Supporting Information for:

Synthesis of Cyclic Carbonates from CO_2 and Epoxides Using Ionic Liquids and Related Catalysts Including Choline Chloride-Metal Halide Mixtures

Qing He, Jeremy W. O'Brien, Kayla A. Kitselman, Lindsay E. Tompkins, Gregory C. T. Curtis and Francesca M. Kerton

Department of Chemistry, Memorial University of Newfoundland, St. John's, Newfoundland, Canada A1B 3X7

Experimental

General

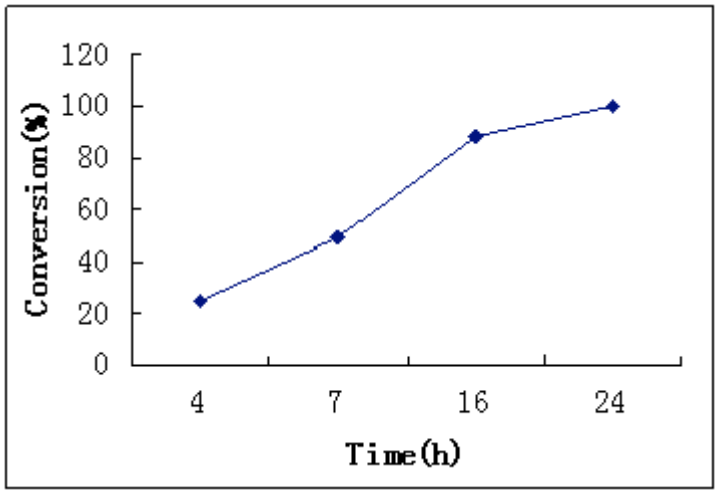
Choline chloride (ChCl) (98+%), chromium (III) chloride hexahydrate (98%), cobalt (II) chloride hexahydrate (98%) and nickel (II) chloride hexahydrate (98%) were purchased from Alfa-Aesar. Manganese (II) chloride tetrahydrate (98+%), anhydrous iron (III) chloride (98%), and copper (II) chloride dihydrate (99+%) were purchased from Strem Chemicals. Styrene oxide (97%) was purchased from Sigma-Aldrich and used as received. Acetonitrile (HPLC grade) was purchased from Caledon Laboratories. The ChCl-metal halide mixtures were used within 6 h of their preparation.

^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were recorded on a Bruker Avance 300 MHz spectrometer at 25 °C and were referenced internally using the residual proton and ^{13}C resonances of the solvent. GC-MS spectra were obtained using an Agilent 7890A GC System equipped with an autoinjector, coupled to a 5975C MSD (EI). The column used was a DB-5 capillary column.

Typical catalytic procedure

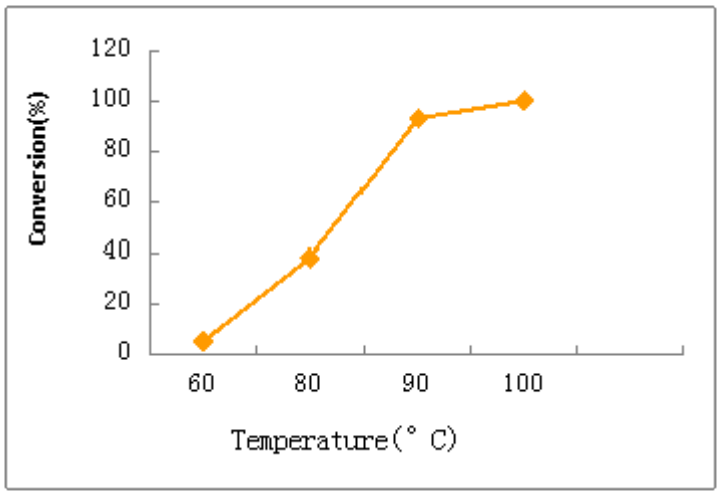
The reactions were carried out in a 300 mL or 100 mL steel Parr reactor equipped with a stirring impeller/overhead mechanical stirrer. In a typical reaction set-up, the catalyst (DES) and epoxide were loaded into the reactor. The reactor was then pressurized with the appropriate amount of CO_2 and heated to the desired temperature. After the required reaction time, the reactor was cooled in an ice bath and vented. Then NMR and GC-MS were carried to obtain conversion values. For yields, carbonates were separated from catalyst and unreacted epoxide by silica column chromatography. All reactions were performed in triplicate and the average of runs is presented in tables in the paper. For all reactions, no other products (e.g. diols) were observed and selectivity for carbonate was >99%.

Optimization of reaction time for styrene carbonate production using Co(II) DES



Pressure = 10 bar, 100 °C, SO:Co 130:1

Optimization of reaction temperature for styrene carbonate production using Co(II) DES



Pressure = 10 bar, 24 h, SO:Co 130:1