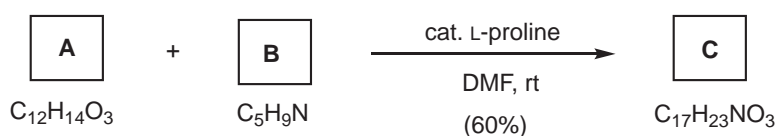


Morofuji, T.*; Kinoshita, H.; Kano, N.* *Chem. Commun.* **2019**, 55, 8575-8578

Q: Specify unsaturation number and structure of **A**, **B** and **C**. Also propose reasonable mechanisms of the following reactions.

To a solution of 1-(triphenylphosphoranylidene)-2-propanone in dichloromethane was added a solution of 3,4-dimethoxybenzaldehyde in dichloromethane at 0°C and the mixture was left to stir overnight at room temperature. After consumption of the aldehyde (TLC), the solvent was removed in vacuo and the crude product was purified using flash column chromatography on silica gel (hexane/EtOAc, 9:1) to give (*E*)-3-(4-dimethoxyphenyl)but-3-en-2-one (**A**).

To a solution of *N*-chlorosuccinimide in diethyl ether was added a solution of distilled piperidine in diethyl ether over 30 min at room temperature. The reaction mixture was stirred for 3 h at ambient temperature after which it was filtered through a pad of Celite[®] and the residue was washed with ether. The combined filtrates were washed with water, dried over anhydrous Na_2SO_4 and concentrated without heating to give $\text{C}_5\text{H}_{10}\text{NCl}$. An ethereal solution of $\text{C}_5\text{H}_{10}\text{NCl}$ thus prepared was added dropwise to ethanolic KOH (prepared by heating 2.1 equiv. of solid KOH in ethanol to 85°C) at room temperature and the reaction was left to stir overnight. The white precipitate of KCl formed was separated by filtration through a pad of Celite[®]. The filtrate was concentrated to give **B**.



Data for **C**.

IR (neat): 1717 cm^{-1}

$^1\text{H NMR}$ (300 MHz, CDCl_3): δ 6.92 (br s, 1H), 6.87-6.78 (br m, 2H), 3.90 (s, 3H), 3.87 (s, 3H), 3.21 (d, 1H, $J = 12.1, 3.3\text{ Hz}$), 2.84-2.62 (m, 2H), 2.57-2.21 (m, 4H), 1.80-1.18 (m, 7H)

$^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ 207.8, 149.3, 148.3, 135.2, 119.5, 111.1, 109.8, 70.0, 62.5, 56.0, 55.9, 52.8, 50.9, 48.7, 34.3, 25.8, 24.2.