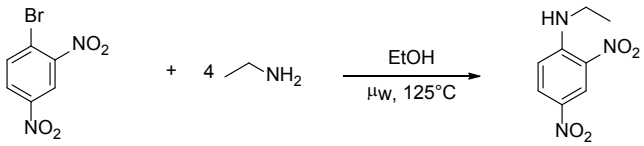
Method Visualization with the Discover 2.0 Camera

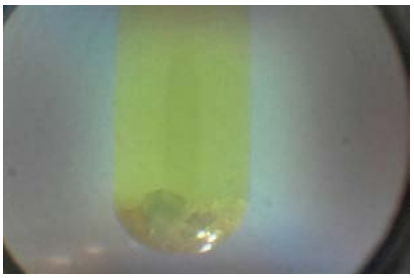
Introduction

The benefits of using microwave technology, including access to elevated temperatures and pressures, have allowed microwave assisted synthesis to become commonplace. However, with the added safety benefits of dedicated synthetic microwaves came a closed cavity, allowing monitoring of temperature and pressure but preventing simple visual observation. This inability to observe the reaction has prevented such vital observations as color changes, solubility, stirring and gas evolution. With the built in camera for the Discover 2.0, the reaction can be observed (and recorded) with ease.

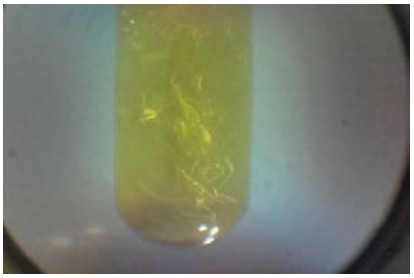
Synthesis of 2,4-dinitro-N-ethylaniline by Nucleophilic Aromatic Substitution

The synthesis of 2,4-dinitro-N-ethylaniline by nucleophilic substitution with ethyl amine proceeds in under 5 minutes using microwave irradiation.[1] While the crystalline 1-bromo-2,4-dinitrobenzene is insoluble in ethanol at room temperature, dissolution occurs during heating, resulting in a homogeneous reaction. Following completion of the reaction, the product, 2,4-dinitro-N-ethylaniline crystallizes out of solution during cooling.

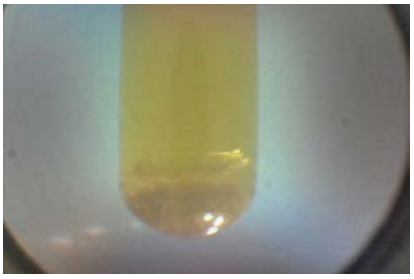




T = 25°C (before reaction)



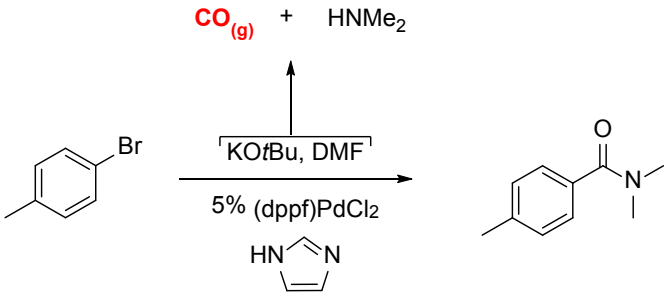
T = 125°C (during reaction)

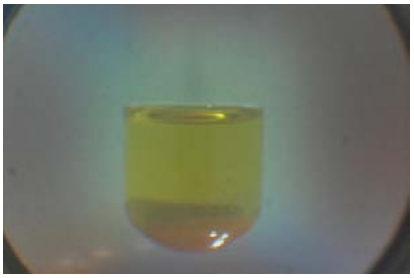


T = 50°C (during cooling)

Palladium Catalyzed Aminocarbonylation of Aryl Halides

Carbonylation reactions – those which incorporate CO into a molecule – often pose experimental challenges due to the difficulties which accompany the use of carbon monoxide gas at elevated pressure. In situ generation of CO gas from reaction of potassium tert-butoxide and dimethylformamide provides a safe and convenient route for carbonylation reactions. The microwave assisted, palladium catalyzed aminocarbonylation of aryl halides with in situ generated carbon monoxide has been reported.[2] With the Discover 2.0 camera, both color change and continued generation of carbon monoxide gas can be observed throughout the reaction, assisting with rapid reaction optimization.





[1] Leadbeater, N. E.; McGowan, C. C. *Clean, Fast Organic Chemistry*; CEM Publishing, 2006.

[2] Wan, Y.; Alterman, M.; Larhed, M.; Hallberg, A. *J. Org. Chem*. **2002**, 67, 6232.612–618.

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