Article

Supporting Information

Pd-Catalyzed Cyclocarbonylation of Allylic Alcohol under Benign Conditions with Ionic Liquid as Stabilizer

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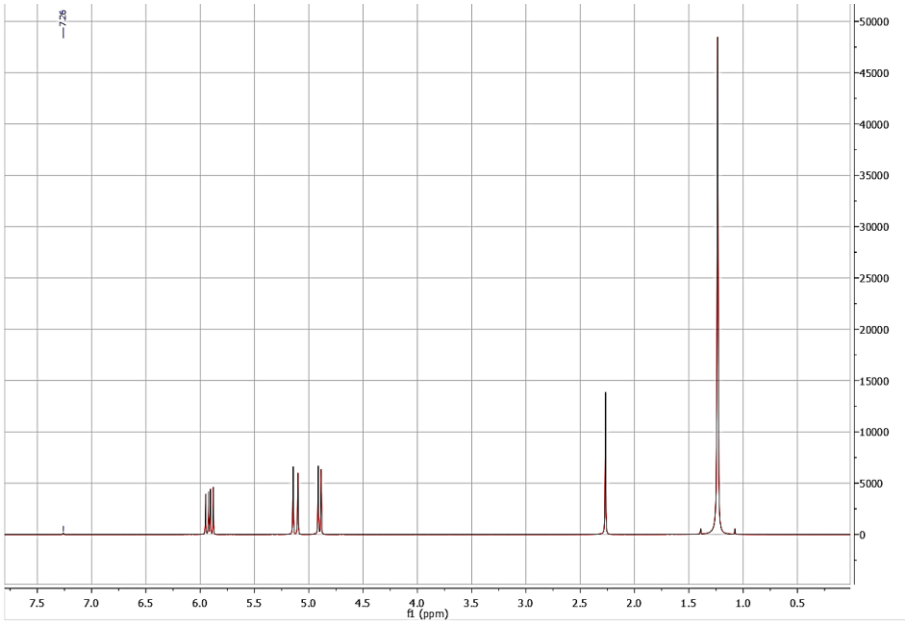
(**a**)



(**b**)

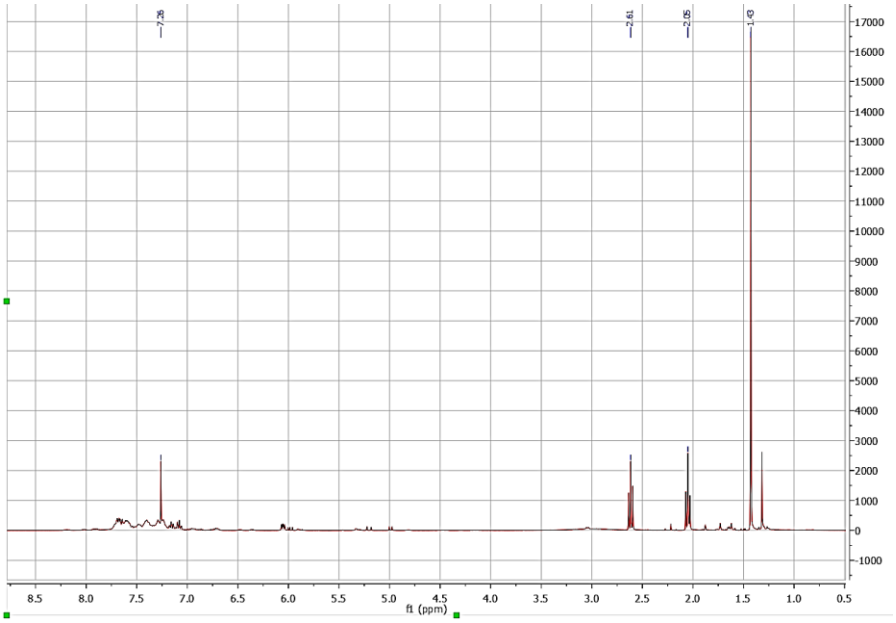


Figure S1. ^{1}H NMR spectra (400 MHz, $CDCl_3$) of (**a**) 2-methyl-3-buten-2-ol (δ/ppm: 1.2 (s, 2xCH_3, 6H), 2.3 (s, O*H*, 1H), 4.8 (d, *H*CH, 1H), 5.2 (d, HC*H*, 1H), 5.9 (dd, C*H*, 1H) and (**b**) reaction mixture after cyclocarbonylation with the product 4,4-dimethyl-γ-butyro-lactone (δ/ppm: 1.43 (s, CH_3, 6H), 2.05 (t, CH_2, 2H), 2.61 (t, COCH_2, 2H) and by-product(s) (δ/ppm: 1.63 (d), 3.06 (m), 5.28 (q)). Reaction conditions: 0.1 M 2-methyl-3-buten-2-ol, 4 mol% $Pd(OAc)_2$, 16 mol% DPEPhos, 5 ml DCM, 95 °C, 28 bar (CO/H_2: 23/5), 18 h.

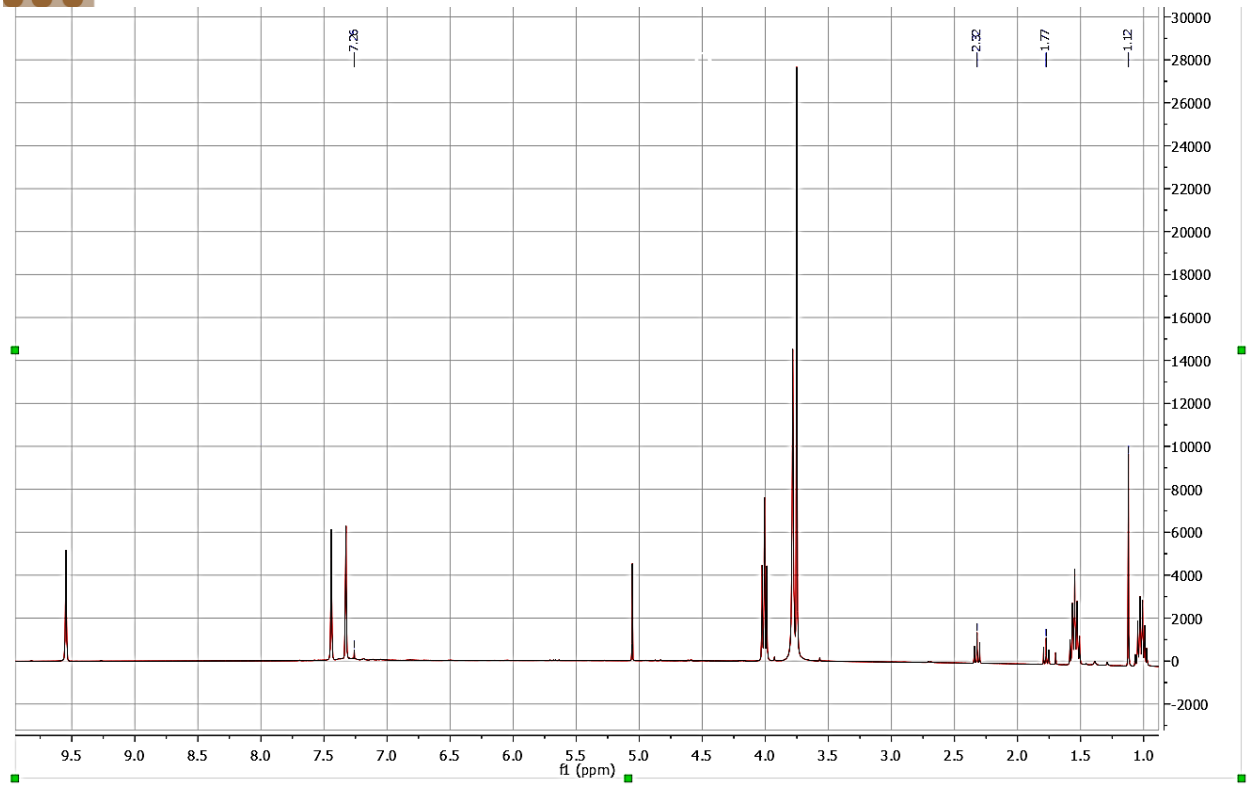
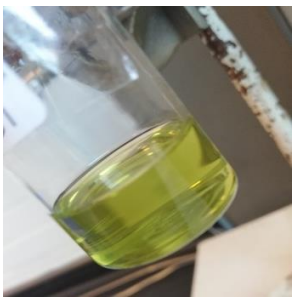


Figure S2. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the reaction mixture after cyclocarbonylation of 2-methyl-3-buten-2-ol with presence of the IL [BMIM]Cl (δ/ppm: 1.05 (m, N-$CH_2CH_2CH_2CH_3$, 5H), 1.55 (q, N-$CH_2CH_2CH_2CH_3$, 2H), 4.0 (t, N-$CH_2CH_2CH_2CH_3$, 2H), 5.1 (s, N-CH_3, 3H), 7.3 (s, N-CH_2CH_2-N, 2H), 7.45 (s, N-CH_2CH_2-N, 2H), 9.6 (s, N-CH-N, 1H) and the product 4,4-dimethyl-γ-butyro-lactone (δ/ppm: 1.15 (s, CH_3, 6H), 1.75 (t, CH_2, 2H), 2.3 (t, $COCH_2$, 2H). Reaction conditions: 0.1 M 2-methyl-3-buten-2-ol, 1.0 g [BMIM]Cl, 4 mol% $Pd(OAc)_2$, 16 mol% DPEPhos, 5 ml DCM, 95 °C, 28 bar (CO/H_2: 23/5), 18 h.

(**a**)

(**b**)



(**c**)

(**d**)

Figure S3. Reaction mixture after (**a**) first reaction run (no Pd-black), (**b**) second reaction run (no Pd-black), (**c**) third reaction run (no Pd-black) and (**d**) fourth reaction run (Pd-black) during recycling of the IL Pd-DPEPhos catalyst system in the cyclocarbonylation of 2-methyl-3-buten-2-ol. Reaction conditions: 0.1 M 2-methyl-3-buten-2-ol, 4 mol% $Pd(OAc)_2$, 16 mol% DPEPhos, 1.0 g [BMIM]Cl, 5 ml DCM, 100 °C, 28 bar ($CO/H_2/N_2$: 20/5/3), 18 h.

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