“Click” Reaction – CuAAC with Benzyl Azide & Phenyl Acetylene

CHEM HELP *ASAP*

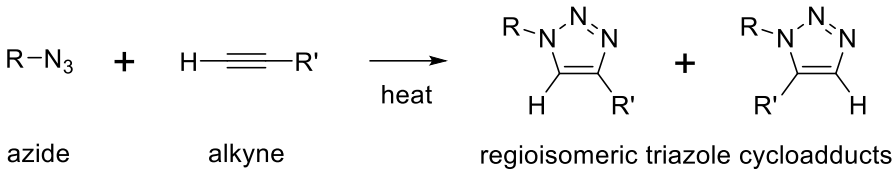
experiment video: https://youtu.be/IdZxOjCT8sg

Purpose

The purpose of this experiment is to synthesize a 1,2,3-triazole ring through a copper-catalyzed azide-alkyne cycloaddition. The crude product will be isolated by simple filtration and is pure enough to be characterized in its crude form.

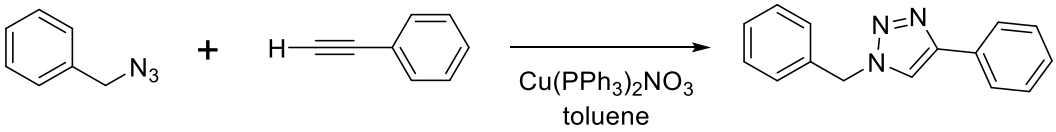
Background

While some dipolar cycloadditions have been known for over 100 years, the reactions received renewed interest in the 1960s with research from Rolf Huisgen. One example of a 1,3-dipolar cycloaddition is the azide-alkyne cycloaddition. The reaction typically requires heating and generates a mixture of regioisomeric 1,2,3-triazole products (Scheme 1).



Scheme 1. Thermal azide-alkyne 1,3-dipolar cycloaddition

In 1998, two research groups, one led by Meldal in Denmark and another led by Sharpless and Fokin, discovered a copper-catalyzed azide-alkyne cycloaddition (CuAAC) (Scheme 2). The CuAAC often occurs at room temperature, gives a single regioisomer, and gives higher yields. Sharpless’ work popularized the term “click” reaction to represent the simplicity and efficiency of the reaction.



Scheme 2. Today’s reaction – aromatic bromination with NBS

Procedure – 1-benzyl-4-phenyl-1,2,3-triazole

In a 20 mL scintillation vial charged with a stir bar mix a 1.0 M toluene solution of benzyl azide (1.0 mL, 1.0 mmol), a 1.0 M toluene solution of phenyl acetylene (1.0 mL, 1.0 mmol), and 5-10 mg of $Cu(PPh_3)_2NO_3$ (MW 650 g/mol, ~1 mol%). The mixture was gently heated to 40-50 °C and stirred for 1 h. Cool the mixture in an ice bath. Filter the solids through a Buchner funnel with a 50-mL side-arm flask. Seat the filter paper with toluene, and rinse the collected solid with a minimal amount of cold toluene. Spread the collected solid on a tared watchglass, and let the solid air dry. Once the product is dry, determine the mass of the product. Determine your percent yield. Determine the melting point (melting range – both upper and lower limits) of the product. Take a TLC of the product using 50% ethyl acetate/50% hexane as the mobile phase. Once you finish your TLC, dispose your mobile phase in a waste jug. Interpret the provided NMR spectrum. Record all your observations in your notebook.