Claims

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

$R_1R_2P\text{-}N(R_3)\text{-}P(R_4)\text{-}N(R_5)\text{-}H$

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:

a) dissolving the PNPNH-compound in a first solvent;

b) metalating the PNPNH-compound,

c) isolating the metalated compound obtained in step b), preferably by precipitating the metalated compound obtained in step b), separating from the solvent and optionally washing with the solvent,

d) re-protonating the metalated compound in a second solvent, and

e) optionally removing the solvent and optionally recrystallizing the re-protonated PNPNH ligand.

2. The method according to claim 1, wherein the first and second solvents are identical.

3. The method according to claim 1, wherein the first and/or second solvent is a non-polar solvent, preferably an aromatic and/or aliphatic solvent, preferably toluene, n-hexane, cyclohexane, 1-hexene, or a combination comprising at least one of the foregoing.

4. The method according to any of the preceding claims, wherein metalating in step b) i by adding an organometallic compound, a base, sodium metal, or potassium metal in an amount equivalent to or in excess of the molar concentration of the PNPNH ligand into the solution obtained in step a).

5. The method according to claim 4, wherein metalation is by adding n-butyl lithium, sec-butyl lithium, tert-butyl lithium, sodium cyclopentadienide, sodium hydride, sodium amide, an alkyl- or aryl magnesium halide, sodium bis(trimethylsilyl) amide,

dialkylmagnesium, diarylmagnesium, trialkylaluminium, dialkylzinc, sodium metal, or potassium metal, preferably n-butyl lithium.

6. The method according to any of the preceding claims, wherein re-protonation in step d) is achieved by addition of an acid, preferably a weak acid.

7. The method according to claim 6, wherein the acid is an ammonium halogenide, preferably ammonium chloride, phosphoric acid, hydrogen sulphide or boric acid.

Claims

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

$R_1R_2P\text{-}N(R_3)\text{-}P(R_4)\text{-}N(R_5)\text{-}H$

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:

a) dissolving the PNPNH-compound in a first solvent;
b) metalating the PNPNH-compound,
c) isolating the metalated compound obtained in step b),
d) re-protonating the metalated compound in a second solvent, and
e) optionally removing the second solvent and optionally recrystallizing the re-protonated PNPNH ligand.

2. The method according to claim 1, wherein the first and second solvents are identical.

3. The method according to claim 1, wherein the first and/or second solvent is a non-polar solvent, or a combination comprising at least one of the foregoing.

4. The method according to claim 1, wherein metalating in step b) is by adding an organometallic compound, a base, sodium metal, or potassium metal in an amount equivalent to or in excess of the molar concentration of the PNPNH ligand into the solution obtained in step a).

5. The method according to claim 4, wherein metalation is by adding n-butyl lithium, sec-butyl lithium, tert-butyl lithium, sodium cyclopentadienide, sodium hydride, sodium amide, an alkyl- or aryl magnesium halide, sodium bis(trimethylsilyl) amide, dialkylmagnesium, diarylmagnesium, trialkylaluminium, dialkylzinc, sodium metal, or potassium metal.

6. The method according to claim 1, wherein re-protonation in step d) is achieved by addition of an acid.

7. The method according to claim 6, wherein the acid is an ammonium halogenide, phosphoric acid, hydrogen sulphide or boric acid.

8. The method according to claim 7, wherein the acid is ammonium chloride.

9. A method for purifying a crude PNPN-H compound comprising a PNPN-H ligand of the general structure

$R_1R_2P\text{-}N(R_3)\text{-}P(R_4)\text{-}N(R_5)\text{-}H$

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 PNPN-H-structure by substitution, and an impurity,

the method comprising:

(i) dissolving the crude PNPN-H compound in a first solvent;

(ii) metalating the PNPN-H ligand,

(iii) separating the metalated PNPN-H ligand from the first solvent and the impurity,

(iv) re-protonating the metalated PNPN-H ligand in a second solvent, and

(v) optionally removing the second solvent and optionally recrystallizing the re-protonated PNPNH ligand.

to provide a purified PNPN-H compound.

10. The method according to claim 9, wherein the impurity comprises PNPN-H ligand synthesis products, PNPN-H ligand reaction products with oxygen and/or water, or a combination thereof.

11. The method according to claim 10, wherein the purified PNPN-H compound has a PNPN-H ligand purity greater than 99.9 wt-%.

12. The method according to claim 11, wherein the first solvent and second solvent are the same.

13. The method according to claim 12, wherein the first and second solvent is a non-polar solvent.

14. The method according to claim 13, wherein metalating in step (ii) is by adding an organometallic compound, a base, sodium metal, or potassium metal in an amount equivalent to or in excess of the molar concentration of the PNPN-H ligand into the solution obtained in step (i).

15. The method according to claim 14, wherein metalation is by adding n-butyl lithium, sec-butyl lithium, tert-butyl lithium, sodium cyclopentadienide, sodium hydride, sodium amide, an alkyl- or aryl magnesium halide, sodium bis(trimethylsilyl) amide, dialkylmagnesium, diarylmagnesium, trialkylaluminium, dialkylzinc, sodium metal, or potassium metal.

16. The method according to claim 15, wherein re-protonation in step (iv) is achieved by addition of an acid.

17. The method according to claim 16, wherein the acid is an ammonium halogenide, phosphoric acid, hydrogen sulphide or boric acid.

18. The method according to claim 17, wherein metalation is by adding n-butyl lithium and the acid is ammonium chloride.