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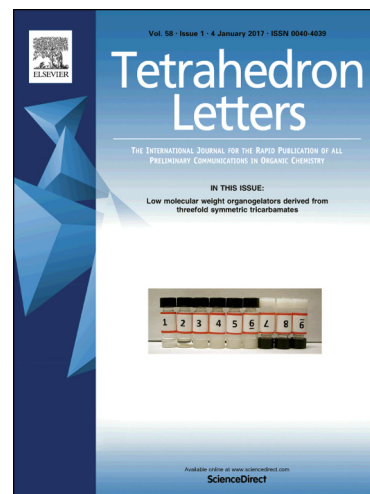
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Nickel-Catalyzed Decarboxylative Coupling of an Alkynyl Carboxylic Acid with Aryl Iodides

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ABSTRACT

A decarboxylative coupling reaction with an alkynyl carboxylic acid and aryl iodides in the presence of a nickel catalyst was developed. When the reaction was conducted with NiCl₂ (10 mol%), Xantphos (15 mol%), Mn (1.0 equiv), and Cs₂CO₃ (1.5 equiv), the desired diaryl alkynes were formed in moderated to good yields. Furthermore, this method does not produce the diyne, which is formed in the homocoupling of alkynyl carboxylic acids.

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Introduction

Transition metal-catalyzed cross-coupling reactions are among the most important and widely used tools in organic synthesis for the formation of carbon-carbon or carbon-heteroatom bonds.¹ A variety of named reactions, including the Heck,² Kumada,³ Negishi,⁴ Stille,⁵ Suzuki,⁶ Sonogashira,⁷ Hiyama,⁸ and Buchwald-Hartwig⁹ reactions, have been developed and improved over the last four decades. As a coupling substrate, aryl halides are the most commonly employed because they are readily available. Generally, aryl iodides are much more reactive than aryl bromides and chlorides.¹⁰

Sonogashira first showed that aryl halides are capable of coupling with terminal alkynes in the presence of palladium, copper, and a base to provide aryl alkynes. Since his first report of this reaction in 1979, it has become the most common method for sp and sp² carbon bond formation, and is now known as the Sonogashira reaction.¹¹ It is very often employed as a useful tool for the synthesis of conjugated polymers in the electroactive material field.¹² Palladium is used as the catalyst for this reaction far more often than other transition metals such as copper and nickel.¹³

As an alternative method, the decarboxylative coupling of alkynyl carboxylic acids has received much attention since we first reported the palladium-catalyzed coupling of alkynyl carboxylic acids and aryl halides in 2008.¹⁴ Alkynyl carboxylic acids such as propiolic acid have been widely employed as

alkyne surrogates, and exhibit good activity in this reaction with a variety of coupling partners.¹⁵ Furthermore, they can be readily prepared in one step without the need for column chromatography for purification.¹⁶

We have previously reported a number of decarboxylative coupling reactions. For example, synthetic strategies for diaryl alkynes, diynes, diketones, allenes, alkynyl ketones, and alkynyl amides have been developed.¹⁷ In addition, several multicomponent reactions have been developed.¹⁸ These reactions are catalyzed by palladium or copper in most cases, with only a few rare examples of nickel catalysts being reported, even though it is more abundant and less expensive than palladium. The first example of a nickel-catalyzed decarboxylative coupling reaction was that with an allyl acetate, as shown in Figure 1a.¹⁹ Recently, we reported nickel-catalyzed decarboxylative coupling reactions with aryl siloxanes or aryl boronic acids (Figure 1b).²⁰ These successes prompted us to develop a nickel-catalyzed decarboxylative coupling reaction with aryl halides, which has long been regarded as a challenge.

Herein, we report for the first time the nickel-catalyzed decarboxylative coupling of an alkynyl carboxylic acid and aryl iodides (Figure 1c).

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