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for the Preparation
of Organic Compounds

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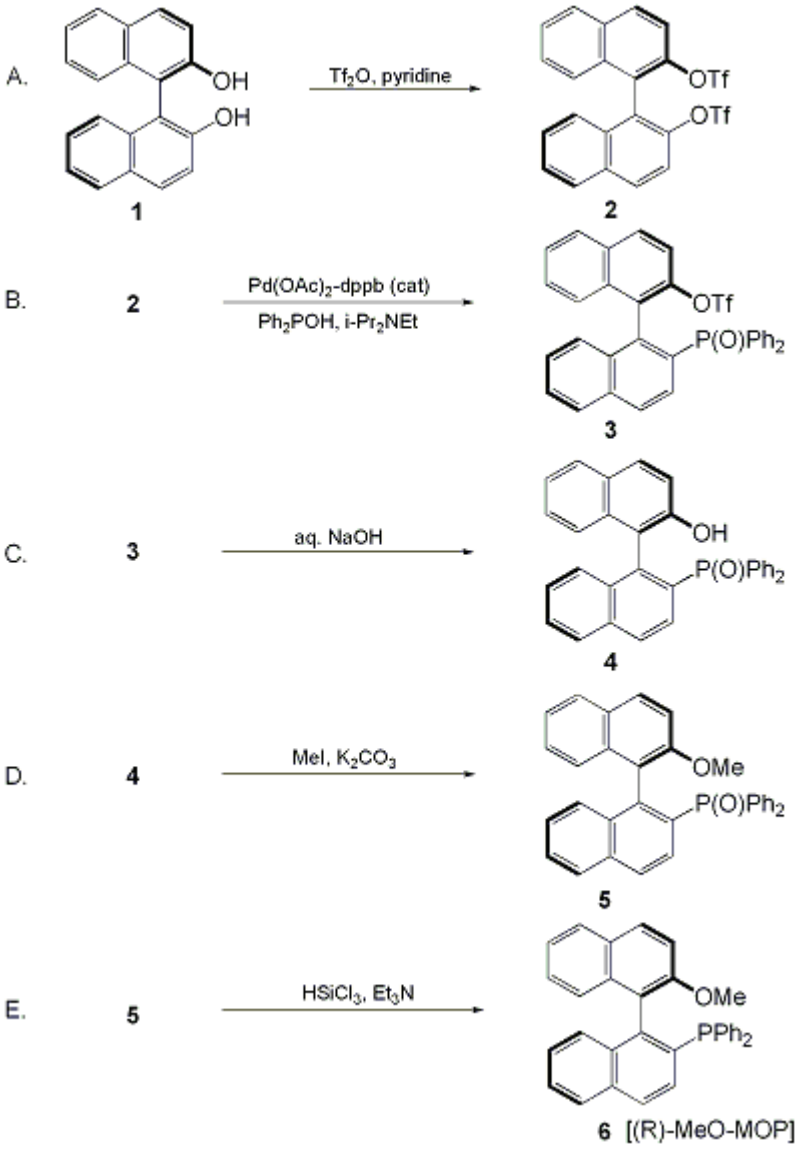
September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

DOI:10.15227/orgsyn.078.0001

Organic Syntheses, Coll. Vol. 10, p.363 (2004); Vol. 78, p.1 (2002).

(R)-2-DIPHENYLPHOSPHINO-2'-METHOXY-1,1'-BINAPHTHYL

[Phosphine, (2'-methoxy[1,1'-binaphthalen]-2-yl)diphenyl-, (R)-]



Submitted by Yasuhiro Uozumi, Motoi Kawatsura, and Tamio Hayashi[1] .
Checked by Sarge Salman and Louis S. Hegedus.

1. Procedure

Caution! All reactions should be conducted in a well-ventilated hood.

A. (R)-2,2'-Bis(trifluoromethanesulfonyloxy)-1,1'-binaphthyl (**2**). A dry, 200-mL, two-necked, round-bottomed flask is fitted with a magnetic stirring bar and a 30-mL pressure-equalizing addition funnel, and flushed with nitrogen gas. The flask is charged with 14.3 g (50.0 mmol) of (R)-(+)-1,1'-bi-2-naphthol (**1**) (Note 1), 12.0 mL (148 mmol) of pyridine (Note 2), and 100 mL of dichloromethane (Note 3), and the entire mixture is cooled to 0°C with an ice-water bath. Trifluoromethanesulfonic anhydride, (20.0 mL, 33.5 g, 119 mmol) (Note 2) is added dropwise over a period of 10 min to the stirred solution.

After the mixture is stirred at 0°C for 6 hr, the reaction mixture is concentrated on a rotary evaporator. The residual brown oil is diluted with 200 mL of ethyl acetate and transferred to a 500-mL separatory funnel. The organic phase is washed with 5% hydrochloric acid (70 mL), saturated sodium bicarbonate (70 mL), and saturated sodium chloride (70 mL). The organic phase is dried over anhydrous sodium sulfate , and concentrated under reduced pressure on a rotary evaporator. The residue is chromatographed (10 × 20 cm column) on silica gel (700 g) (Note 4). The column is eluted with dichloromethane and the fractions are analyzed by TLC on silica gel (Note 5) using 30% dichloromethane-hexane as eluant. Fractions containing the product are combined and the solvent is evaporated on a rotary evaporator to give 26.3 g (96%) of **2** as a white powder (Note 6).

B. (R)-(+)-2-Diphenylphosphinyl-2'-trifluoromethanesulfonyloxy-1,1'-binaphthyl (**3**). A dry, 500-mL, Schlenk tube is fitted with a magnetic stirring bar and a rubber septum, and flushed with nitrogen gas. The flask is charged with 25.0 g (45.4 mmol) of (R)-2,2'-bis(trifluoromethanesulfonyloxy)-1,1'-binaphthyl (**2**), 18.4 g (91.0 mmol) of diphenylphosphine oxide (Note 7), 1.02 g (4.54 mmol) of palladium acetate (Note 8), 1.94 g (4.55 mmol) of 1,4-bis(diphenylphosphino)butane (dppb) (Note 9), 23.4 g of diisopropylethylamine (181 mmol) (Note 10) and 200 mL of dimethyl sulfoxide (Note 11), and the entire mixture is heated with stirring at 100°C for 12 hr (Note 12). After the reaction mixture is cooled to room temperature, it is concentrated under reduced pressure (0.1-0.2 mm) on a rotary evaporator. The dark brown residue is diluted with 400 mL of ethyl acetate and transferred to a 1-L separatory funnel. The organic phase is washed successively with water (two 100-mL portions), 5% hydrochloric acid (100 mL), saturated sodium bicarbonate (100 mL), and saturated sodium chloride (100 mL), and the organic phase is dried over anhydrous sodium sulfate . After filtration the organic phase is concentrated under reduced pressure on a rotary evaporator, and the residue is chromatographed (14 × 30-cm column) on silica gel (ca. 1.5 kg) (Note 4). The column is eluted with 50% ethyl acetate-hexane and the fractions are analyzed by TLC on silica gel (Note 5) using the same eluant. Fractions containing the product are combined and the solvent is evaporated on a rotary evaporator to give 23.8 g (87%) of **3** as a white powder (Notes 13, 14).

C. (R)-(−)-2-Diphenylphosphinyl-2'-hydroxy-1,1'-binaphthyl (**4**) . A 100-mL round-bottomed flask containing a magnetic stirring bar is charged with 6.07 g (10.1 mmol) of (R)-(+)-2-diphenylphosphinyl-2'-trifluoromethanesulfonyloxy-1,1'-binaphthyl (**3**), 30 mL of 1,4-dioxane , and 14 mL of methanol . (Note 15) To the solution is added 14.1 mL of 3 N aqueous sodium hydroxide (NaOH) solution at ambient temperature. The reaction mixture is stirred for 12 hr then acidified (to pH 1) by the addition of a few drops of concd hydrochloric acid. The mixture is transferred to a separatory funnel and extracted twice with ethyl acetate (EtOAc). The organic phase is dried over magnesium sulfate ($MgSO_4$), filtered, and concentrated under reduced pressure to afford 6.19 g of **4** as a solid. (Note 16). This crude material is carried on to the next step without purification, assuming 100% yield.

D. (R)-(+)-2-Diphenylphosphinyl-2'-methoxy-1,1'-binaphthyl (**5**). A 250-mL round-bottomed flask is charged with crude **4**, 5.55 g (40.2 mmol) of potassium carbonate(K_2CO_3), and 66 mL of acetone . To this mixture is added 2.5 mL (40.2 mmol) of methyl iodide (MeI) (Note 17). The reaction mixture is refluxed for 3 hr. After the reaction is cooled to room temperature it is filtered through a Celite pad (Note 18), and the filter cake is washed with diethyl ether (Et_2O). The filtrate is concentrated under reduced pressure to give 6.88 g of **5** as a brown powder (Note 19). This crude material is carried on to the next step without purification, assuming 100% yield.

E. (R)-(+)-2-Diphenylphosphino-2'-methoxy-1,1'-binaphthyl (**6**). A 250-mL round-bottomed flask containing a magnetic stirring bar is charged with crude **5**, 7 mL (50 mmol) of triethylamine (Et_3N) (Note 20), and 84 mL of toluene . (Note 21). The mixture is cooled to 0°C then 4 mL (40 mmol) of trichlorosilane (Cl_3SiH) (Note 22) is added via syringe. The reaction is heated to 120°C and stirred for 5 hr. Upon cooling to ambient temperature the reaction is diluted with Et_2O and quenched with aqueous saturated sodium bicarbonate. The resulting suspension is filtered through a Celite pad, and the filter cake is washed with Et_2O. The organic phase is dried over $MgSO_4$ then concentrated under reduced pressure to afford 4.94 g of a yellow solid. This crude material is dissolved in a minimal amount of dichloromethane (CH_2Cl_2) and chromatographed on a 10 × 30-cm column containing 615 g of silica gel (SiO_2). The column is eluted with Et_2O to afford 4.08 g (8.71 mmol, 86% yield over last 3 steps) of **6** as an off-white powder (Note 23).

2. Notes

1. (R)-(+)-1,1'-Bi-2-naphthol (>99% op) was purchased from Aldrich Chemical Company, Inc. , and used without further purification.
2. Pyridine and trifluoromethanesulfonic anhydride were purchased from Aldrich Chemical Company, Inc. , and used without further purification.
3. Dichloromethane was purchased from Fisher Scientific, and distilled from calcium hydride before use.
4. Silica gel 60 230-400 mesh ASTM was used.
5. Merck silica gel 60F-254 plates were used.
6. Specific rotation value of **2**: $[\alpha]_D^{22}$ −143.5° ($CHCl_3$, *c* 1.24) [literature rotation for (S)-**2**;[2] $[\alpha]_D^{22}$ +142° ($CHCl_3$, *c* 1.035)]; ^{1}H NMR δ: 7.27 (d, 2 H, J = 8.7), 7.41 (t, 2 H, J = 7.8), 7.58 (t, 2 H, J = 7.8), 7.62 (d, 2 H, J = 9.6), 8.00 (d, 2 H, J = 8.3), 8.13 (d, 2 H, J = 9.2). If the material does not solidify, addition of equal amounts of diethyl ether and hexane, followed by reevaporation, should produce solid material.
7. Diphenylphosphine oxide is commercially available from Aldrich Chemical Company, Inc. , and was recrystallized from 1:1 hexanes/ethyl acetate prior to use. The submitters have prepared and used this reagent. Preparation of diphenylphosphine oxide : To a solution of diethyl phosphite (65.0 mL, 505 mmol) in 250 mL of tetrahydrofuran (THF) is added sodium metal (11.5 g, 500 mg-atom) and the mixture is stirred under reflux for 20 hr. The resulting solution is added to 1.9 M solution (THF/Et_2O = 1/2) of phenylmagnesium bromide (580 mL, 1.10 mol) at 0°C and the mixture is refluxed for 6 hr. After the mixture is quenched with a small amount of water, it is diluted with ethyl acetate and washed once with 5% hydrochloric acid (HCl) and twice with water. The organic phase is dried over anhydrous sodium sulfate and concentrated under reduced pressure to give crude diphenylphosphine oxide . The crude solid is purified by silica gel column chromatography (eluant: ethyl acetate) to give diphenylphosphine oxide (77.0 g, 75%).
8. Palladium acetate was purchased from Aldrich Chemical Company, Inc. , and purified as follows: Palladium acetate is dissolved in hot benzene and filtered from insoluble material. After removal of the solvent, the residue is triturated with a small amount of diethyl ether to give brown powder that is collected by filtration, washed with diethyl ether and dried.
9. 1,4-Bis(diphenylphosphino)butane was purchased from Aldrich Chemical Company, Inc. , and used without further purification.
10. Diisopropylethylamine was purchased from Aldrich Chemical Company, Inc. , and used without further purification.
11. Dimethyl sulfoxide was purchased from Aldrich Chemical Company, Inc. , and distilled from calcium hydride before use.
12. The use of 5 mol% of the catalyst [$Pd(OAc)_2$-dppb] also gave a high yield of **3**, but the submitters recommend the use of 10 mol % of the catalyst to ensure high chemical yield in the 12-hr reaction.
13. The physical properties of **3** are as follows: $[\alpha]_D^{20}$ +44.4° ($CHCl_3$, *c* 1.20), $[\alpha]_D^{22}$ +7.4° (CH_2Cl_2, *c* 1.40) [literature rotation for (R)-**3**;[3] $[\alpha]_D$ +6.29° (CH_2Cl_2, *c* 1.00)]; IR (KBr) cm^{-1} ν: 1410, 1205, 1140, and 945 ; ^{1}H NMR δ: 6.9-8.1 (m, 22 H, aromatic) ; ^{31}P NMR δ: 28.9 (s) ; EIMS m/z 603 (M+1), 454, 201 (base peak) . Anal. Calcd for $C_{33}H_{22}F_3O_4PS$: C, 65.78; H, 3.68. Found: C, 65.67; H, 3.89.
14. Assignment of all peaks in the ^{13}C NMR is difficult because of ^{13}C-^{31}P coupling and the overlapping of peaks.
15. Methanol and 1,4-dioxane were purchased from Aldrich Chemical Company, Inc. , and used without further purification.
16. An analytically pure sample is isolated by column chromatography on silica gel (see Note 4). The column is eluted with 50% ethyl acetate-hexane and the fractions are analyzed by TLC on silica gel (see Note 5) using the same eluent. Fractions containing the product are combined and the solvent is evaporated on a rotary evaporator to give **4** as a white powder. The physical properties of **4** are as follows (see Note 14): $[\alpha]_D^{20}$ −105° ($CHCl_3$, *c* 0.55), $[\alpha]_D^{20}$ −113° (CH_2Cl_2, *c* 1.00) [literature rotation for (R)-**4**;[3] $[\alpha]_D$ −108.3° (CH_2Cl_2, *c* 1.00)]; ^{1}H NMR δ: 6.35-8.10 (m, 22 H), 9.01 (br s, 1 H) ; ^{31}P NMR δ: 31.42 (s) ; EIMS m/z 470 (M^+), 268 (base peak) ; HRMS calcd for $C_{32}H_{23}PO_2$ 470.1436, found 470. 1415. Anal. Calcd for $C_{32}H_{23}O_2P$: C, 81.69; H, 4.93. Found: C, 81.66; H, 4.96.
17. Methyl iodide, potassium carbonate, and acetone were purchased from Aldrich Chemical Company, Inc. , and used without further purification.

18. Celite 535 (45 gals/sq.ft/hour), purchased from J.T. Baker, was used.
19. An analytically pure sample is isolated by column chromatography on silica gel (see Note 4). The column is eluted with ethyl acetate and the fractions are analyzed by TLC on silica gel (see Note 5) using the same eluent. Fractions containing the product are combined and the solvent is evaporated on a rotary evaporator to give **5** as a white powder. The physical properties of **5** are as follows (Note 14): $[\alpha]_D^{20}$ +121.5° ($CHCl_3$, *c* 1.30); ^{1}H NMR δ: 3.58 (s, 3 H), 6.75-8.01 (m, 22 H); ^{31}P NMR δ: 28.88 (s) ;EIMS m/z 484 (M^+), 453, 282 (base peak) ; HRMS calcd for $C_{33}H_{25}O_2P$ 484.1592, found 484.1574. Anal. Calcd for $C_{33}H_{25}O_2P$: C, 81.80; H, 5.20. Found: C, 81.77; H, 5.38.
20. Triethylamine was purchased from Aldrich Chemical Company, Inc., and used without further purification.
21. Toluene was purchased from Aldrich Chemical Company, Inc., and distilled from calcium hydride before use.
22. Trichlorosilane was purchased from Aldrich Chemical Company, Inc. , and used without further purification. While it is difficult to measure the volume of trichlorosilane used accurately because of its volatility, accurate measurement is not essential in the reduction of **5**. The submitters found that a large excess of trichlorosilane does not interfere with reduction of phosphine oxide in Part E, and recommend the use of trichlorosilane (4 equiv or more to **5**) to complete the reduction in an appropriate reaction time.
23. Crystallization of the crude material from dichloromethane-hexane gave product **6** of 50% yield or lower. For efficiency of isolation, the submitters recommend purifying **6** by column chromatography. The physical properties of product **6** are as follows (see Note 14): $[\alpha]_D^{20}$ +95° ($CHCl_3$, *c* 0.27), $[\alpha]_D^{16}$ +75.7° (benzene, *c* 1.50) [literature rotation for (S)-**6**;[4] $[\alpha]_D^{16}$ −59.3° (benzene, *c* 1.0)]; mp 174-176°C (recrystallization from CH_2Cl_2/n-hexane); ^{1}H NMR δ: 3.34 (s, 3 H), 6.95-8.05 (m, 22 H) ; ^{31}P NMR δ: −12.74 (s) ; EIMS m/z 468 (M^+), 437 (base peak) ; HRMS calcd for $C_{33}H_{25}PO_2$ 468.1643, found 468.1672. Anal. Calcd for $C_{33}H_{25}OP$: C, 84.60; H, 5.38. Found: C, 84.35; H, 5.44.

Waste Disposal Information

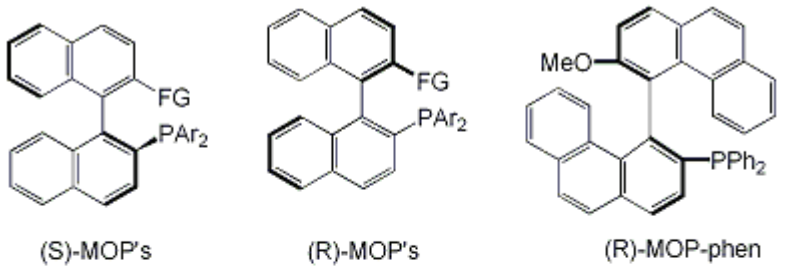
All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

Most of the chiral phosphine ligands prepared so far and used for catalytic asymmetric reactions are the bisphosphines,[5] which are expected to construct an effective chiral environment by bidentate coordination to metal; they have been demonstrated to be effective for several types of asymmetric reactions. On the other hand, there exist transition metal-catalyzed reactions where the bisphosphine-metal complexes cannot be used because of their low catalytic activity and/or low selectivity towards a desired reaction pathway. Therefore chiral monodentate phosphine ligands are required for the realization of new types of catalytic asymmetric reactions. Unfortunately, only a limited number of monodentate chiral phosphine ligands have been reported,[6] which with few exceptions are not so useful as bisphosphine ligands. Recently, the monodentate, optically active phosphine ligand, 2-diphenylphosphino-2'-methoxy-1,1'-binaphthyl (MeO-MOP), and its analogs[7] have been demonstrated to provide high enantioselectivity in palladium-catalyzed hydrosilylation of olefins[8] and palladium-catalyzed reduction of allylic esters by formic acid.[9] The procedures described here allow the convenient preparation of MOP and have advantages over previously published sequences.[4] MeO-MOP can be prepared in five steps from binaphthol without racemization and the overall yield is 72%. The key step in this process is the palladium-catalyzed monophosphinylation of 2,2'-bis (trifluoromethanesulfonyloxy)-1,1'-binaphthyl , which was originally reported by Morgans and co-workers.[3] Under the slightly modified conditions, ditriflate (R)-**2** was efficiently converted into (R)-**3** (87% yield) without racemization. Hydrolysis of the remaining triflate with aqueous sodium hydroxide in 1,4-dioxane and methanol (2/1) gave (R)-**4**. The phenolic hydroxyl group of (R)-**4** was methylated by treatment with methyl iodide in the presence of potassium carbonate in acetone to give (R)-**5**. Reduction of phosphine oxide (R)-**5** was carried out with trichlorosilane and triethylamine[10] and references cited therein. in toluene with heating to give the corresponding phosphine (R)-**6** (86% yield over last 3 steps).

A variety of MOP derivatives bearing various alkoxy or siloxy groups were readily prepared by

changing the reagent used for the alkylation of **4**.[7] Furthermore, the presence of the triflate group[11] in compound **3** allows one to prepare a wide range of MOP derivatives functionalized at the 2'-position. Thus, the 2'-alkyl, carboxyl, cyano, aminomethyl groups, etc. were introduced into the 2'-position of MOP via transition metal-catalyzed cross-coupling, carbonylation, and cyanation reactions.[7,12] Bis (substituted phenyl)phosphino groups were readily introduced into the binaphthyl by the palladium-catalyzed reaction with the corresponding diarylphosphine oxides. The same procedures used for the preparation of **3** were followed with di(p-methoxyphenyl)phosphine oxide and **2**. Subsequent hydrolysis, alkylation, and reduction processes gave 2-di(p-methoxyphenyl)phosphino-MOP.[7] The flexibility of the synthetic route allows fine tuning of the phosphine ligand by the introduction of several types of side chains and control of the steric and electronic effects of the phosphino group. Needless to say, the synthetic procedures shown here can be used for the preparation of MOPs having the (S)-absolute configuration by using (S)-binaphthol as a starting material. In addition, a MOP analog having the biphenanthryl skeleton (MOP-phen) was also prepared from optically active 4,4'-biphenanthrol through the same sequences mentioned above.[9b]



FG = OR, alkyl, Et, COOR, CH_2OH, CN, CH_2NMe_2, etc. (R = H, alkyl, silyl, etc.)

Ar = Ph, C_6H_4OMe-p

References and Notes

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

(R)-2-Diphenylphosphino-2'-methoxy-1,1'-binaphthyl:
Phosphine, (2'-methoxy[1,1'- binaphthalen]-2-yl)diphenyl-, (R)- (12);(145964-33-6)

(R)-2,2'-Bis(trifluoromethanesulfonyloxy)-1,1'-binaphthyl:
Methanesulfonic acid, trifluoro-, [1,1'-binaphthalene]-2,2'-diyl ester, (R)- (12); (126613-06-7)

(R)-(+)-1,1'-Bi-2-naphthol:
[1,1'-Binaphthalene]-2,2'-diol, (R)-(+)- (8);
[1,1'-Binaphthalene]-2,2'-diol, (R)- (9); (18531-94-7)

Pyridine (8,9); (110-86-1)

Trifluoromethanesulfonic anhydride:
Methanesulfonic acid,
trifluoro-, anhydride (8,9); (358-23-6)

(R)-(+)-2-Diphenylphosphinyl-2'-trifluoromethanesulfonyloxy-1,1'-binaphthyl:
Methanesulfonic acid, trifluoro-, 2'-(diphenylphosphinyl)[1,1'-binaphthalen]-2-yl ester, (R)- (12);
(132532-04-8)

Diphenylphosphine oxide:
Phosphine oxide, diphenyl- (8,9); (4559-70-0)

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

1,4-Bis(diphenylphosphino)butane (dppb):
Phosphine, tetramethylenebis[diphenyl- (8);
Phosphine, 1,4-butanediylbis[diphenyl-(9); (7688-25-7)

N,N-Diisopropylethylamine:
Triethylamine, 1,1'-dimethyl- (8);
2-Propanamine, N-ethyl-N-(1-methylethyl)- (9); (7087-68-5)

Dimethyl sufoxide:
Methyl sulfoxide (8);
Methane, sulfinyl bis- (9); (67-68-5)

(R)-(−)-2-Diphenylphosphinyl-2'-hydroxy-1,1'-binaphthyl:
[1,1'-Binaphthalene]-2-ol, 2'-(diphenylphosphinyl)-, (R)- (12); (132548-91-5)

1,4-Dioxane: CANCER SUSPECT AGENT:
p-Dioxane (8);
1,4-Dioxane (9); (123-91-1)

(R)-(+)-2-Diphenylphosphinyl-2'-methoxy-1,1'-binaphthyl:
Phosphine oxide, (2'-methoxy[1,1'-binaphthalen]-2-y1)diphenyl-, (R)- (13); (172897-73-3)

Methyl iodide:
Methane, iodo-(8,9); (74-88-4)

Triethylamine (8);
Ethanamine, N,N-diethyl-(9); (121-44-8)

Trichlorosilane:
Silane, trichloro- (8,9); (10025-78-2)

Diethyl phosphite:
Phosphonic acid, diethyl ester (8,9); (762-04-9)

Sodium (8,9); (7440-23-5)

Phenylmagnesium bromide:
Magnesium, bromophenyl- (8,9); (100-58-3)