



Palladium-Catalyzed Cross-Coupling of C-H Bonds

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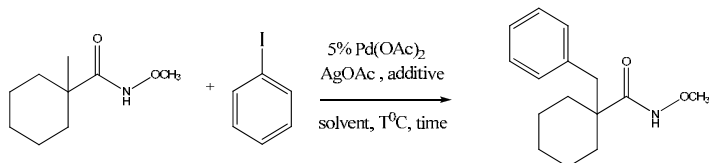


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Introduction

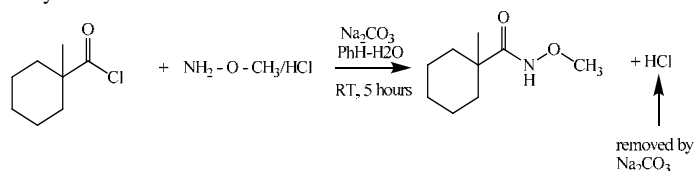
The goal of our research group is to develop new and useful reactions to produce novel materials via carbon-hydrogen bond activation. Our group has developed an effective general method for the coupling of R-CH₃ and Ar'-I by using palladium (II) acetate via activation of carbon-hydrogen bonds to achieve arylation of sp³ C-H bonds. The development of this method allows decreasing the cost and the number of steps needed to obtain such materials.

Coupling Reaction

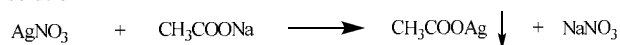


Technical Procedure

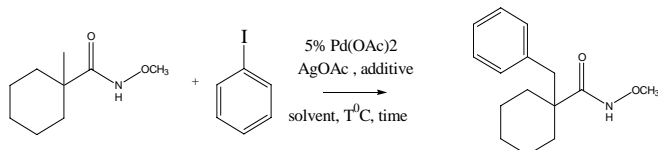
STEP 1: Synthesize Weinreb amide substrate



STEP 2: Prepare silver acetate reagent by mixing silver nitrate solution and sodium acetate solution



STEP 3: Optimize conditions to achieve the good yield for coupling reaction



Optimizing Results

Entry	Temperature	Time(Hours)	Additive	Solvent	Yield(%)
1	50°C	24	N/A	CH ₃ COOH	0
2	50°C	24	N/A	t-BuOH	0
3	50°C	24	N/A	C ₂ H ₄ Cl ₂	0
4	50°C	24	NaOAc	CH ₃ COOH	0
5	50°C	24	NaOAc	t-BuOH	30
6	50°C	24	NaOAc	C ₂ H ₄ Cl ₂	0
7	60°C	24	NaOAc	t-BuOH	50
8	60°C	36	NaOAc	t-BuOH	72
9	70°C	24	NaOAc	t-BuOH	0
10	80°C	24	NaOAc	t-BuOH	0

Discussion

- Although the yield of reaction increases as temperature increases, it will give undesired product if the temperature is above 70°C. It also may cause formation of diphenyl.
- The optimization is still in progress with increasing ratio of amount of substrate to amount of iodobenzene and the time of reactions up to 48 hours.

Conclusion and Future Work

- Tert-butyl alcohol is a good solvent for this substrate and the reaction should run at least 24 hours and not exceed 70°C.
- In the future, reaction will be extended to other substrates.
- We acknowledge the Welch Foundation (Grant E-1571) for partial support of this research.