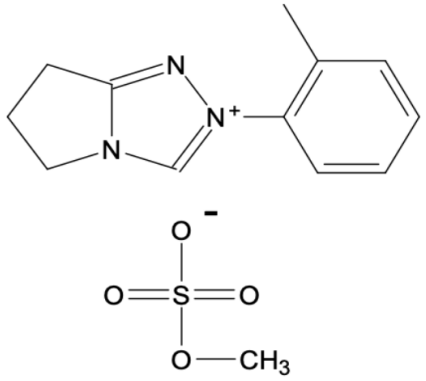
Claims

1. A method for the synthesis of an N-Heterocyclic Carbene catalyst salt of Formula 1,



Formula 1

the method comprising: (a) contacting 2-methylaniline with aqueous hydrochloric acid to form the amine chloride; (b) contacting the amine chloride in solution with a diazotization reagent to form the diazonium chloride salt; (c) adding a reducing agent to convert the diazonium chloride salt to 2-methylphenylhydrazine hydrochloride; (d) filtering to recover the 2-methylphenylhydrazine hydrochloride salt as a solid; (e) contacting the recovered 2-methylphenylhydrazine hydrochloride salt with an aqueous base to form free 2-methylphenylhydrazine; (f) extracting the 2-methylphenylhydrazine from the aqueous basic solution with an organic solvent to provide a solution of the 2-methylphenylhydrazine in the organic solvent; (g) drying the solution of 2-methylphenylhydrazine by addition of a drying agent; (h) removing the drying agent by filtration; (i) contacting the dry 2-methylphenylhydrazine solution with the reaction products of 2-pyrrolidine and dimethylsulfate to make iminohydrazone of Formula 2 in an organic solvent; (j) distilling the solution of the iminohydrazone of Formula 2 to remove excess solvents; (k) recovering the iminohydrazone of Formula 2 as a solid salt; (l) contacting the iminohydrazone salt of Formula 2 with an organic solvent and trimethylorthoformate to make methylsulfate salt of the N-Heterocyclic Carbene catalyst of Formula 1; (m) recovering the N-Heterocyclic Carbene catalyst of Formula 1 as a salt.

2. A method as in claim 1, wherein the diazotization reagent is sodium nitrite ($NaNO_2$).

3. A method as in claim 1, wherein the reducing agent is stannous dichloride.

4. A method as in claim 1, wherein the temperature in steps (a), (b) and (c) is maintained locally, where locally means 250 cc volume increments, throughout the reactor between 0 degrees C and 5 degrees C.

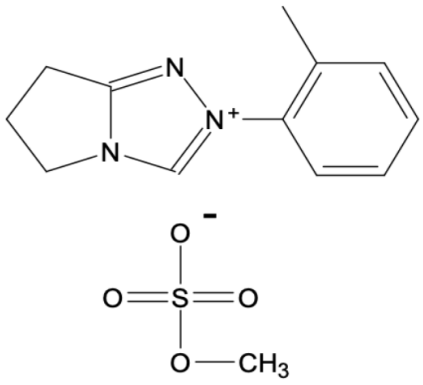
5. A method as in claim 1, wherein the temperature in step (i) is 60 degrees C to 80 degrees C.

6. A method as in claim 1, wherein and the temperature in step (l) is 80 degrees C to 100 degrees C.

7. A method as in claim 1, wherein the solvent in steps (f), (i) and (l) is an aromatic hydrocarbon.

8. A method as in claim 1, wherein the solvent in steps (f), (i) or (l) is selected from the group of toluene, a mixture of xylenes, m-xylene, o-xylene, p-xylene.

9. A method for the synthesis of an N-Heterocyclic Carbene catalyst salt of Formula 1,

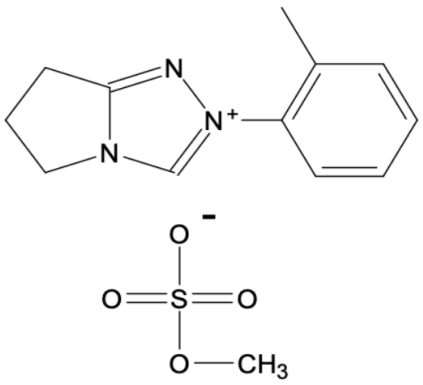


Formula 1

the method comprising (e) contacting 2-methylphenylhydrazine hydrochloride salt with an aqueous base to form free 2-methylphenylhydrazine; (f) extracting the 2-methylphenylhydrazine from the aqueous basic solution with an organic solvent to provide a solution of the 2-methylphenylhydrazine in the organic solvent; (g) drying the solution of 2-methylphenylhydrazine by addition of a drying agent; (h) removing the drying agent by filtration; (i) contacting the dry 2-methylphenylhydrazine solution with the reaction products of 2-pyrrolidine and dimethylsulfate to make iminohydrazone of Formula 2 in an organic solvent; (j) distilling the solution of the iminohydrazone of Formula 2 to remove excess solvents; (k) recovering the iminohydrazone of Formula 2 as a solid salt; (l) contacting the iminohydrazone salt of Formula 2 with an organic

solvent and trimethylorthoformate to make methylsulfate salt of the N-Heterocyclic Carbene catalyst of Formula 1; (m) recovering the N-Heterocyclic Carbene catalyst of Formula 1 as a salt.

10. A method for the synthesis of an N-Heterocyclic Carbene catalyst salt comprising of Formula 1,



Formula 1

the method comprising (i) contacting dry 2-methylphenylhydrazine solution with the reaction products of 2-pyrrolidine and dimethylsulfate to make iminohydrazone of Formula 2 in an organic solvent; (j) distilling the solution of the iminohydrazone of Formula 2 to remove excess solvents; (k) recovering the iminohydrazone of Formula 2 as a solid salt; (l) contacting the iminohydrazone salt of Formula 2 with an organic solvent and trimethylorthoformate to make methylsulfate salt of the N-Heterocyclic Carbene catalyst of Formula 1; (m) recovering the N-Heterocyclic Carbene catalyst of Formula 1 as a salt.

11. The method of claim 1, wherein tin salts are recovered from the filtrate after step (d) by neutralization with a hydroxide base to produce stannic hydroxide which is subsequently filtered and dried to produce stannic oxide (SnO_2) for reprocessing to tin and subsequently to the stannous dichloride.