Supplementary Material (ESI) for Chemical Communications

Electronic Supplementary Information

Probing catalytic activity of halide salts by electrical conductivity in the coupling reaction of CO_2 and propylene oxide

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Experimental Section

The conductivity measurements were carried out with a DDS-307 conductivity instrument. Before and after the measurements, the instrument was calibrated with an aqueous KCl solution. Because of the limitation of the boiling point of PO (34 ^{o}C), the conductivity measurements were carried out at 25 ^{o}C.

All the coupling reactions were conducted in a 100 mL stainless-steel reactor equipped with a magnetic stirrer and automatic temperature control system. In a typical reaction, the reactor was charged with appropriate amount of Bu_4NBr (0.323 g, 1 mmol), PO (11.6 g, 0.2 mol), and water (1.2 g). After the reactor was heated to 105 ^{o}C, the reaction mixture was pressurized with CO_2 to the desired pressure (2 MPa) and stirred at ca. 1200 rpm for 1 h. The CO_2 was charged for several times during the experiment to maintain a constant pressure (2 MPa). Then the reactor was cooled in an ice-water bath, and the remaining CO_2 was released slowly. The resulting product mixture was defied by GC-MS and ^{1}H NMR. All the products were quantitatively analyzed by gas chromatography with acetophenone as internal standard.

The results of GC-MS (as shown in Fig. S1) and ^{1}H NMR indicated that the main product was propylene carbonate (PC), and the main byproduct was 1,2-propanediol. Signals in ^{1}H NMR ($CDCl_3$) spectrum: δ 4.86-4.83 (m, 1H), 4.57-4.53 (t, 1H), 4.04-4.00 (t, 1H), 1.49-1.47 (d, 3H), responded to PC; Slight signals in ^{1}H NMR ($CDCl_3$) spectrum: δ 3.89-3.86 (m, 1H), 3.62-3.58 (m, 1H), 3.39-3.35 (m, 1H), 3.30-3.27 (m, 1H), 1.14-1.13 (d, 3H), responded to 1, 2-propanediol. When acetic acid was used, the selectivity of PC was 94%. 1,2-Propanediol 2-acetate and 1,2-propanediol diacetate were main byproducts, and a slight amount of 1,2-propanediol was also observed. While other hydroxyl solvents were added, the selectivity of PC was ≥ 98%, and the main byproduct was 1,2-propanediol.

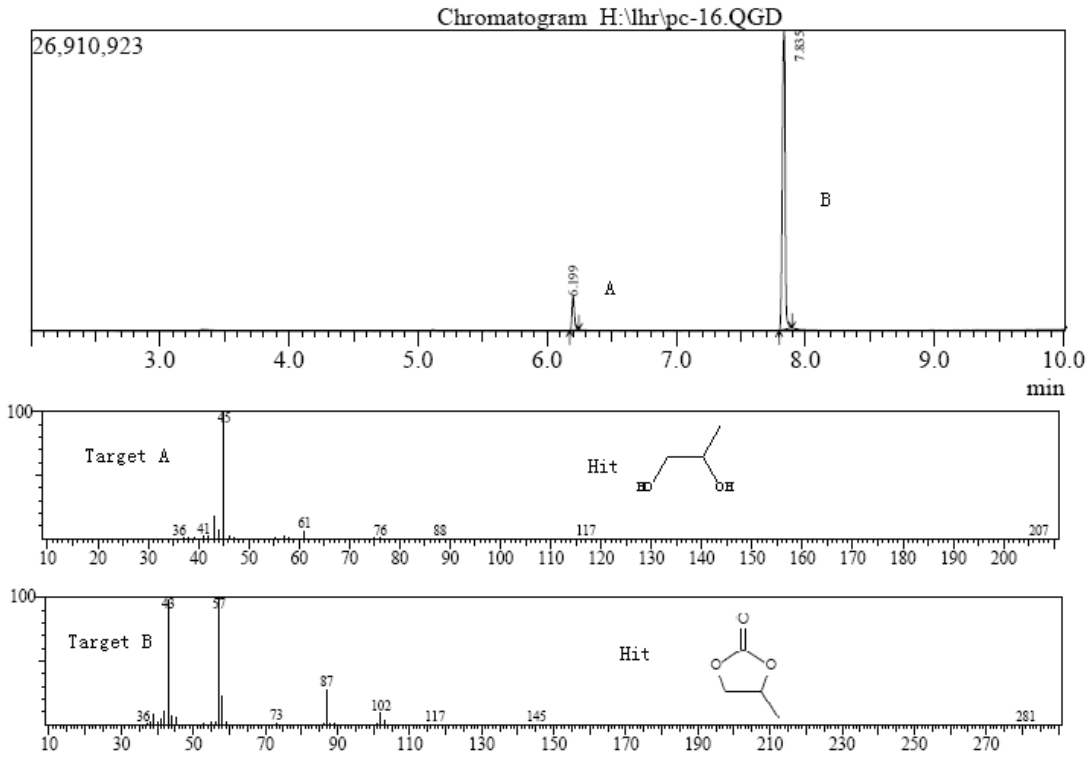

Figure S1. A typical GC-MS spectrum of the product and byproduct (for the reaction condition, see Table 2, entry 1).