-4-methylphenol: Phenol, 4-methyl-2-tricyclo[3.3.1.13,7]-dec-1-yl-; (41031-50-9)

(1*R*,2*S*)-1-Aminoindan-2-ol: 1H-Inden-2-ol, 1-amino-2,3-dihydro-,(1*S*-*cis*); (126456-43-7)

(1*R*,2*S*)-1-[(3-Adamantyl)-2-hydroxy-5-methylbenzylidenamino]indan-2-ol: 1H-Inden-2-ol, 2,3-dihydro-1-[[(2-hydroxy-5-methyl-3-tricyclo[3.3.1.13,7]dec-1-decylphenyl)methylene]amino]-, (1*R*,2*S*)-; (231963-92-1)

Chromium(III) Cl Complex: Chromium, chloro[(1*R*,2*S*)-2,3-dihydro-1-[[[2-(hydroxy-κO)-5-methyl-3-tricyclo[3.3.1.13,7dec-1-ylphenyl]methyl-ene]amino-κN]-1H-indene-2-olato-(2-)-κO],(SP-4-4); (231963-76-1)

1-Methoxy-1,3-butadiene: 1,3-Butadiene, 1-methoxy-; (3036-66-6)

(*tert*-Butyldimethylsilyloxy)acetaldehyde: Acetaldehyde, [[(1,1-dimethylethyldimethylsilyl]oxy]-; (102191-92-4)

(2*S*,6*R*)-6-(tert-Butyldimethylsilyloxymethyl)-2-methoxy-2,5-dihydropyran: Silane, [[(2*R*,6*S*)-3,6-dihydro-6-methoxy-2H-pyran-2-yl]methoxy](1,1-dimethylethyl)dimethyl-; (231963-89-6)

(1S,2R)-1-[3-Adamantyl)-2-hydroxy-5-methylbenzylidene-amino]-indan-2-ol

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PROCNO                1

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TD                 32768
SOLVENT             DMSO
NS                    32
DS                     2
SWH             6561.680 Hz
FIDRES          0.200247 Hz
AQ              2.4969716 sec
RG                   128
DW                76.200 usec
DE                 6.00 usec
TE                300.0 K
D1             0.10000000 sec

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PL1               -5.00 dB
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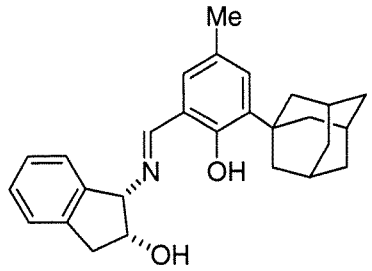

(2R,6S)-6-(*tert*-Butyldimethylsilyloxymethyl)-2-methoxy-2,5-dihydropyran

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AQ               2.4969716 sec
RG                     64
DW                 76.200 usec
DE                  6.00 usec
TE                 300.0 K
D1              0.10000000 sec

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PL1                -5.00 dB
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PC                   1.00
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7.05 5.99 5.98 5.98 5.97 5.97 5.96 5.96 5.95 5.95 5.67 5.66 5.66 5.65 5.64 5.64 5.63 5.03 5.03 5.02 5.02 5.02 5.01 3.87 3.86 3.84 3.83 3.78 3.77 3.75 3.74 3.68 3.66 3.65 3.64 3.47 2.10 2.09 2.09 2.08 2.08 2.08 2.07 2.07 2.07 2.06 0.94 0.93 0.93