# The synthesis of 1,3-disubstituted pyrazoles with quantitative deuterium labeling by [3+2]-cycloaddition of in situ generated nitrile imines and acetylene

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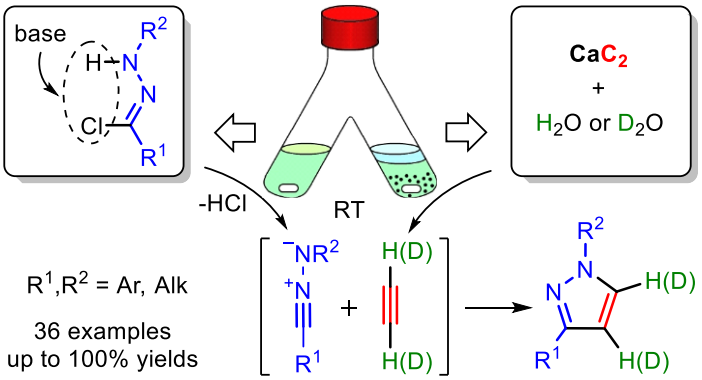
A novel synthetic methodology for preparation of 1,3-disubstituted pyrazoles from *in situ* generated nitrile imines and acetylene was developed. The reactions were performed in a simple two-chamber reactor. One part of the reactor was loaded with hydrazonoyl chloride precursors of active nitrile imine species and a base. The other part was used to generate acetylene from $CaC_2$ and water.

This reaction, undoubtedly, can be performed in one vessel. But, the nitrile imines are highly reactive and water-sensitive. Because of this, the yields of pyrazoles in the tube or flask are low. Partitioning of the reactants improves the yields of desired pyrazoles up to 100% and simplifies their isolation to an elementary procedure of solvent evaporation.[1] The approach requires no complex equipment and utilizes inexpensive, safe and easy to handle calcium carbide as a starting material. A model deuterium incorporation is carried out according to the developed methodology, producing a series of novel 4,5-dideuteropyrazoles with excellent deuterium enrichment.




1. V.V. Voronin, M.S. Ledovskaya, E.G. Gordeev, K.S. Rodygin, V.P. Ananikov, *J. Org. Chem.*, **2018**, 83, 3819–3828

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