Electronic Supplementary Information

Rh(II) Catalysed Intramolecular C-H Insertion of Diazo Substrates in Water: A Simple and Efficient Approach for Catalyst Reuse

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General Procedure for the Rhodium(II)-Catalysed Cyclization of α-diazo- α-phosphono substrates in water.

To a solution of dirhodium(II) tetraacetate (1.0 mol %) in water (1.5 mL) was added the appropriate diazo compound (0.154 mmol). The reaction mixture was heated at 80 ºC until disappearance of the substrate (determined by TLC). The mixture was concentrated under reduced pressure and the residue was purified by flash chromatography (basic alumina with AcOEt/Hexane), yielding the desired compounds.

Procedure for Rhodium(II) Recycling

To a solution of dirhodium(II) tetraacetate (1.0 mol %) in water (1.5 mL) was added α-diazo-α-(diethoxyphosphoryl)-*N*,*N*-(diisopropyl)acetamide **1a** (0.154 mmol). The reaction mixture was heated at 80 ºC for 24h, the product was extracted from the reaction medium with Et_2O (10×1.5mL) and more substrate was added (0.154 mmol) to the system.

Figure 1: Dirhodium(II) tetraacetate aqueous solution

Figure 2: Dirhodium(II) tetraacetate aqueous solution upon 1.5 ml ether extraction

Figure 3: ^{1}H NMR crude spectra of cyclization reaction on substrate **1a**.

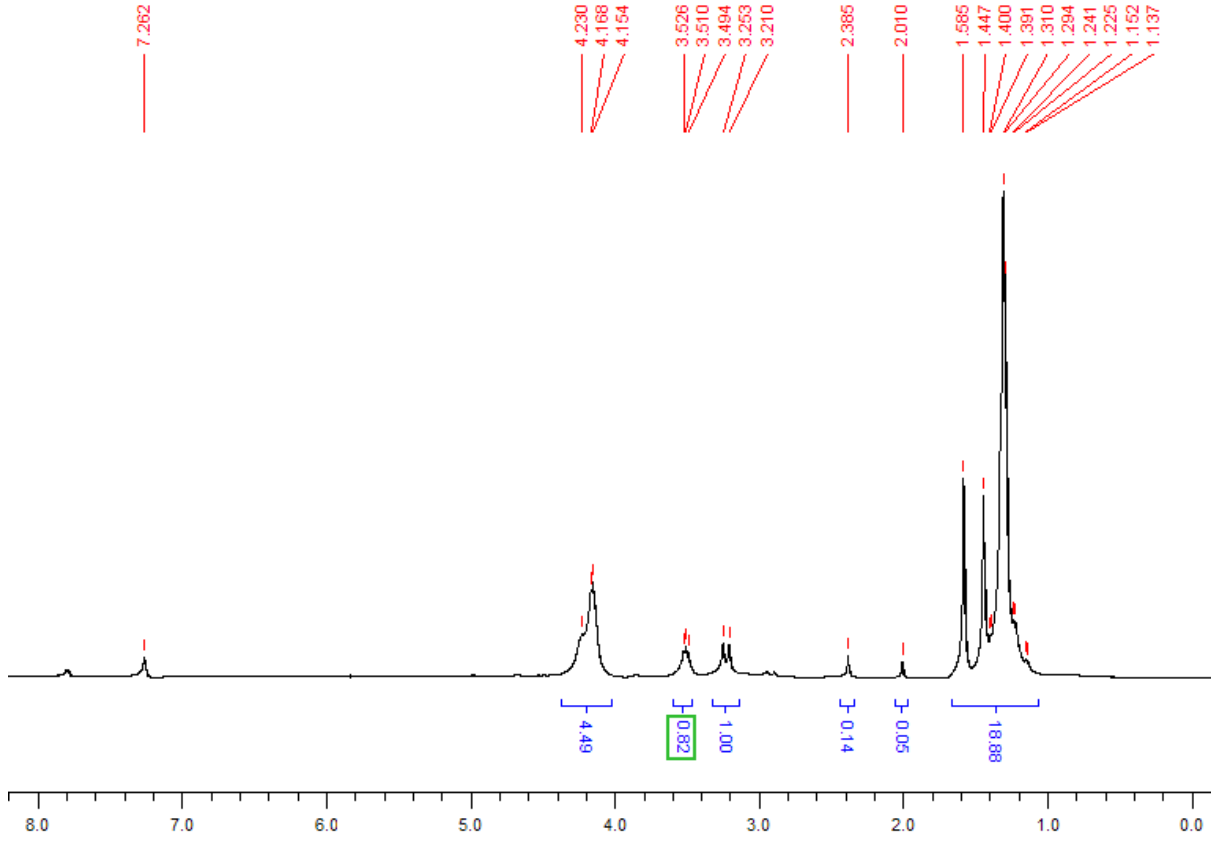

Figure 4: ^{31}P NMR spectra of **1a**.

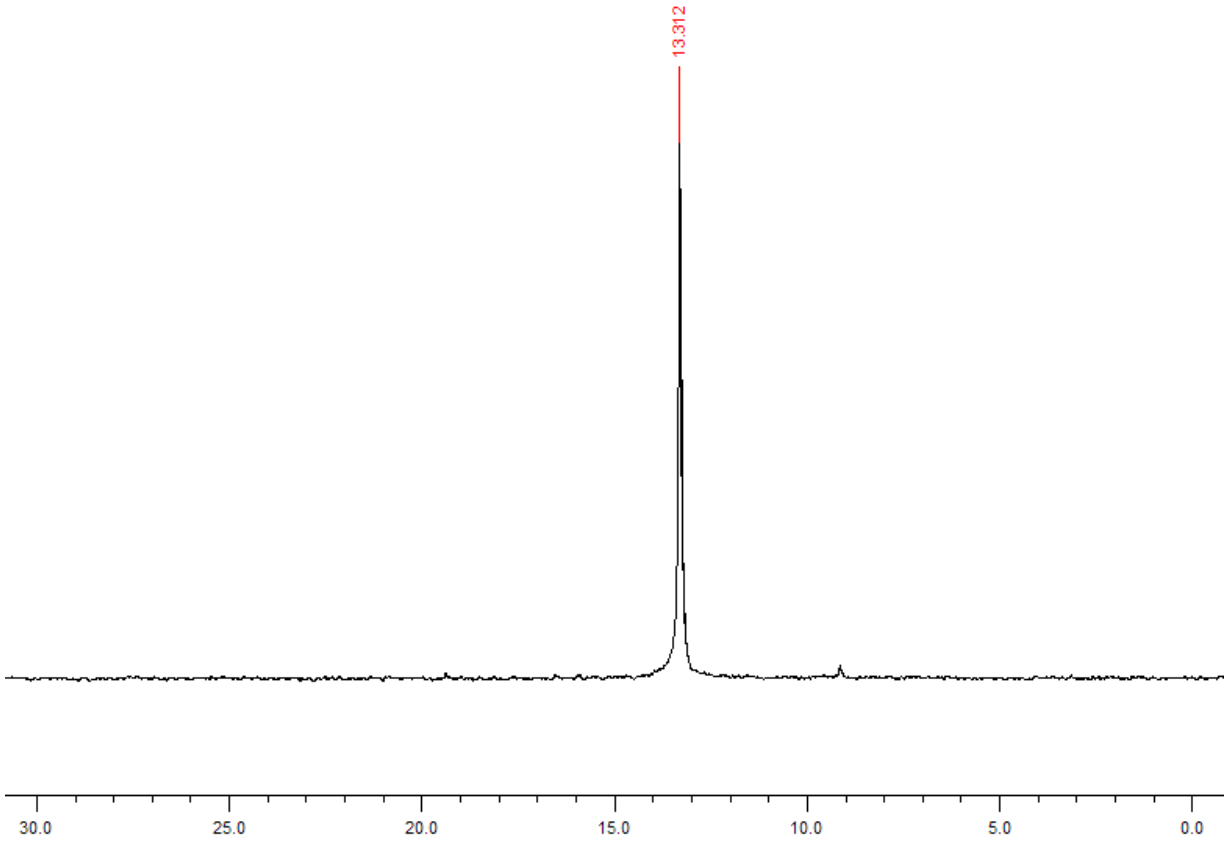

Figure 5: ^{31}P NMR crude spectra of cyclization reaction on substrate **1a** .

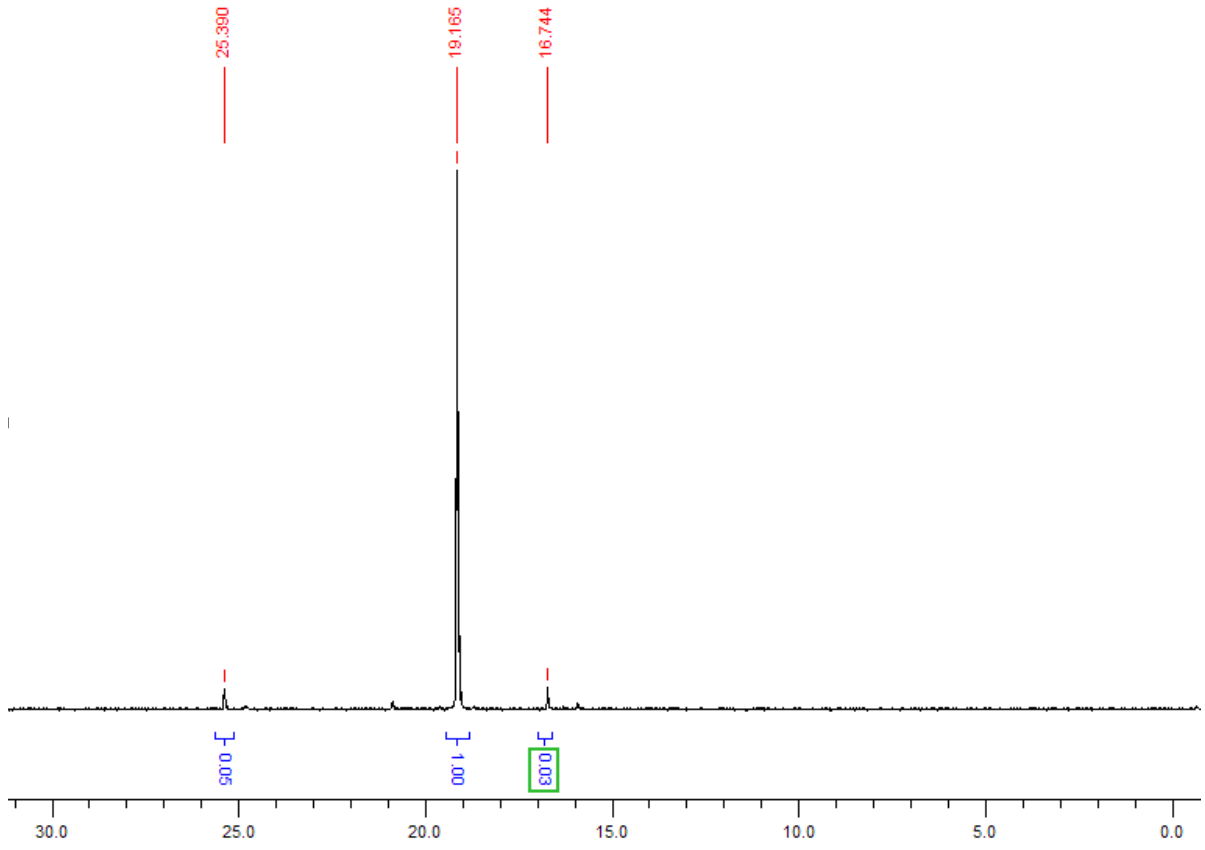

Figure 6: ^{1}H NMR crude spectra of cyclization reaction on substrate **1c**.

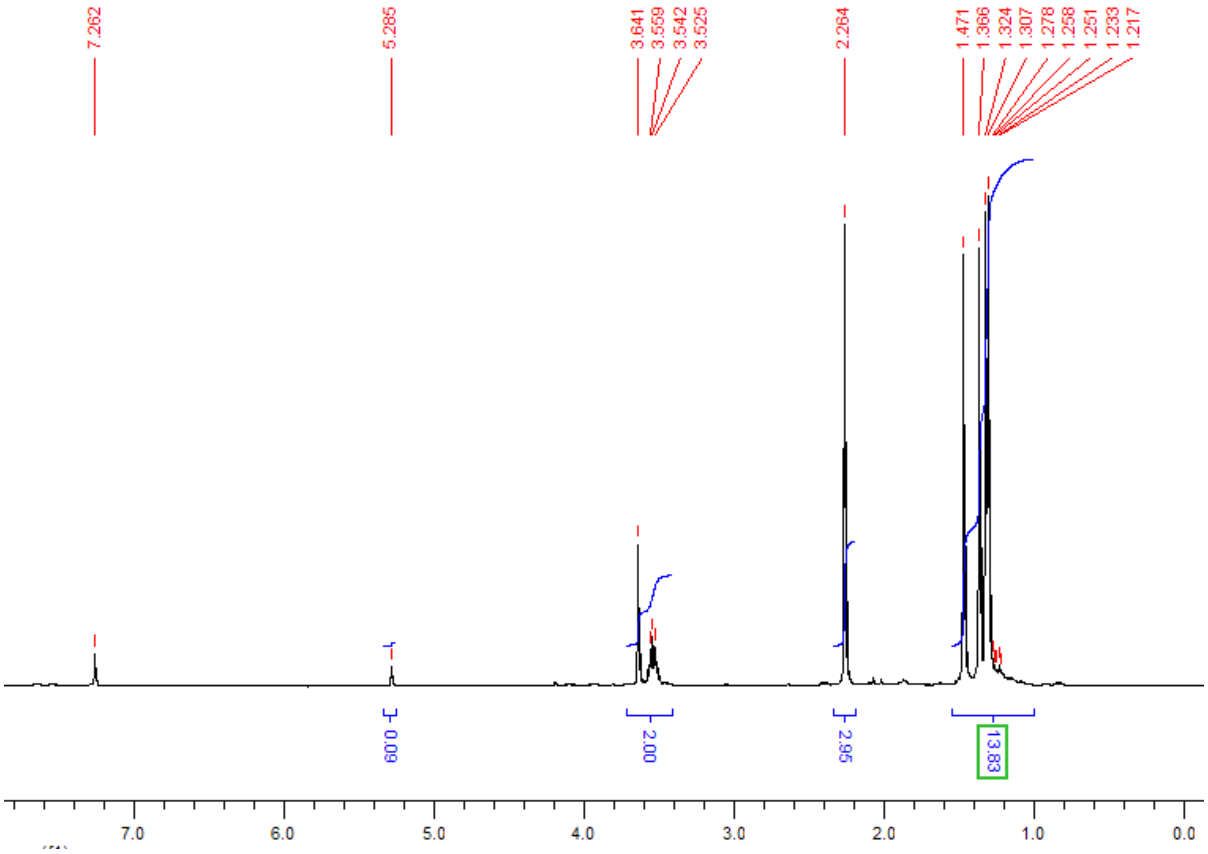

Figure 7: ^{1}H NMR spectra of isolated -β-lactam **2c**.