Supplementary Material (ESI) for Green Chemistry


# Rate Enhancement of the Morita-Baylis-Hillman Reaction through Mechanochemistry.

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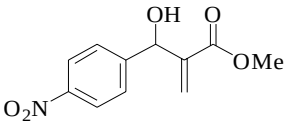
Supporting Information

## General Information:

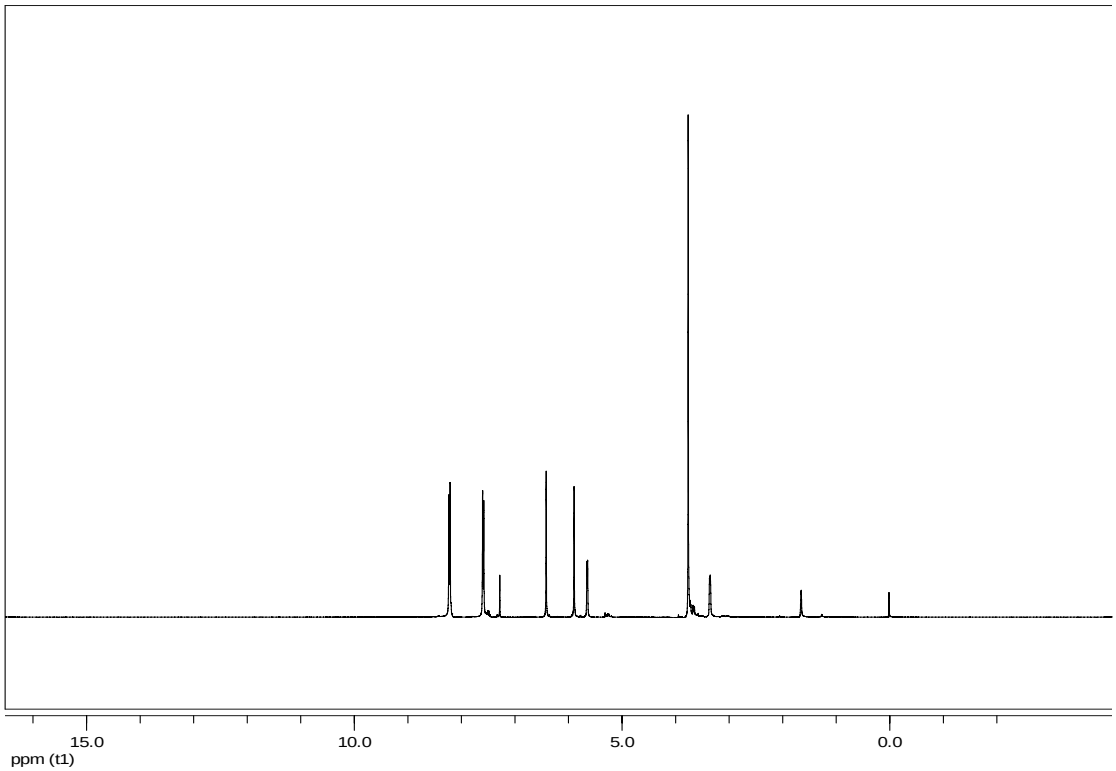
$^1$H NMR spectra were recorded on a Bruker Avance 400 spectrometer. Dueterated NMR solvents were obtained from Cambridge Isotope Laboratories, Inc., Andover MA, and used without further purification. Methyl acrylate, *p*-nitrobenzaldehyde, *p*-bromobenzaldehyde, *p*-chlorobenzaldehyde, anisaldehyde, benzaldehyde, diazabicyclo[2.2.2]octane, quinuclidine, tetramethyl guanidine, quinuclidinol, 4-dimethylaminopyridine, 1,5-diazabicyclo[4.3.0.]non-5-ene, hexamethylenetetramine, triphenylphosphine, imidazole and sodium sulfide were purchased from Acros Organics and used without further purification. 2,8,9 trimethyl-2,5,8,9-tetraaza-1phospha-bicyclo[3.3.3]undecane was purchased from Aldrich Chemical Company and used without further purification. Ball milling was carried out in an 8000M SpexCertiprep Mixer/Mill. Separation was done using an Isco Combiflash Companion column system.

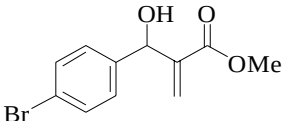
**Typical Procedure:** methyl acrylate (0.43g, 5 mMol), p-nitrobenzaldehyde (0.76g, 5mMol) and DABCO (0.11g, 1mMol) were added to a custom-made 2.0 inch by 0.5 inch screw capped stainless steel vial along with a 0.125 inch stainless steel ball bearing. The vial was placed in an 8000M Spex Certiprep mixer/mill and the contents were ball milled for 0.5 h. The resulting mixture was dissolved in $CH_2Cl_2$ (10mL) and extracted with 10%HCl (3 x 10mL). The combined organic layers were dried over anhydrous $MgSO_4$ and the solvent was evaporated under reduced pressure. The resulting yellow mixture was purified by column chromatography over silica gel (1:1 $CH_2Cl_2$/ethyl acetate) to afford 2-Hydroxy-(4-nitro-phenyl)-methyl]-acrylic acid methyl ester in 98% yield.

$^1$H NMR Data:

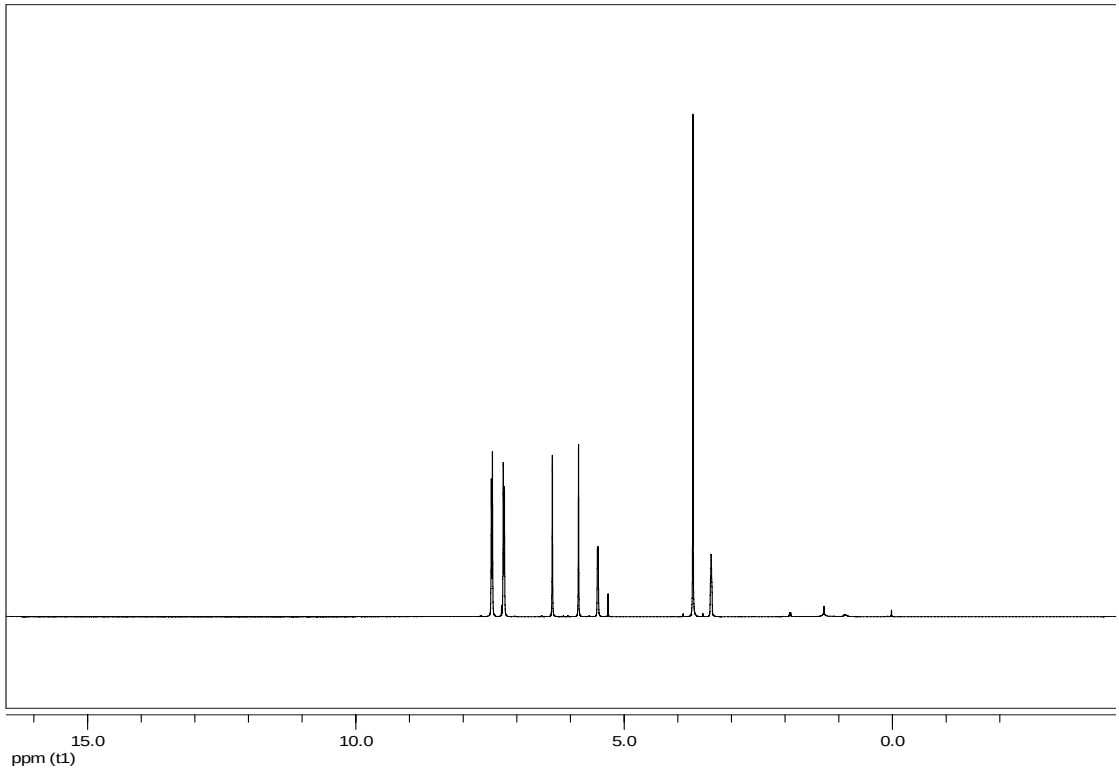


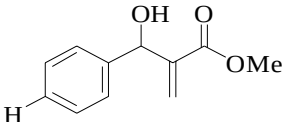

(Table 1, entry 1) The data for his compound is in agreement with the literature: Aggarwal, V. K.; Emme, I.; Fulford, S. Y. *J.Org.Chem* **2003**, *68*, 692-700.



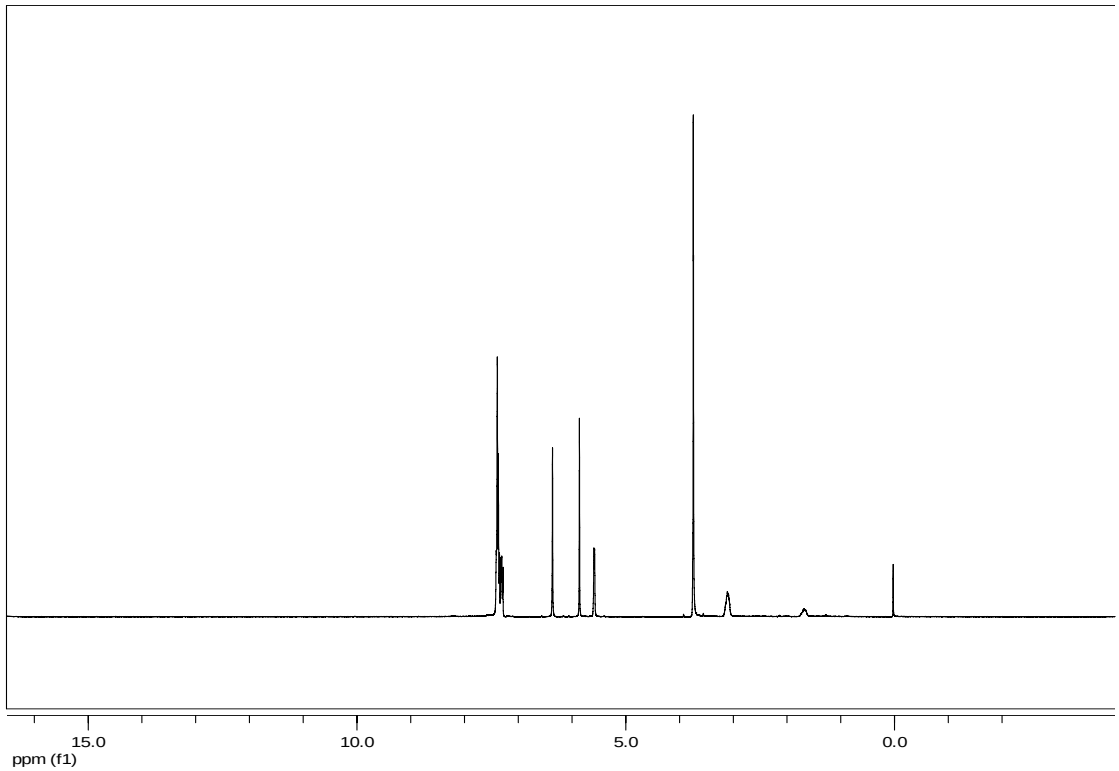


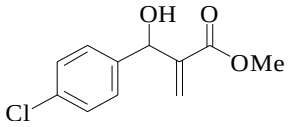

(Table 1, entry 2)



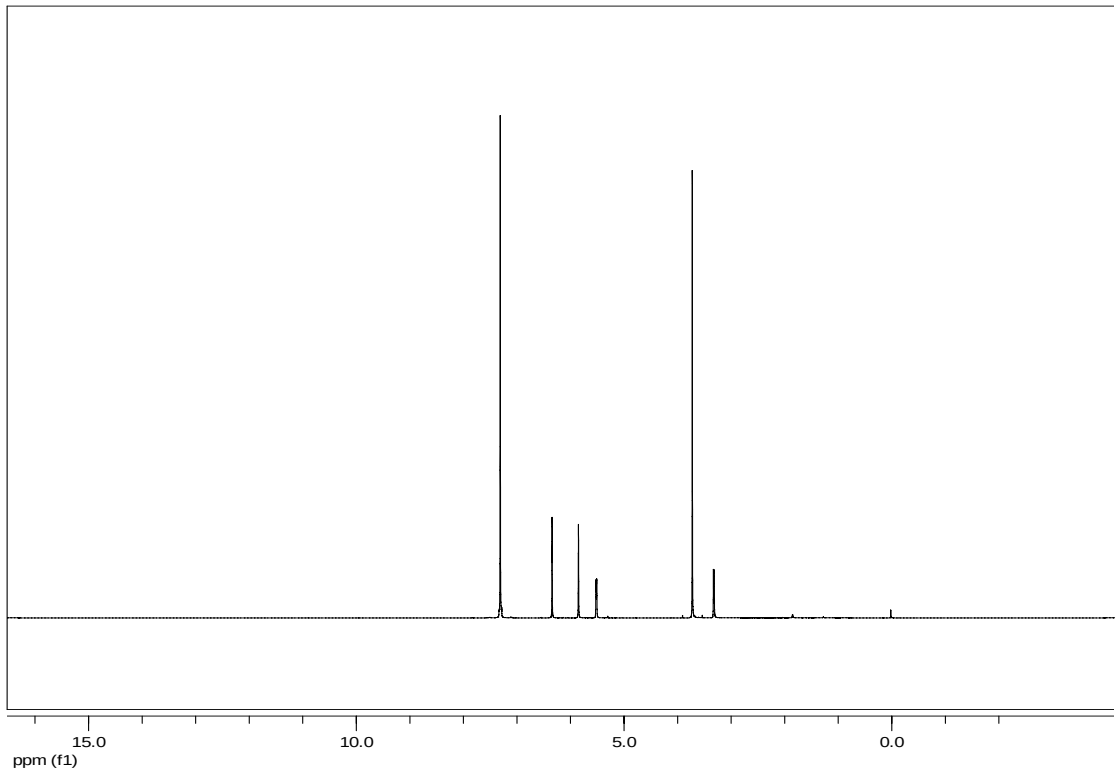


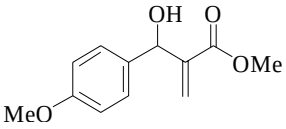

(Table 1, entry 3) The data for his compound is in agreement with the literature: Aggarwal, V. K.; Emme, I.; Fulford, S. Y. *J.Org.Chem* **2003**, *68*, 692-700.



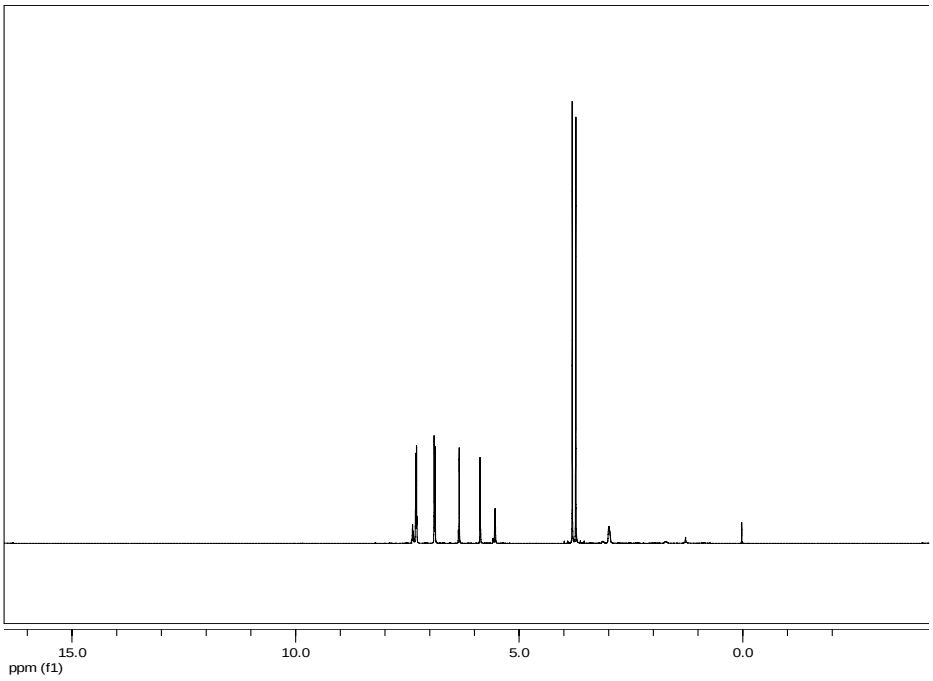



(Table 1, entry 4) The data for his compound is in agreement with the literature: Aggarwal, V. K.; Emme, I.; Fulford, S. Y. *J.Org.Chem* **2003**, *68*, 692-700.






(Table 1, entry 5) The data for his compound is in agreement with the literature: Aggarwal, V. K.; Emme, I.; Fulford, S. Y. *J.Org.Chem* **2003**, *68*, 692-700.



## Design of Reaction Vials