**Mechanistic Insights through *In Situ* Reaction Monitoring in Photocatalytic Synthesis**

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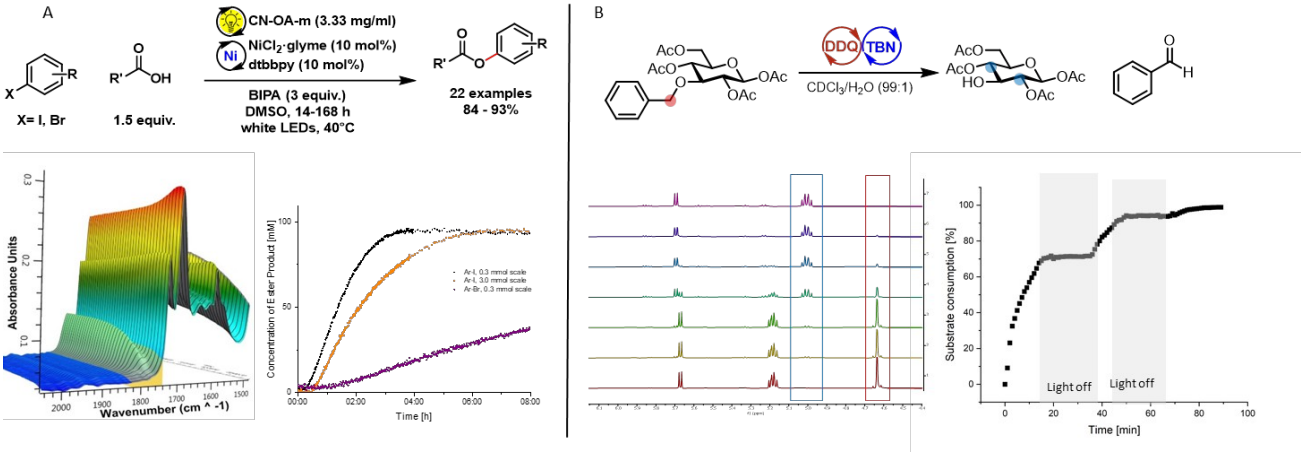
*In situ* reaction monitoring is a powerful tool to track reactions in real time under synthetically relevant conditions.[1] Such investigations unveil intermediates and allow to determine rate dependencies to understand reaction kinetics, which is essential to elucidate the underlying mechanism.

In this talk, I will present how *in situ* reaction monitoring was used to provide mechanistic insights in two photocatalytic reactions. First, a comprehensive kinetic examination of a dual nickel/photocatalytic C–O arylation using a homogeneous photocatalyst or a heterogeneous photocatalyst will be discussed. [2-3] We used *in situ* infrared spectroscopy for in-depth kinetic studies of both catalytic systems have been carried out by applying variable time normalization analysis (VTNA). The studies revealed arguments against the current mechanistic hypothesis, which states that the photocatalyst is only involved to trigger reductive elimination.

Second, I will present our efforts to track a newly developed photo oxidative cleavage of benzyl ether protecting groups using a LED-NMR setup.[4] These investigations support the underlying mechanistic hypothesis and supports the notion that the reaction ceases upon light removal.




**Scheme 1**. In situ reaction monitoring of a dual nickel/photocatalytic C–O arylation through infrared spectroscopy (**A**) and a photo oxidative debenzylation using a LED-NMR setup (**B**).

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2. Welin, E. R.; Le, C.; Arias-Rotondo, D. M.; McCusker, J. K.; MacMillan, D. W. C., *Sci* **2017,** *355*, 380-385.
3. Malik, J. A.; Madani, A.; Pieber, B.; Seeberger, P. H., *J. Am. Chem. Soc.* **2020,** *142*, 11042-11049.
4. Cavedon, C.; Sletten, E. T.; Madani, A.; Niemeyer, O.; Seeberger, P. H.; Pieber, B., *Org Lett* **2021,** *23*, 514-518.