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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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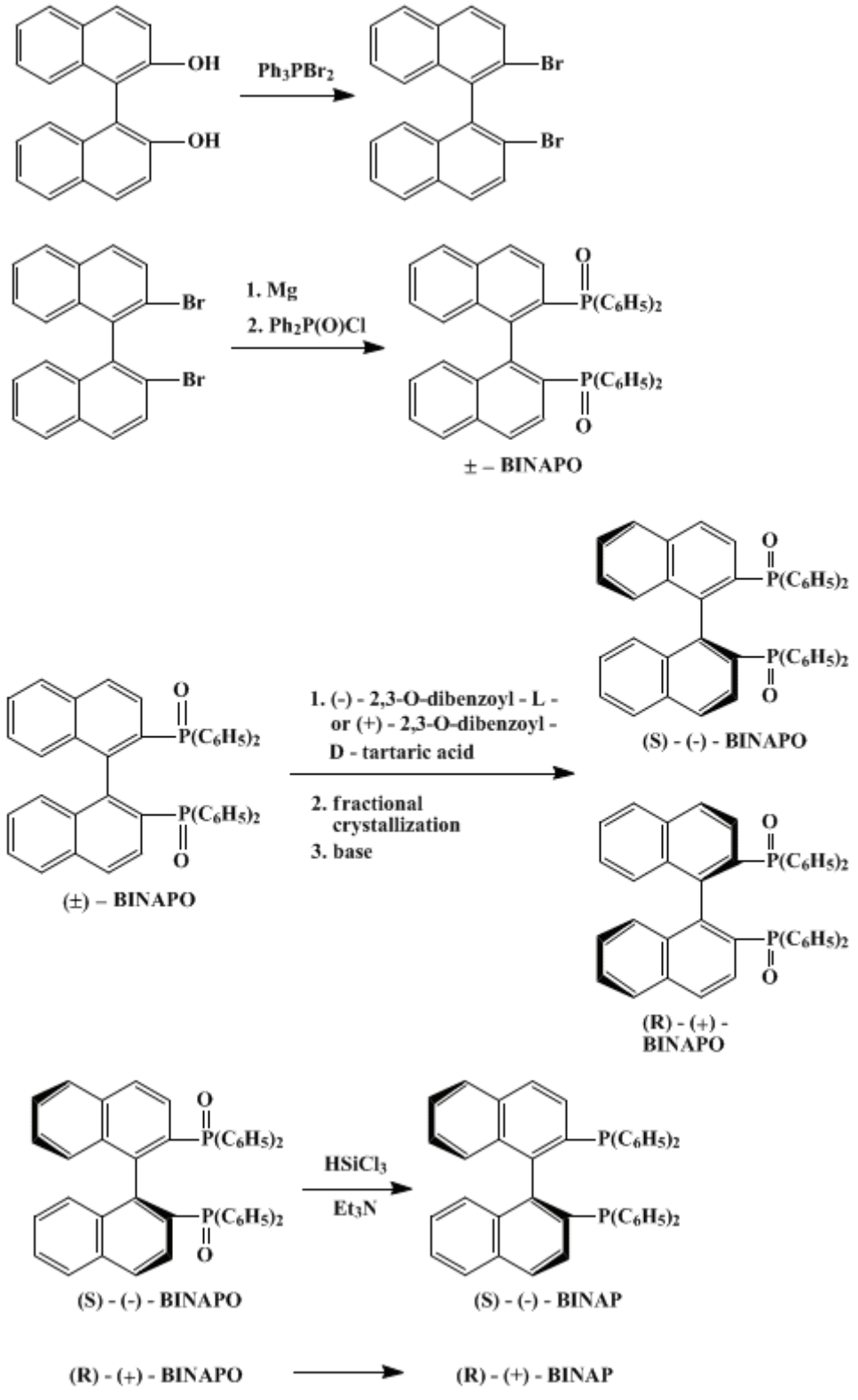
(*R*)-(+)- AND (*S*)-(−)-2,2'-BIS(DIPHENYLPHOSPHINO)-1,1'-BINAPHTHYL (BINAP)

[Phosphine, [1,1'-binaphthalene]-2,2'-diylbis[diphenyl-, (*R*)- or (*S*)-]



Submitted by Hidemasa Takaya[1], Susumu Akutagawa[2], and Ryoji Noyori[3].
Checked by Marco Cereghetti, Alain Rageot, Max Vecchi, and Gabriel Saucy.

1. Procedure

Caution! These operations, which involve toxic reagents, should be conducted in an efficient hood.

A. *2,2'-Dibromo-1,1'-binaphthyl*. A 2-L, three-necked, round-bottomed flask is equipped with an efficient mechanical stirrer, a thermometer, and a dropping funnel. The flask is charged with 240 g (0.915 mol) of triphenylphosphine and 500 mL of dry acetonitrile (Note 1). Stirring is begun and the solid is dissolved by warming the flask with hot water. The solution is then cooled with an ice–water mixture, and to this is added dropwise with stirring 155 g (50 mL, 0.969 mol) of bromine over a 1-hr period. The ice–water bath is removed and 120 g (0.420 mol) of 2,2'-dihydroxy-1,1'-binaphthyl is added to the solution (Note 2). The viscous slurry is stirred at 60°C for 30 min. The flask is now fitted for a simple distillation, and most of the solvent is removed by applying partial vacuum. The last trace of acetonitrile is removed at aspirator vacuum using a bath temperature of 100°C. The temperature of the resulting mass is raised carefully by means of a heating mantle (Note 3) to 240–260°C over a period of 1 hr, at which temperature an exothermic reaction occurs (Note 4), with evolution of hydrogen bromide. After the exothermic reaction subsides, the reaction mixture is further stirred at 260–270°C for 1 hr, and then the temperature is gradually raised and kept at 310–320°C for 30 min to complete the reaction. The reaction mixture, a homogenous melt, is allowed to cool to ca. 200°C with stirring and to this is added 1000 mL of Celite with stirring (Note 5). After the mixture is cooled below 70°C, it is extracted with 500 mL of a boiling 1 : 1 mixture of benzene and hexane. The solid material, separated by filtration through a sintered-glass funnel, is extracted further with three 200-mL portions of a boiling 1 : 1 mixture of benzene and hexane. The combined extracts are evaporated to give an orange-yellow viscous oil, which is dissolved in 200 mL of ethanol. The solution is left in a refrigerator for 2 days (Note 6). 2,2'-Dibromo-1,1'-binaphthyl precipitates and is collected on a sintered-glass funnel to give 90 g of the crude product. Recrystallization from ethanol affords the pure dibromide (78.0 g, 45% yield) as pale-yellow, fine crystals (Note 7).

B. *(±)-2,2'-Bis(diphenylphosphinyl)-1,1'-binaphthyl* [*(±)-BINAPO*]. A 1-L, three-necked, round-bottomed flask is provided with a mechanical stirrer, an addition funnel, a thermometer, and a reflux condenser, the top of which is connected with a bubbler and an argon line by way of a three-way stopcock. The flask is flushed with argon and charged with 2.84 g (0.117 g-atom) of magnesium turnings, 50 mL of dry, degassed tetrahydrofuran (Note 8), 50 mg of iodine, and 0.5 mL of 1,2-dibromoethane. The mixture is stirred at room temperature until the color of iodine fades and evolution of ethylene ceases. The flask is placed in an oil bath, the reaction mixture is stirred and heated at 50–70°C, and 20.0 g (50.0 mmol) of 2,2'-dibromo-1,1'-binaphthyl in 400 mL of dry, degassed toluene (Note 9) is added over a period of 3.5 hr. The mixture is stirred at 75°C for 2 hr and then cooled to 10°C. To this is added dropwise over a 20-min period a solution of 28.4 g (120 mmol) of diphenylphosphinyl chloride (Note 10) in 35 mL of toluene (Note 9) while the temperature is held at 10–15°C. After the addition is completed, the mixture is further stirred at 60°C for 2 hr, and then cooled to 15°C. To the solution is added dropwise 350 mL of 10% aqueous ammonium chloride and the mixture is stirred for another 10 min at 60°C. The organic layer is separated, washed successively with 150 mL of 10% aqueous ammonium chloride, two 150-mL portions of 1 *N* sodium hydroxide, and finally with two 150-mL portions of water. The toluene layer is dried for a short time over anhydrous sodium sulfate (Note 11), filtered, and concentrated under reduced pressure to give 38.8 g of a pale-yellow solid. This crude product is stirred with 150 mL of boiling toluene for a few minutes, and to this is added 100 mL of heptane. The mixture is allowed to stand at room temperature overnight. The solid product is separated by filtration through a sintered-glass funnel and dried at 70°C (0.05 mm) for 2 hr to give 24.5 g (75%) of (±)-BINAPO as a slightly-pale-yellow solid (Note 12). Concentration of the filtrate and recrystallization of the residue twice from 30-mL portions of toluene gives an additional 3.6 g (11%) of (±)-BINAPO. This product is suitable for use in Part C without further purification.

C. *Optical resolution of (±)-BINAPO*. A 2-L, round-bottomed flask is equipped with a magnetic stirring bar and a reflux condenser. The flask is charged with 10.5 g (16.0 mmol) of racemic BINAPO

and 700 mL of chloroform (Note 13). The solid is dissolved by heating at reflux temperature with stirring, followed by rapid addition of a warm solution of 6.0 g (16.0 mmol) of (−)-2,3-*O*-dibenzoyl-L-tartaric acid monohydrate [(−)-DBT monohydrate] (Note 14) in 460 mL of ethyl acetate (Note 13). The mixture is stirred under reflux for 2–3 min and then allowed to stand at room temperature overnight. The crystals formed are collected on a sintered-glass funnel and the filtrate is stored for recovery of (*R*)-(+)-BINAPO (see below). The solid product is dried at room temperature (0.05 mm) for 6 hr to give 7.2 g (89% of theory) of a 1 : 1 complex of (*S*)-BINAPO and (−)-DBT, mp 238–240°C (dec), $[\alpha]_D^{25}$ −170° (ethanol, c 0.503) (Note 15).

This complex (7.1 g, 7.0 mmol) is treated with 150 mL of 0.75 *N* aqueous sodium hydroxide and the mixture is extracted with two 150-mL portions of chloroform. The combined organic layers are washed with 100 mL of 0.75 *N* sodium hydroxide and water and dried over anhydrous sodium sulfate. The drying agent is removed by filtration and the solvent is evaporated. The residue is washed with 20 mL of cold ethyl acetate to furnish 5.3 g of white solid, which is dried at 80°C (0.05 mm) overnight to give 4.6 g (100% based on the complex used) of (*S*)-BINAPO, mp 256–258°C, $[\alpha]_D^{25}$ −392° (benzene, c 0.530) (Note 16).

The mother liquor and the filtrate from the first resolution (see above) are combined and concentrated to dryness to give 9.0 g of solid material ((*R*)-BINAPO and (−)-DBT) after being dried at 80°C (0.05 mm) for 3 hr, mp 228–230°C (dec). This solid is treated with 150 mL of 0.75 *N* aqueous sodium hydroxide and extracted with two 150-mL portions of chloroform. The combined extract is washed with 70 mL of 0.75 *N* sodium hydroxide, two 100-mL portions of water, and dried over sodium sulfate. The drying agent is removed by filtration and the filtrate is evaporated to give 7.7 g of colorless solid, which is dried at 80°C overnight to afford 5.9 g (9.0 mmol) of crude (*R*)-BINAPO, mp 249–251° C, $[\alpha]_D^{20}$ +304° (benzene, c 0.522). This recovered (*R*)-BINAPO is dissolved in 350 mL of refluxing chloroform, and to this added is added with stirring a solution of 3.4 g (9.0 mmol) of (+)-DBT monohydrate in 280 mL of warm ethyl acetate. The mixture is stirred at reflux temperature for 5 min and then allowed to stand at room temperature overnight. The white precipitates are collected on a sintered-glass funnel, washed with two 20-mL portions of cold ethyl acetate, and dried at 70°C (0.05 mm) for 12 hr to give 7.5 g [92% yield based on the initially used (*R*)-BINAPO] of the (*R*)-BINAPO-(+)-DBT-complex, mp 235–236°C (dec), $[\alpha]_D^{25}$ +172° (ethanol, c 0.527).

This complex (7.3 g, 7.2 mmol) is treated with 200 mL of 0.75 *N* aqueous sodium hydroxide and extracted twice with 150-mL portions of chloroform. The combined chloroform layer is washed with 60 mL of 0.75 *N* aqueous sodium hydroxide and two 100-mL portions of water and is dried over anhydrous sodium sulfate, and filtered. Evaporation of the filtrate affords 5.25 g of colorless solid that is dried at 80°C (0.05 mm) to give 4.65 g (99% yield based on the complex used) of (*R*)-BINAPO, mp 256–258°C, $[\alpha]_D^{25}$ +388° (benzene, c 0.514) (Note 17).

D. *Reduction of (S)-(−)-BINAPO to (S)-(−)-BINAP.* In a 300-mL, three-necked flask, fitted with a magnetic stirring bar, a thermometer, and a reflux condenser which is connected through a three-way stopcock to an argon inlet tube and a bubbler, is placed 4.5 g (6.9 mmol) of (*S*)-BINAPO. The flask is flushed with argon followed by the addition of 100 mL of dry, degassed xylene (Note 9), 4.2 mL of triethylamine (3.1 g, 30 mmol) (Note 9), and 3.0 mL (4.0 g, 29 mmol) of trichlorosilane (Note 13) by means of syringes. The mixture is stirred and heated at 100°C for 1 hr, at 120°C for 1 hr, and finally at refluxing temperature for 6 hr (Note 18). After the solution is cooled to room temperature, 70 mL of 30% aqueous sodium hydroxide solution is carefully added. The mixture is then stirred at 60°C until the organic and aqueous layers become clear, and it is transferred into a 300-mL separatory funnel. The organic layer is separated, and the aqueous layer is extracted with two 50-mL portions of warm toluene. The combined organic layer is washed with 70 mL of 30% sodium hydroxide solution and three 100-mL portions of water, and then dried over anhydrous sodium sulfate. The organic layer is concentrated under reduced pressure to a volume of about 15 mL, and to this added 15 mL of degassed methanol. The precipitates are collected on a sintered-glass funnel, washed with 15 mL of methanol, and dried at 80°C (0.05 mm) for 6 hr to give 4.2 g (97% yield) of (*S*)-BINAP as colorless solid, mp 236–238°C, $[\alpha]_D^{25}$ −223° (benzene, c 0.502) (Note 19) and (Note 20).

2. Notes

1. Reagent-grade acetonitrile was dried over Linde 3A molecular sieves and then heated at reflux for several hours over calcium hydride and distilled under dry argon.
2. Commercial reagent grade 2,2'-dihydroxy-1,1'-binaphthyl from Aldrich Chemical Company, Inc. (1,1'-bi-2-naphthol) was used as obtained. It also can be prepared by the oxidative coupling of β-naphthol with ferric chloride[4] and used after recrystallization from ethanol and then benzene.
3. In order to obtain a homogeneous melt, the checkers had to raise the bath temperature to 300–335°C. They preferred a Woods metal bath.
4. Temperature should be carefully controlled. Too rapid heating can result in an uncontrollably vigorous reaction.
5. This facilitates the smooth extraction of the products.
6. This procedure removes triphenylphosphine oxide.
7. The product melts at 184–185°C (lit.[5] mp 180°C), R_f 0.50 (E. Merck Kieselgel 60 PF_{254}, 1 : 4 benzene–hexane).
8. Reagent-grade tetrahydrofuran was distilled from sodium benzophenone ketyl under argon before use.
9. Commercial guaranteed-grade solvents were distilled over finely powdered calcium hydride under argon before use.
10. Commercial reagent-grade diphenylphosphinyl chloride from Aldrich Chemical Company, Inc. was used as obtained. This compound can be prepared either by oxidation of diphenylphosphinous chloride with dimethyl sulfoxide[6] or by the treatment of diphenylphosphinic acid with phosphorus pentachloride.[7]
11. Prolonged standing of the solution at room temperature may cause precipitation of (±)-BINAPO. In such a case, warm chloroform can be used to dissolve the solid.
12. The checkers obtained first crops ranging from 76 to 84%, mp 295.5–297°C. The submitters report mp 299–300°C. An analytically pure sample was obtained by recrystallization from a mixture of hexane and toluene, mp 304–306°C. One gram of (±)-BINAPO dissolves in 28 mL of boiling toluene.
13. Commercial reagent-grade chemicals were used.
14. Guaranteed-grade (−)-2,3-*O*-dibenzoyl-L-tartaric acid monohydrate and its enantiomer were purchased from Tokyo Kasei Kogyo Co., Ltd., and used without further purification.
15. The checkers had yields ranging from 69 to 90%. They determined the enantiomeric purity of the *S*-BINAPO component, $[\alpha]_D^{20}$ −168° (ethanol, c 0.5), to be 99.6/0.4 using a Pirkle column (Note 16). The submitters report that recrystallization from a 1 : 2 mixture of ethyl acetate and chloroform gave an analytically pure sample, mp 240–241°C (dec), $[\alpha]_D^{24}$ −174° (ethanol, c 0.523).
16. The checkers obtained first crops of 87% and 75.8%, mp 263–263.5°C, $[\alpha]_D^{20}$ −389° (benzene, c 0.5), and mother liquors of 11% and 13.5%, respectively. These materials were analyzed on a Pirkle column (Baker bond II) with hexane/ethanol mixtures and found to have *S/R* ratios of 99.7/0.3 (first crop) and 93/1.7 (mother liquor). The submitters report obtaining analytically pure (*S*)-BINAPO by recrystallization from a mixture of hexane and toluene, mp 261–262°C, $[\alpha]_D^{24}$ −396° (benzene, c 0.467).
17. The submitters obtained analytically pure (*R*)-BINAPO by recrystallization from a mixture of hexane and toluene, mp 262–263°C, $[\alpha]_D^{24}$ +399° (benzene, c 0.500). See (Note 16) for determination of optical purity. The checkers found an *R/S* ratio of 98.8/1.2 for unrecrystallized material, mp 261–263°C, $[\alpha]_D^{20}$ +379° (benzene, c 0.5).
18. During this period a white solid forms at the bottom of the reflux condenser. Use of a ground-glass joint as large as possible is recommended to avoid clogging.
19. GLC analysis (OV-101, capillary column, 5 m, 200–280°C) indicates that the product has a purity of 97%. Trace amounts of BINAPO and the monooxide of BINAP were detected by TLC analysis (E. Merck Kieselgel 60 PF_{254}, 1 : 19 methanol-$CHCl_3$); R_f 0.42 (BINAPO), 0.67 (monooxide of BINAP), and 0.83 (BINAP). The submitters report that recrystallization from a 1 : 1 mixture of toluene and ethanol affords optically pure (*S*)-BINAP, mp 241–242°C, $[\alpha]_D^{25}$ −229° (benzene, c 0.312). The checkers oxidized a sample of first-crop material, mp 241–242.5°C, $[\alpha]_D^{20}$ −221° (benzene, c 0.5), for Pirkle analysis (see (Note 16)). This gave an *S/R* ratio of 98.2/1.7.
20. The checkers also reduced (*R*)-(+)-BINAPO to (*R*)-(+)-BINAP by this procedure. In the best of two runs, first-crop material (94.8%), mp 241–242°C, $[\alpha]_D^{20}$ +217° (benzene, c 0.5), with an *R/S* ratio of 99.0/0.8 was obtained.

3. Discussion

BINAP is a new type of fully aryl-substituted diphosphine with only an axial element of chirality. Optically pure BINAP was first synthesized by the optical resolution of (±)-BINAP using optically active di-μ-chlorobis[(*S*)-*N,N*-dimethyl-α-phenylethyl-amine-2*C,N*]dipalladium.[8] [9] The phosphine is also obtained by stereospecific transformation of optically active 2,2'-dibromo-1,1'-binaphthyl.[9,10] The procedure outlined here,[11] however, is the best preparative-scale synthesis of both enantiomers of BINAP in an optically pure state starting from easily accessible racemic 2,2'-dihydroxy-1,1'-binaphthyl. This method is applicable to various BINAP analogues.[11] The absolute configuration of (+)-BINAP was determined to be *R* by X-ray analysis of the complex [Rh((+)-binap)(norbornadiene)]ClO_4.[12] BINAP serves as an excellent ligand for the Rh(I)-catalyzed asymmetric hydrogenations of α-(acylamino) acrylic acids and esters.[8,9] The ligand has also been successfully applied to the Rh(I)-catalyzed asymmetric isomerization of diethylnerylamine or diethylgeranylamine into citronellal (*E*)-*N,N*-diethylenamine (this volume, p. 183).[13] [14] [15] This reaction is now used for commercial production of (−)-methanol. In addition, BINAP-based Ru(II) complexes[16] catalyze highly enantioselective hydrogenation of alkyl- or aryl-substituted acrylic acids,[17] [18] enamides leading to isoquinoline alkaloids,[19] [20] allylic and homoallylic alcohols,[21] [22] β-keto esters,[23] [24] [25] [26] [27] other functionalized ketones,[28] [29] and other compounds.[30] [31]

This preparation is referenced from:

- Org. Syn. Coll. Vol. 8, 183
- Org. Syn. Coll. Vol. 9, 169
- Org. Syn. Coll. Vol. 9, 589
- Org. Syn. Coll. Vol. 10, 112

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1. Chemical Materials Center, Institute for Molecular Science, Myodaiji, Okazaki 444, Japan;
2. Central Research Laboratory, Takasago Perfumery Co., Ltd., 5-Chome, Kamata, Ohtaku, Tokyo 144, Japan;
3. Department of Chemistry, Nagoya University, Chikusa, Nagoya 464, Japan.
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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

sodium benzophenone ketyl

(R)-(+)- AND (S)-(−)-2,2'-BIS(DIPHENYLPHOSPHINO)-1,1'-BINAPHTHYL (BINAP)

(±)-2,2'-Bis(diphenylphosphinyl)-1,1'-binaphthyl [(±)-BINAPO]

(±)-BINAPO

BINAPO

(−)-2,3-O-dibenzoyl-L-tartaric acid monohydrate [(−)-DBT monohydrate

(R)-(+)-BINAPO

(S)-BINAPO

(−)-DBT

((R)-BINAPO

(−)-DBT)

(R)-BINAPO

(+)-DBT monohydrate

(R)-BINAPO-(+)-DBT-complex

(S)-(−)-BINAPO

(S)-BINAP

(−)-2,3-O-dibenzoyl-L-tartaric acid monohydrate

S-BINAPO

R)-(+)-BINAPO

(R)-(+)-BINAP

BINAP

(±)-BINAP

di-μ-chlorobis[(S)-N,N-dimethyl-α-phenylethyl-amine-2C,N]dipalladium

(+)-BINAP

citronellal (E)-N,N-diethylenamine

Phosphine, [1,1'-binaphthalene]-2,2'-diylbis[diphenyl-, (R)- or (S)-

ethanol (64-17-5)

Benzene (71-43-2)

ethyl acetate (141-78-6)

methanol (67-56-1)

ammonium chloride (12125-02-9)

acetonitrile (75-05-8)

sodium hydroxide (1310-73-2)

phosphorus pentachloride (10026-13-8)

chloroform (67-66-3)

magnesium turnings (7439-95-4)

hydrogen bromide (10035-10-6)

bromine (7726-95-6)

sodium sulfate (7757-82-6)

β-naphthol (135-19-3)

iodine (7553-56-2)

toluene (108-88-3)

ethylene (9002-88-4)

1,2-dibromoethane (106-93-4)

ferric chloride (7705-08-0)

xylene (106-42-3)

Tetrahydrofuran (109-99-9)

heptane (142-82-5)

phosphine (7723-14-0)

hexane (110-54-3)

dimethyl sulfoxide (67-68-5)

triethylamine (121-44-8)

argon (7440-37-1)

calcium hydride (7789-78-8)

triphenylphosphine (603-35-0)

triphenylphosphine oxide (791-28-6)

homoallylic alcohols (627-27-0)

diphenylphosphinous chloride (1079-66-9)

TRICHLOROSILANE (10025-78-2)

diphenylphosphinyl chloride (1499-21-4)

diphenylphosphinic acid (1707-03-5)

diethylnerylamine (40137-00-6)

diethylgeranylamine (40267-53-6)

1,1'-bi-2-naphthol,
2,2'-dihydroxy-1,1'-binaphthyl (18531-99-2)

(S)-(−)-BINAP (76189-56-5)

2,2'-Dibromo-1,1'-binaphthyl (74866-28-7)