

New

CHEMISTRY

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# Paraffin–Ni(cod)<sub>2</sub> Capsule for Use in Ni-Catalyzed Cross-Couplings on the Benchtop

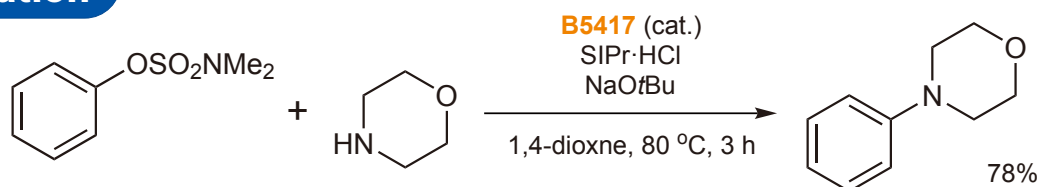


Ni(COD)<sub>2</sub>  
(Wax encapsulated)  
[B5417]

## Advantages

- No Glovebox Handling Required
- General Use in Ni(cod)<sub>2</sub>-Mediated Reactions
- Long-Term Air and Moisture Stability

## Application



### Experimental Procedure:

A vial containing a magnetic stir bar is flame-dried under reduced pressure, and then allowed to cool under N<sub>2</sub>. The vial is charged with morpholine (52.4 μL, 0.600 mmol, 1.2 eq.), sulfamate (100.5 mg, 0.500 mmol, 1.0 eq.), anhydrous powdered NaOtBu (72.0 mg, 0.750 mmol, 1.5 eq.), SIPr·HCl (42.7 mg, 0.100 mmol, 20 mol%), and **B5417** (13.8 mg, 0.050 mmol, 10 mol%). The vial is flushed with N<sub>2</sub>, and subsequently 1,4-dioxane (2.5 mL, 0.20 M) is added. The vial is capped with a Teflon-lined screw cap under a flow of N<sub>2</sub> and the reaction mixture is stirred at 80 °C for 3 h. After removing the vial from heat, the reaction mixture is transferred to a 100 mL round bottom flask containing 3.0 g of silica gel with hexane (6.0 mL) and CH<sub>2</sub>Cl<sub>2</sub> (6.0 mL). The mixture is adsorbed onto the silica gel under reduced pressure and filtered over a plug of silica gel (100 mL of hexanes eluent to remove paraffin, then 100 mL of 1:1 Hexanes:EtOAc eluent). The volatiles are removed under reduced pressure, and the crude residue is purified by preparative thin-layer chromatography (19:1 Benzene:EtOAc) to yield amine (78% yield, average of two experiments) as a white solid.

J. E. Dander, N. A. Weires, N. K. Garg, *Org. Lett.* **2016**, *18*, 3934.

Ni(COD)<sub>2</sub> (= Bis(1,5-cyclooctadiene)nickel(0)) (Wax encapsulated) (ca. 0.05 mmol/capsule)  
5 each [B5417]

## Related Products

Ni(COD)<sub>2</sub>

5g [B3095]

SIPr·HCl (= 1,3-Bis(2,6-diisopropylphenyl)imidazolium Chloride)

500mg / 1g / 5g [B3157]

For further information please refer to our website at [www.TCIchemicals.com](http://www.TCIchemicals.com).

nickel catalysts



# Paraffin–Ni(cod)<sub>2</sub> Capsules for Use in Ni-Catalyzed Cross-Couplings on the Benchtop

## Introduction of the Researcher



## Garg Research Group

Natural Product Synthesis & Reaction Discovery

Professor **Neil K. Garg, Ph.D.**  
UCLA Chemistry and Biochemistry



## Research Description

Prof. Garg's lab is interested in the development of catalytic methodologies to activate amide C–N bonds for subsequent manipulation. Toward this end, they have developed a number of nickel-catalyzed amide activation reactions to access esters, other amides, and ketones from this traditionally inert functional group. The general use of these methodologies has been fettered by the requirement of glovebox handling of the synthetically important, air sensitive Ni(cod)<sub>2</sub> precatalyst. As a means to eliminate glovebox requirements for use of this chemistry, Garg and Co. developed paraffin–Ni(cod)<sub>2</sub> capsules that enable the transformations to be performed entirely on the benchtop. The capsules have been demonstrated to work in the aforementioned amide activation reactions as well as a number of other Ni(cod)<sub>2</sub>-mediated cross-coupling reactions. These capsules are expected to broaden the use of nickel catalysis in both academia and industry.

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