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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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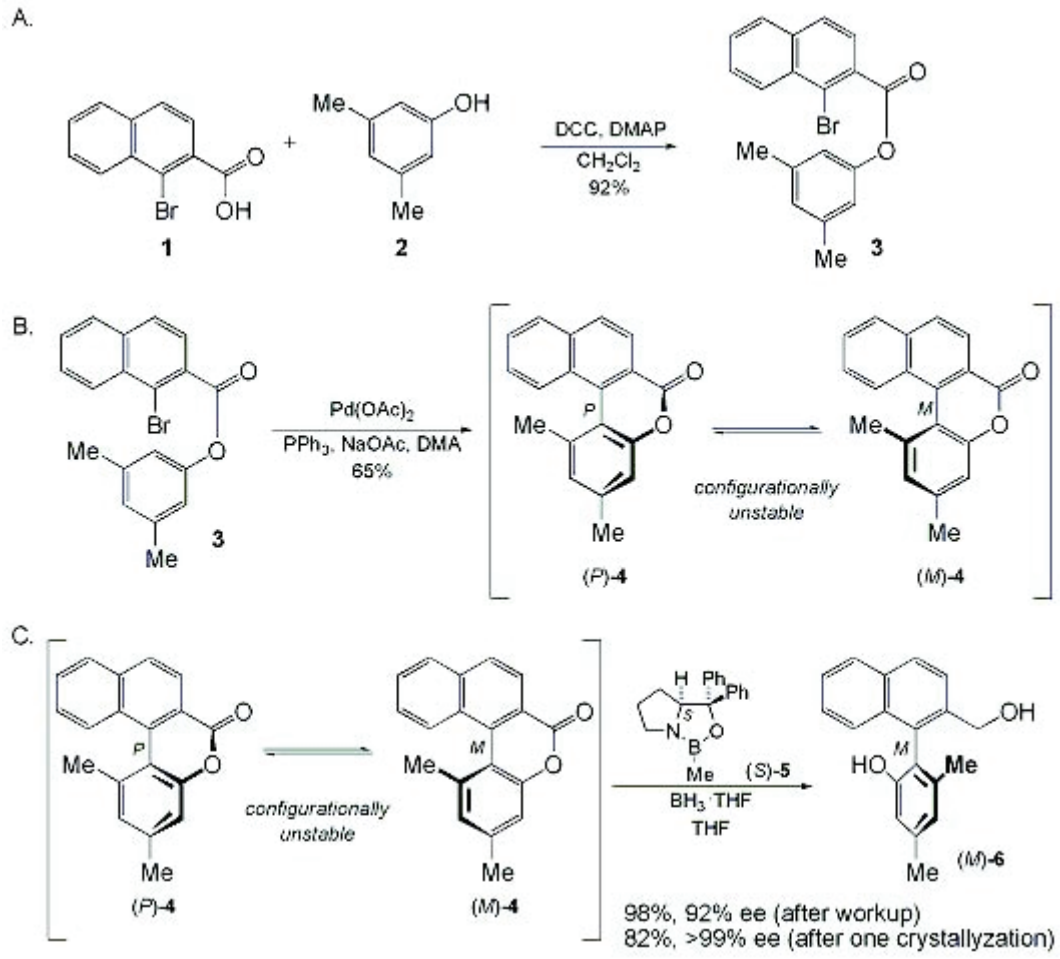
ASYMMETRIC SYNTHESIS OF (M)-2-HYDROXYMETHYL-1-(2-HYDROXY-4,6-DIMETHYLPHENYL)NAPHTHALENE VIA A CONFIGURATIONALLY UNSTABLE BIARYL LACTONE

[2-Naphthalenemethanol, 1-(2-hydroxy-4,6-dimethylphenyl)-, (R)-]



Submitted by Gerhard Bringmann[1], Matthias Breuning, Petra Henschel, and Jürgen Hinrichs. Checked by Peter M. Greenen and Dennis P. Curran.
Discussion Addendum *Org. Synth.* **2011**, *88*, 70.

1. Procedure

*A. 3,5-Dimethylphenyl 1-bromo-2-naphthoate (**3**)* . Under a nitrogen atmosphere, a 250-mL, oven-dried, round-bottomed flask containing anhydrous dichloromethane (100 mL) is charged with 1-bromo2-naphthoic acid (**1**, 2.51 g, 10.0 mmol), 3,5-dimethylphenol (**2**, 1.23 g, 10.1 mmol), dicyclohexylcarbodiimide (DCC, 2.26 g, 11.0 mmol), and 4-(dimethylamino)pyridine (DMAP, 244 mg, 2.00 mmol) (Note 1). After the mixture is stirred for 12 hr at room temperature, the white precipitate that forms (Note 2) is discarded by filtration through a Buchner funnel. From the clear filtrate, the solvent is removed by rotary evaporation (35°C, 720 mbar, 540 mm) to give a colorless solid. Filtration through a short silica gel column (5 × 40-cm column, silica gel 0.063 - 0.2 mm, 150 g ; eluent: hexane / diethyl ether 5:1) delivers 3.35 g (94%) of the ester **3**, which is recrystallized from diethyl ether / hexane to give 3.28 g (92%) of a colorless solid (Note 3).

*B. 1,3-Dimethyl-6H-benzo[b]naphtho[1,2-d]pyran-6-one (**4**)* . Under an argon atmosphere, a 250-

mL, oven-dried, round-bottomed flask, equipped with a reflux condenser, is charged with freshly distilled N,N-dimethylacetamide (DMA, 130 mL), 3,5-dimethylphenyl 1-bromo-2-naphthoate (**3**, 3.24 g, 9.12 mmol), palladium(II) acetate (205 mg, 0.913 mmol), triphenylphosphine (481 mg, 1.83 mmol), and sodium acetate (1.50 g, 18.3 mmol) (Notes 4 and 5). The orange suspension is degassed three times, placed in a preheated (130°C) oil bath (Note 5), and stirred at 130°C for 12 hr (Note 6). Removal of the solvent at 40°C (0.1 mbar, 0.075 mm) gives a black oily residue, which is chromatographed (5 × 40 cm column, silica gel 0.063 - 0.2 mm, 170 g , 1 cm of charcoal at the top of the column; eluent: hexane / diethyl ether 5:1), to yield 2.00 g (80%) of the lactone **4** as a slightly yellow solid. Recrystallization from diethyl ether / hexane delivers 1.63 g (65%) of colorless or pale yellow crystals (Note 7).

*C. (M)-2-Hydroxymethyl-1-(2-hydroxy-4,6-dimethylphenyl)naphthalene [(M)- **6**]* (Note 8). Under an argon atmosphere, an oven-dried Schlenk tube is charged with the CBS-catalyst (S)-**5** (1.0 M in toluene, 8.39 mL, 8.39 mmol) (Note 9). The solvent is removed under high vacuum (0.1 mbar. 0.075 mm) at room temperature and tetrahydrofuran (THF, 110 mL) (Note 9) is added. After the solution is cooled to 0°C, it is treated with the borane-THF complex (1.0 M in THF, 10.1 mL, 10.1 mmol) (Note 9) and stirred at room temperature for 30 min. This reagent and a solution of the lactone **4** (1.84 g, 6.71 mmol) in THF (110 mL) are added simultaneously from two dropping funnels into an oven-dried, round-bottomed, three-necked flask containing THF (110 mL) at a temperature of 30°C over a period of 2 hr (Note 10). After the reaction mixture is stirred for another 30 min, it is adjusted to pH 4 by careful addition of hydrochloric acid (2.0 M). Water (20 mL) is added, the organic solvent is removed by rotary evaporation (40°C / 350 mbar, 263 mm), and the remaining aqueous phase is extracted with diethyl ether (4 × 100 mL) (Note 11). The combined organic layers are dried over magnesium sulfate ($MgSO_4$). Removal of the solvent by rotary evaporation and filtration through a short silica gel column (5 × 40-cm column, silica gel 0.063 - 0.2 mm, 100 g ; eluent: hexane / diethyl ether 1:1) gives 1.83 g (98%) of the biaryl alcohol (M)-**6** as a slightly yellow solid with 92% ee (Notes 12 and 13). Crystallization from diethyl ether / hexane delivers 1.53 g (82%) of colorless crystals with >99% ee (Notes 14 and 15).

2. Notes

1. 1-Bromo-2-naphthoic acid (**1**, 98%, Sigma Chemical Co.), 3,5-dimethylphenol (**2**, >98%, Merck-Schuchard), 1,3-dicyclohexylcarbodiimide (DCC, 99%, Merck-Schuchard), and 4-(dimethylamino) pyridine (DMAP, 99%, Aldrich Chemical Co., Inc.) were used as received. Dichloromethane was distilled from phosphorus pentoxide and stored over activated molecular sieves (4Å).
2. The precipitate consists of 1,3-dicyclohexylurea .
3. The physical properties of **3** are as follows: mp 85°C; IR (KBr) $cm^{-1:}$ 1594, 1618, 1744, 2916, 3060 ; ^1H NMR (250 MHz, $CDCl_3$) δ: 2.38 (s, 6 H), 6.95 (s, 3 H), 7.67 (m_c, 2 H), 7.88 (m_c, 3 H), 8.50 (m_c, 1 H) ; ^{13}C NMR (63 MHz, $CDCl_3$) δ: 21.30, 119.1, 123.3, 125.9, 127.9, 128.0, 128.0, 128.2, 128.4, 128.7, 130.7, 132.6, 135.4, 139.5, 150.7, 166.0 ; HRMS: m/z 354.0257, calcd. for $C_{19}H_{15}BrO_2$: 354.0255 .
4. Palladium(II) acetate (98%, Strem Chemicals Inc.), triphenylphosphine (99%, Fisher Scientific Co.), and sodium acetate (99%, Fluka Chemika) were used without further purification. N,N-Dimethylacetamide (DMA) was distilled from calcium hydride through a 25-cm Vigreux column directly before use.
5. Freshly distilled DMA, a preheated oil bath, and the repeated degassing of the reaction mixture are critical to obtain high yields.
6. The reaction progress can easily be followed by TLC (silica, hexane / diethyl ether 5:1) due to the brilliant blue fluorescence of **4** (R_f = 0.30) upon UV excitation at 366 nm.
7. The physical properties of **4** are as follows: mp 158°C; IR (KBr) cm^{-1}: 1594, 1614, 1721, 2928, 2985, 3055 ; ^1H NMR (250 MHz, $CDCl_3$) δ: 2.25 (s, 3 H), 2.46 (s, 3 H), 7.08 (s, 1 H), 7.15 (s, 1 H), 7.56 (m, 1 H), 7.66 (m, 1 H), 7.96 (d, 3 H, J = 8.5 Hz), 8.27 (d, 1 H, J = 8.5 Hz) ; ^{13}C NMR (63 MHz, $CDCl_3$) δ: 21.27, 23.77, 114.7, 116.0, 121.2, 124.0, 125.9, 128.1, 128.3, 128.6, 128.8, 128.9, 135.6, 136.3, 136.3, 140.1, 140.2, 151.9, 161.9 ; HRMS: m/z 274.0987, calcd. for $C_{19}H_{14}O_2$: 274.0994 .
8. For the now recommended M/P denotion for axial chirality, see Helmchen[2].
9. The CBS-catalyst [(S)-2-methyl-CBS-oxazaborolidine] (S)-**5** (1.0 M in toluene) (The CBS catalyst is named after Corey, Bakshi, and Shibata) and the borane-THF complex (1.0 M in THF) were obtained from Aldrich Chemical Co., Inc. and used as received. THF was distilled from potassium directly before use.
10. For optimum stereoselectivity, it is critical to control the temperature of the reaction vessel to

exactly 30°C.
11. For a recovery of (S)-α,α-diphenylprolinol , which is the hydrolysis product of the CBS-catalyst (S)-**5** (and likewise its synthetic precursor[3]), the aqueous phase is carefully adjusted to pH 10 with concentrated ammonia and extracted with diethyl ether (3 × 50 mL). The combined organic layers are washed with brine (50 mL) and dried over $MgSO_4$. Removal of the solvent by rotary evaporation yields 1.68 g (79%) of crude (S)-α,α-diphenylprolinol . This material is dissolved in dichloromethane / methanol 9:1 (3 mL) and filtered over Alox B (act. III, 80 g) with dichloromethane / methanol 9:1 as the eluent, to yield 1.64 g (77%) of (S)-α,α-diphenylprolinol as a white solid.
12. The ee of **6** was determined by HPLC on a chiral phase [DAICEL Chiralcel OD-H (4.6 mm × 250 mm), detection at 280 nm, flow rate 1.0 mL/min, eluent: hexane / isopropyl alcohol 95:5, retention times: t_R = 16 min for (M)-**6** and t_R = 22 min for (P)-**6**].
13. The analogous reduction of **4** with a catalytic amount of the CBS-catalyst (S)-**5** (0.1 equiv) and 1.25 equiv of borane resulted in the formation of (M)-**6** in a slightly lower ee of 88% (94% yield). In several cases, over-stoichiometric amounts of (S)-**5** and BH_3 · THF had to be used to ensure complete conversion.[4 5 6] Treatment of **4** with 3 equiv of (S)-**5** and 4 equiv of the borane complex gave 97% of (M)-**6** with 90% ee.
14. The almost racemic alcohol **6** obtained from the concentrated mother liquor can be recycled by a three-step procedure (1. MnO_2, CH_2Cl_2; 2. $NaClO_2$, H_2NSO_3H, NaOAc, dioxane / acetic acid / water; 3. N-methyl-2-chloropyridinium iodide , $(n\text{-}Bu)_3N$, CH_2Cl_2) to give, in a 49% overall yield, the lactone **4**, which can then be ring-opened atropoenantioselectively once again. Alternatively, the recycling can be done by just oxidizing to the corresponding hydroxy aldehyde, followed by its atropo-enantioselectively reduction.[4, 7]
15. The physical properties of **6** are as follows: mp 140°C; $[\alpha]_D^{23}$ −41.4° ($CHCl_3$, *c* 1.02); IR (KBr) cm^{-1}: 1572, 1620, 2923, 2986, 3055, 3376 ; ^1H NMR (200 MHz, $CDCl_3$) δ: 1.80 (s, 3 H), 2.38 (s, 3 H), 4.52 (m_c, 2 H), 6.73 (s, 1 H), 6.79 (s, 1 H), 7.32-7.55 (m, 3 H), 7.70 (d, 1 H, J = 8.5), 7.92 (m_c, 2 H) ; ^{13}C NMR (50 MHz, $CDCl_3$) δ: 19.73, 21.28, 63.70, 114.0, 121.0, 123.4, 125.4, 126.2, 126.5, 126.8, 128.2, 128.9, 131.2, 132.5, 133.5, 137.4, 138.0, 139.2, 153.1 ; HRMS: m/z 278.1304, calcd. for $C_{19}H_{18}O_2$: 278.1307 .

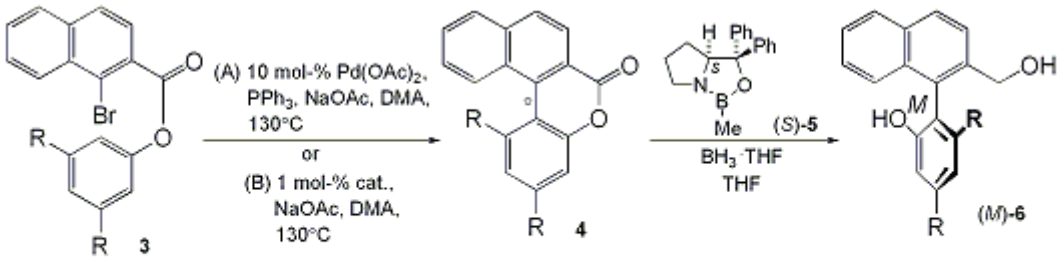
Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

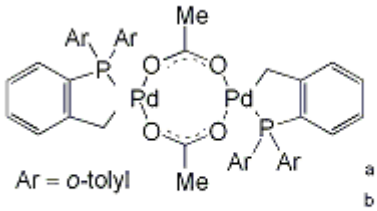
The reaction sequence described here provides a simple and efficient route to the enantiomerically pure axially chiral biaryl alcohol (M)-**6** [8] and illustrates the basic strategy of the 'lactone concept',[9] in which the two crucial steps, the aryl-aryl bond formation and asymmetric induction at the newly created axis, are performed *consecutively*. This stepwise procedure is quite generally applicable and offers several advantages over other known methods[10] of stereoselective biaryl coupling: The prefixation of the aryl moieties as esters of type **3** (Scheme 1), which is easily attainable by standard procedures, allows an intramolecular cross coupling. This coupling reaction proceeds regioselectively and in high yields, even against severe steric hindrance (e.g., with a tert-butyl group ortho to the axis).[11 12] As the catalyst, $Pd(OAc)_2$ or the more effective, now likewise commercially available Herrmann-Beller palladacycle **7** can be employed.[11,12] The resulting biaryl lactones **4** are helically distorted and thus chiral,[11] but because of the bridging lactone function, which dramatically lowers the atropoisomerization barrier, they are still configurationally unstable at the axis (An exception is the sterically highly hindered lactone **4** (R = tBu), which is configurationally stable at room temperature.), and thus exist as a racemic mixture of their rapidly interconverting enantiomers (M)-**4** and (P)-**4**.[11, 13] This is the fundamental prerequisite for the subsequent formation of configurationally stable and stereochemically pure biaryl molecules **6** through oxazaborolidine-mediated dynamic kinetic resolution[8,12] (see Scheme 1).

Scheme 1



° = configurationally unstable (except for R = *t*Bu)

cat.:



7

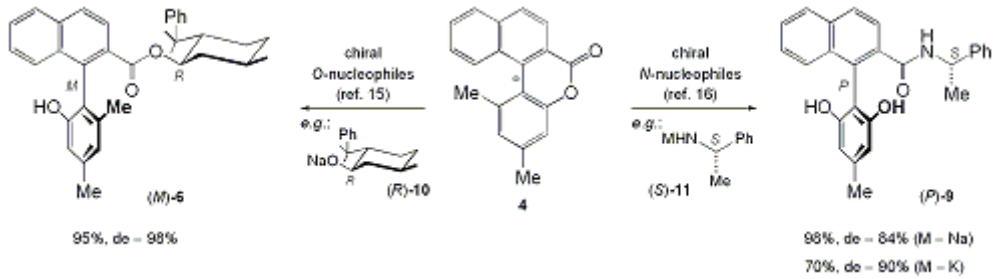
		coupling[8,10]	ring cleavage[7,11]	
entry	R	yield [%]	yield [%]	ee [%]
1	H	70 (91)[a]	94	82
2	OMe	77 (90)	95	94[b]
3	Me	74 (90)	98	92
4	*t*Bu	31 (81)	[c]	99[c]

[a] method (B) in parentheses

[b] note that for formal reasons of the CIP denotation, stereochemically analogous biaryls of this series with R = OMe will have descriptors opposite to those with R = H or alkyl

In the stereochemically deciding key step, cleavage of the lactone bridge can also be performed atropo-diastereo- or -enantioselectively with a wide range of chiral O-,[14] N-,[15] or other H-[16] nucleophiles to give the ring-opened, and now configurationally stable, axially chiral biaryls in high optical and chemical yields. In each case, the stereochemically pure biaryls can be obtained by crystallization or, if diastereomers are formed, by chromatographic separation. Two examples are illustrated in Scheme 2 with **4** (R = Me) as the biaryl lactone.

Scheme 2



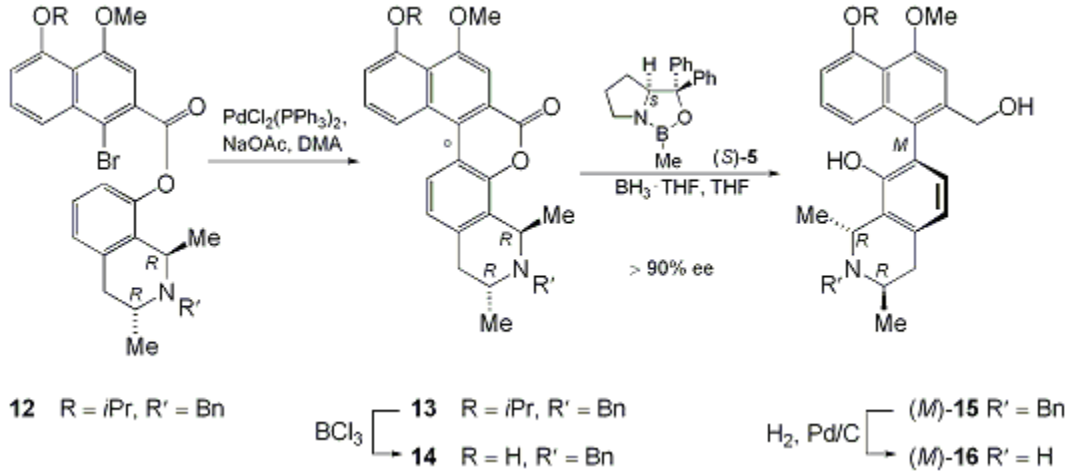
Since, for all the chiral ring cleavage reagents [(S)-**5**, (R)-**10**, and (S)-**11**] used, both enantiomers are commercially available, both atropoisomeric biaryls are readily accessible from the same lactone precursor **4**, which allows flexible atropo-divergent[9] syntheses.[14,15] Furthermore, for precious material prepared in the course of multi-step synthesis, even the minor, undesired atropoisomer, if formed at all in significant quantities, is not lost, but can be recycled, either by acid catalyzed- (for the ester **8** or the amide **9**) or oxidative (for the alcohol **6**) cyclization back to the biaryl lactone **4**, and renewed atroposelective cleavage.[14,15] Differing from most of the existing methods, the decision as to which atropoisomer is to be prepared can be taken at a very late stage of the synthesis.

The lactone concept is not restricted to the simple model biaryl synthesis presented here. It has been successfully expanded to a broad series of structurally diverse biaryl substrates (e.g., lactones with additional stereocenters and functional groups,[9] configurationally stable lactones,[12] seven-membered lactones,[17] and again configurationally unstable biaryl hydroxy aldehydes[7]), to different activation modes in the ring-opening step (e.g., use of metallated nucleophiles, carbonyl activation by Lewis acids,

(η[6]-complexation, etc.),[18] and for various strategies of stereoselection (e.g., external vs. internal asymmetric induction).[19]

The broad applicability of the strategy has been proven in the atroposelective synthesis of a broad series of structurally different bioactive natural biaryl products like the naphthylisoquinoline alkaloid dioncopeltine A (**16**)[4] (Scheme 3), the dimeric sesquiterpene mastigophorene A,[6] the phenyl anthraquinone knipholone,[20] or even molecules without O- or (free) C_1-units next to the axis, i.e., dioncophylline C[19] and korupensamine A.[5] Furthermore, biaryl derivatives resulting from the ring opening of the model lactone **4** were used successfully as chiral catalysts in asymmetric synthesis, like amino alcohols for the enantioselective addition of Et_2Zn to aldehydes[21] or a phosphine for the enantioselective hydrosilylation of styrenes.[22]

Scheme 3



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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

(M)-2-Hydroxymethyl-1-(2-hydroxy-4,6-dimethylphenyl)naphthalene:
2-Naphthalenemethanol, 1-(2-hydroxy-4,6-dimethylphenyl)-, (R)- (13); (140834-52-2)

3,5-Dimethylphenyl-1-bromo-2-naphthoate:
2-Naphthalenecarboxylic acid, 1-bromo-, 3,5-dimethylphenyl ester (13); (138435-66-2)

1-Bromo-2-naphthoic acid:
2-Naphthoic acid, 1-bromo- (9); (20717-79-7)

3,5-Dimethylphenol:
Phenol, 3,5-dimethyl- (9); (108-68-9)

Dicyclohexylcarbodiimide: HIGHLY TOXIC:
Carbodiimide, dicyclohexyl- (8);
Cyclohexanamine, N,N'-methanetetraylbis- (9); (538-75-0)

4-Dimethylaminopyridine: HIGHLY TOXIC:
Pyridine, 4-(dimethylamino)- (8);
4-Pyridinamine, N,N-dimethyl- (9); (1122-58-3)

1,3-Dimethyl-6H-benzo[b]naphtho[1,2-d]pyran-6-one:
6H-Benzo[b]naphtho[1,2-d]pyran-6-one, 1,3-dimethyl- (13); (138435-72-0)

N,N-Dimethylacetamide:

Acetamide, N,N-dimethyl- (8,9); (127-19-5)

Palladium acetate:
Acetic acid, palladium(2+) salt (8,9); (3375-31-3)

Triphenylphosphine:
Phosphine, triphenyl- (8,9); (603-35-0)

(S)-2-Methyl-CBS-oxazaborolidine: (CBS named after Corey, Bakshi, Shibata):
1H, 3H-Pyrrolo[1,2-c][1,3,2]oxazaborole, tetrahydro-1-methyl-3,3-diphenyl-, (S)- (12); (112022-81-8)

Borane-tetrahydrofuran complex:
Furan, tetrahydro-, compd. with
borane (1:1) (8,9); (14044-65-6)