CLAIMS

What is claimed is:

1.A method for purifying a crude PNPNH compound of the general structure

R_1R_2P-$N(R_3)$-$P(R_4)$-$N(R_5)$-H

wherein R_1, R_2, R_3, R_4 and R_5 are independently halogen, amino, trimethylsilyl, C_1-C_{10}-alkyl, substituted C_1-C_{10}-alkyl, C_6-C_{20}-aryl and substituted C_6-C_{20}-aryl, or any cyclic derivative wherein at least one of the P or N atoms of the PNPN-H structure is a member of a ring system, the ring system being formed from one or more constituent compounds of the PNPNH-structure by substitution,

comprising the steps:

A.

a) dissolving the crude PNPNH-compound in a C_1-C_{10} alcohol or mixture thereof under heating;

b) cooling the solution obtained in step a); and

c) isolating the PNPNH compound, preferably by precipitating the PNPNH compound from the solution, separating the PNPNH compound from the solution and optionally drying the PNPNH compound; or

B.

(i) washing the crude PNPNH compound with C_1-C_{10} alcohol or a mixture thereof;

(ii) separating the PNPNH compound; and optionally drying thereof.

2.The method according to claim 1, wherein the crude PNPNH compound is dissolved in step a) in or is washed in step (i) with a C_1-C_5 alcohol, preferably ethanol.

3.The method according to claim 1 or 2, wherein the ethanol/ligand weight ratio is from 5.0-0.1, preferably 1.0-0.3.

4.The method according to any of the preceding claims, wherein heating in step a) is to a temperature of about 50-100°C.

5.The method according to any of the preceding claims, wherein the solution in step b) is cooled to temperature between -20 to +20°C, preferably 0 to 10°C.

6.The method according to any of the preceding claims, wherein PNPNH seed crystals are added before or during step b) and/or c).

7.The method according to any of the preceding claims, wherein the precipitate obtained in step c) is washed with C_1-C_5 alcohol, preferably at a temperature of 0-25°C, and is then dried at 10-50°C, preferably 15-25°C, in vacuum of 5.0-500 millibar, preferably 10-100 millibar.

8.The method according to any of the preceding claims, wherein washing in step i) is at a temperature of about 0-25°C, preferably 15-25°C.

9.The method according to any of the preceding claims, wherein the C_1-C_{10} alcohol is removed in step ii) by drying at 10-50°C, preferably 15-25°C, in vacuum of 5.0-500 millibar, preferably 10-100 millibar.

CLAIMS

1. A method for purifying a crude PNPNH compound comprising a ligand of the general structure

R_1R_2P-$N(R_3)$-$P(R_4)$-$N(R_5)$-H

wherein R_1, R_2, R_3, R_4 and R_5 are independently halogen, amino, trimethylsilyl, C_1-C_{10}-alkyl, substituted C_1-C_{10}-alkyl, C_6-C_{20}-aryl and substituted C_6-C_{20}-aryl, or any cyclic derivative wherein at least one of the P or N atoms of the PNPN-H structure is a member of a ring system, the ring system being formed from one or more constituent compounds of the PNPNH-structure by substitution, wherein the substitution is by formally eliminating per constituent compound either two whole groups R_1-R_5 or H, one atom from each of two groups R_1-R_5 or a whole group R_1-R_5 or H and an atom from another group R_1-R_5, and joining the formally so created valence-unsaturated sites by one covalent bond per constituent compound to provide the same valence as initially present at a given site,

the method comprising:

A.

a) dissolving the crude PNPNH-compound in a C_1-C_{10} alcohol or mixture thereof under heating, wherein an alcohol/ligand weight ratio is from 1.0-0.3;

b) cooling the solution obtained in step a); and

c) isolating the PNPNH compound; or

B.

(i) washing the crude PNPNH compound with C_1-C_{10} alcohol or a mixture thereof;

(ii) separating the PNPNH compound; and optionally drying thereof.

2. The method according to claim 1, wherein the crude PNPNH compound is dissolved in step a) in or is washed in step (i) with a C_1-C_5 alcohol.

3. The method according to claim 1, wherein an alcohol/ligand weight ratio in step B. (i) is from 5.0-0.1.

4. The method according to claim 1, wherein the heating in step A. a) is to a temperature of about 50-100°C.

5. The method according to claim 1, wherein the solution in step A. b) is cooled to temperature between -20 to +20°C.

6. The method according to claim 1, wherein PNPNH seed crystals are added before or during step A. b) and/or A. c).

7. The method according to claim 1, wherein a precipitate obtained in step A. c) is washed with C_1-C_5 alcohol, and is then dried at 10-50°C, in a vacuum of 5.0-500 millibar.

8. The method according to claim 1, wherein the washing in step B. (i) is at a temperature of about 0-25°C.

9. The method according to claim 1, wherein the C_1-C_{10} alcohol is removed in step B. (ii) by drying at 10-50°C, in a vacuum of 5.0-500 millibar.

10. The method according to claim 1, wherein the crude PNPNH compound comprises an impurity comprising ligand synthesis products, ligand reaction products with oxygen and/or water, or a combination thereof.