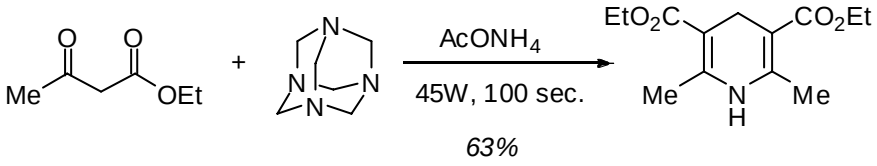
Method Translation with the Camera Option

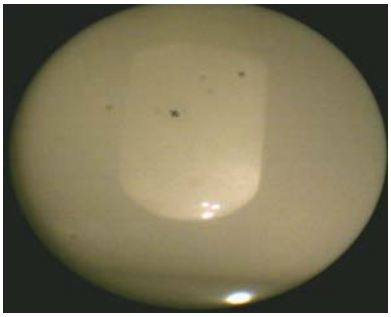
A full equivalent of the Hantzsch ester is necessary for the tandem knoevenagel/hydrogenation chemistry. The Hantzsch 1,4-dihydropyridine is commercially available, but it is relatively expensive and difficult to obtain in large quantities. There are several known methods for the preparation of the Hantzsch esters including a microwave procedure described by Barbry. This reaction involves the condensation of ethyl acetoacetate with ammonia and formaldehyde that is generated in situ by the hexamethylenetetramine. Barbry performed this procedure in a Synthewave 402 Prolabo.

Hantzsch Ester Synthesis

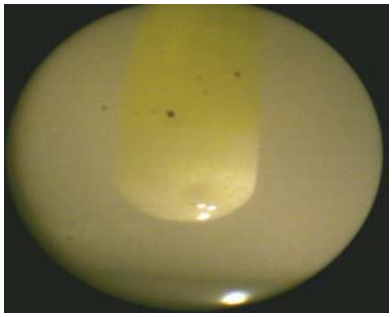


Torchy, S.; Cordonnier, G.; Barbry, D.; Vanden Eynde, J. J. *Molecules* **2002**, *7*, 528-533.

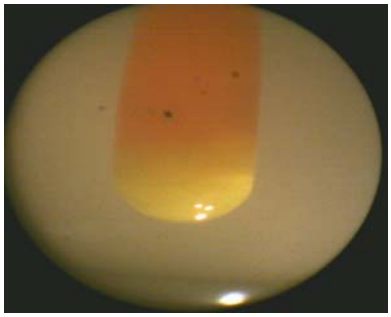
When we performed this reaction in the CEM Applications Laboratory, we were unsure how well the conditions would translate from the Synthewave to the CEM Discover. Therefore we ran this reaction with the camera option to track the progress of the reaction. At t = 0 seconds the reaction mixture is white. After 60 seconds the reaction turns a yellow color. If the reaction is stopped at this point, there is no product present. If the reaction is allowed to proceed for just another 10 seconds, the reaction mixture turns orange and the product is now present. If the reaction is allowed to proceed longer than the 70 seconds, decomposition of the Hantzsch ester begins to occur, and the pressure in the reaction begins to increase rapidly due to the generation of ammonia gas and formaldehyde. This reaction demonstrates the utility of the camera to track the progress of the reaction, and allow the maximum product yield.



t = 0 sec.



t = 60 sec



t = 70 sec.