Supplementary Material (ESI) for Green Chemistry

Supplementary Information for B816551C

Organic Reactions in Low Melting Mixtures based on Carbohydrates and L-Carnitine – A Comparison

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Contents:

Recycling study

The recycling experiments were conducted in resealable vials (5 mL) with septum. For workup deaerated EtOAc (3 mL) was added through a septum into the vial and the mixture

was stirred at the reaction temperature for 5 min. The organic phase was pipetted or after resolidification of the melt simply decanted off. This procedure was repeated five times (total of 15 mL EtOAc), the combined organic phases were washed with water and analysed by GC using toluene as internal standard. After the first run (75 % yield, 89 % conversion for 1-bromo-4-iodobenzene) yield and conversion dropped significantly to 32 % and 45 %, respectively. In a third run, GC analysis revealed 8 % yield and 27 % conversion. For *p*-iodo anisole the measured yields and conversions for the three runs were 62 %/87 %, 41 %/63 % and 32 %/55 %.

GC study:

To the withdrawn samples toluene (20 µmol/L) was added as internal standard. The mixture (200 µL sample and 200 µL standard) was filtered and injected for quantification.

Column: Capillary Column
J+W Scientific / DB-5MS / 30m x 0.25ID / 0.25um Film
Max programmable temp. 325°C (350°C)
GC: HP5890 II
Pressure Control:
Panel Hydrogen: 14 psi (100 kPa)
Panel Air Pressure: 40 psi (260 kPa)
Panel Carrier (H2): -- psi (--- kPa)
Column Head Pressure: 14.5 psi-> 100 kPa -> ~1.7 ml/min
Carrier = ~1.7 ml/min (system off)
Air = -- ml/min (system off)
Carrier + Air = --- ml/ml (system off)
total (FID) = 290 ml/min (system on)
Split: (40 ml/min)
Septum Purge: 6 ml/min
Liner: Supelco split/splitless Injection Sleeve
4mm ID packed w/deactivated glass wool
Septum: Agilent Inlet Septa Part 5183-4757
Bleed/Temp Optimized Non-Stick 11mm
Splitlesstime 0 Minutes

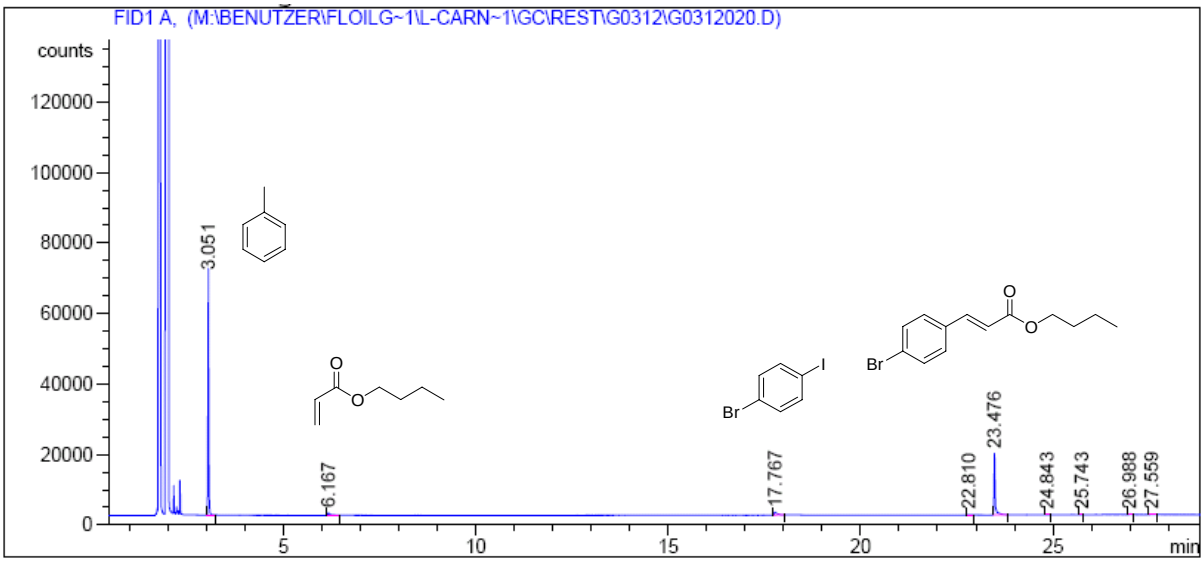

Fig. S1 Chromatogram for the reaction of 1-bromo-4-iodo benzene with *n*-butyl acrylate in sorbitol/urea (1:1, wt/wt).

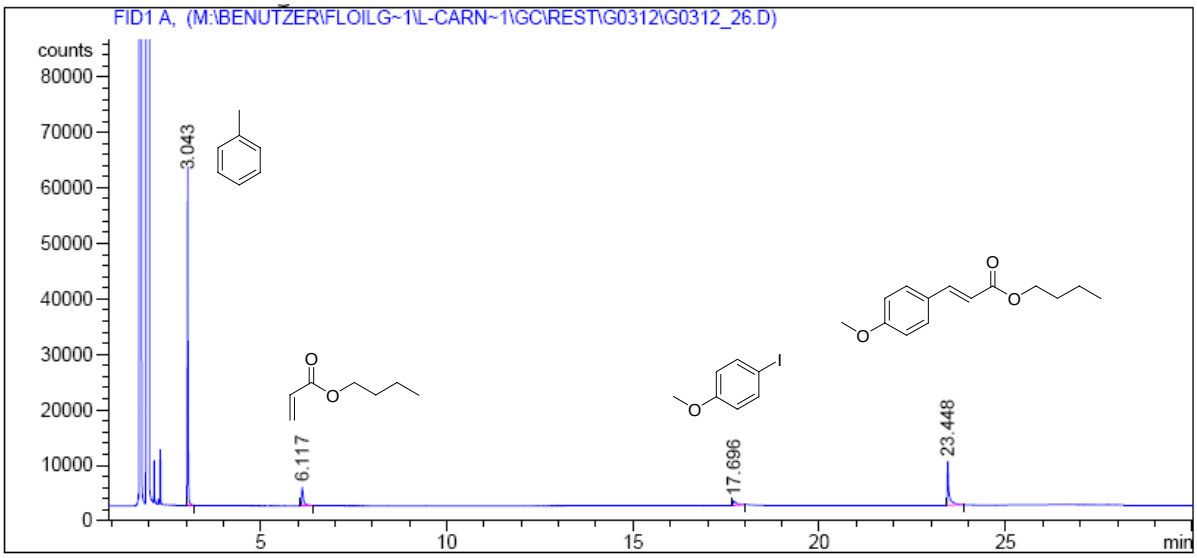

Fig. S2 Chromatogram for the reaction of 4-iodoanisole with *n*-butyl acrylate sorbitol/urea (1:1, wt/wt).

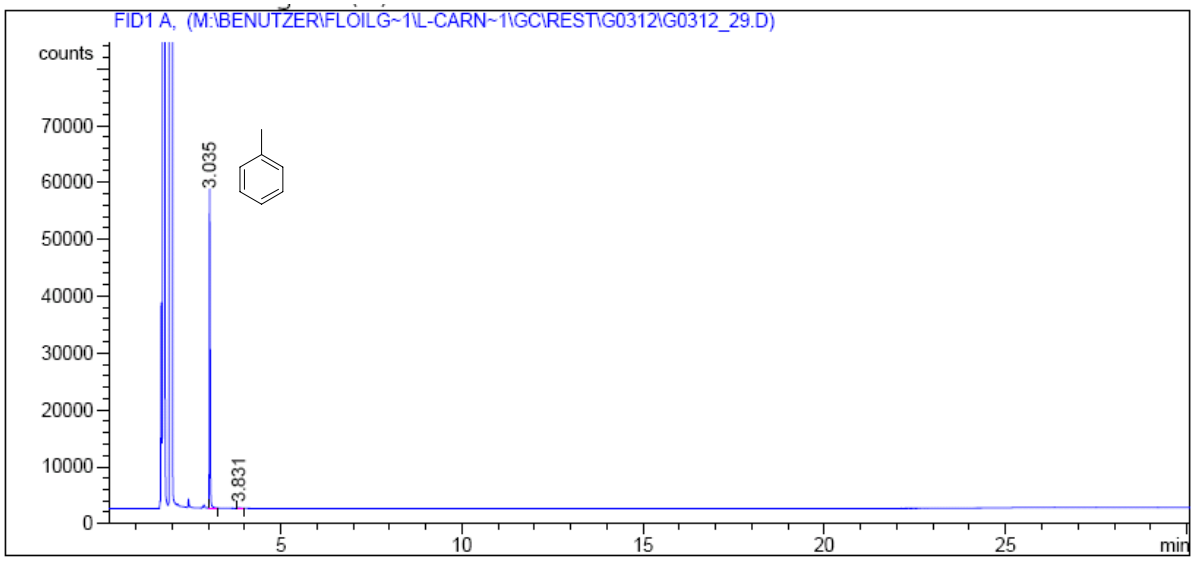

Fig. S3 Chromatogram for the reaction of 1-bromo-4-iodo benzene with *n*-butyl acrylate after 3 recycling runs in sorbitol/urea (1:1, wt/wt) and aqueous work-up (melt was dissolved in water (50 mL) and subsequently extracted with EtOAc (3x20 mL), the combined organic phases were analysed for remaining substances).

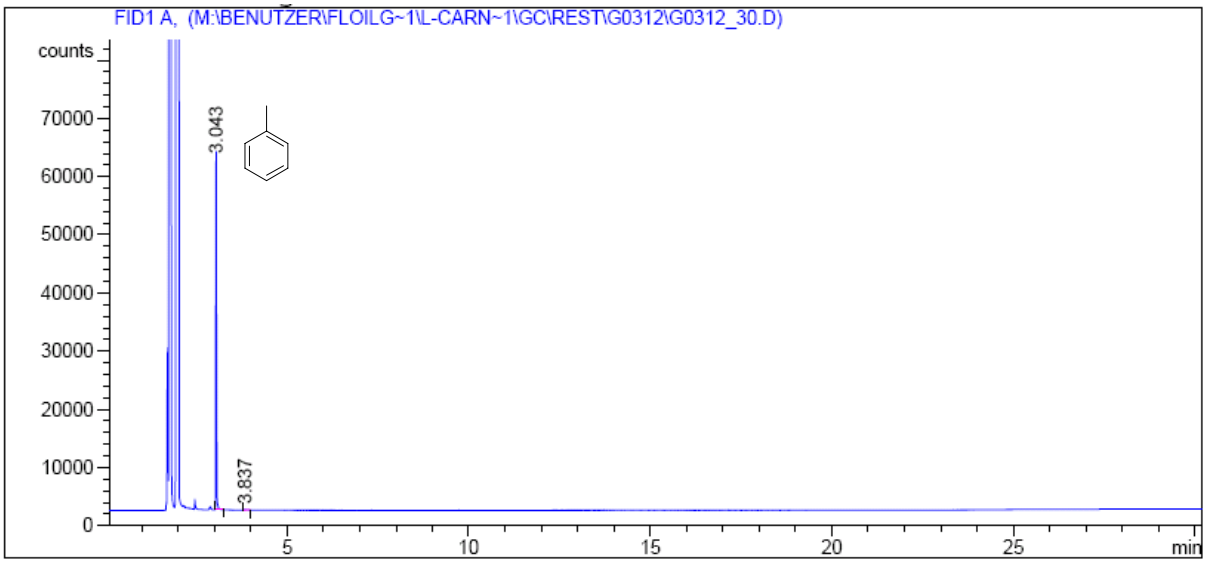

Fig. S4 Chromatogram for the reaction of 4-iodoanisole with *n*-butyl acrylate after 3 recycling runs in sorbitol/urea (1:1, wt/wt) and aqueous work-up (melt was dissolved in water (50 mL) and subsequently extracted with EtOAc (3x20 mL), the combined organic phases were analysed for remaining substances).

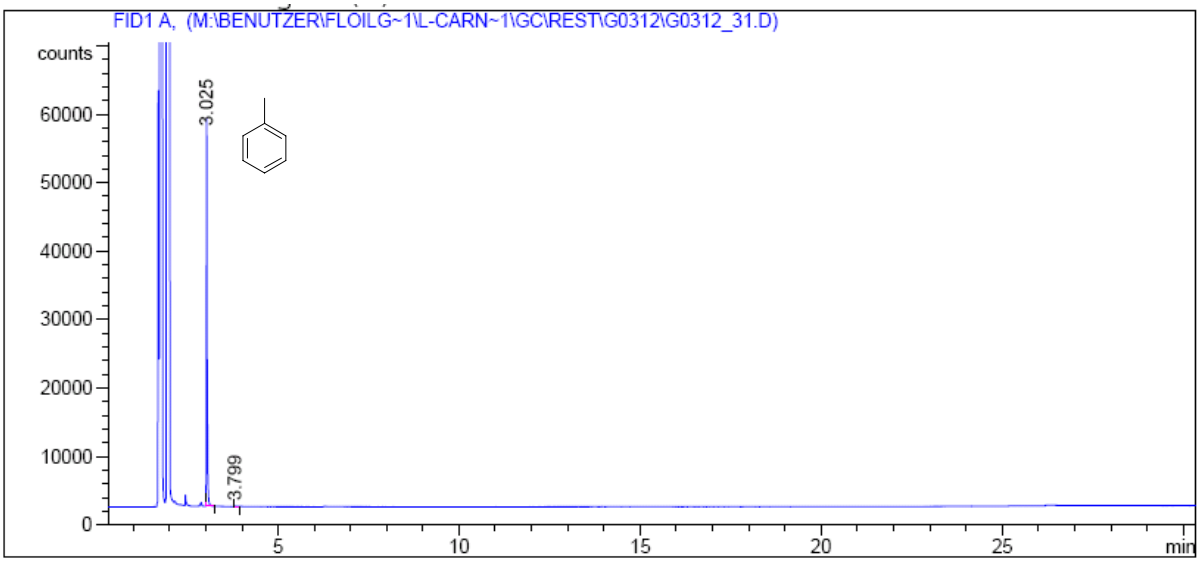

Fig. S5 Chromatogram for the reaction of 4-iodoanisole with *n*-butyl acrylate in L-carnitine/urea after aqueous work-up with 3 mL EtOAc (aqueous phase was extracted with EtOAc (3x20 mL) and analysed).

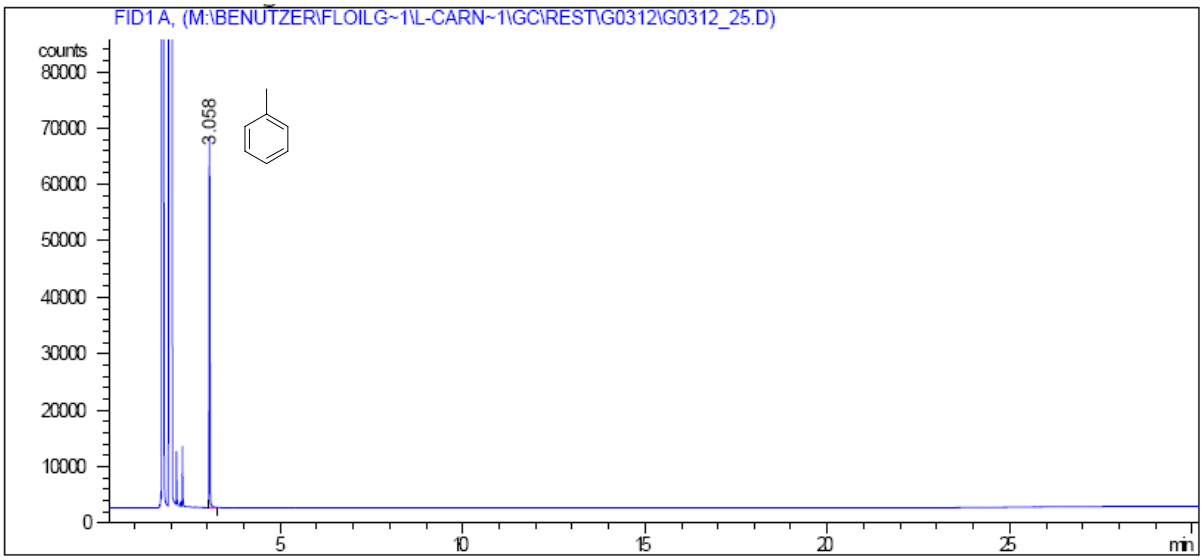

Fig. S6 Chromatogram for the reaction of 4-iodoanisole with *n*-butyl acrylate in sorbitol/urea/NH_4Cl after aqueous work-up with 3 mL EtOAc (aqueous phase was extracted with EtOAc (3x20 mL) and analysed).

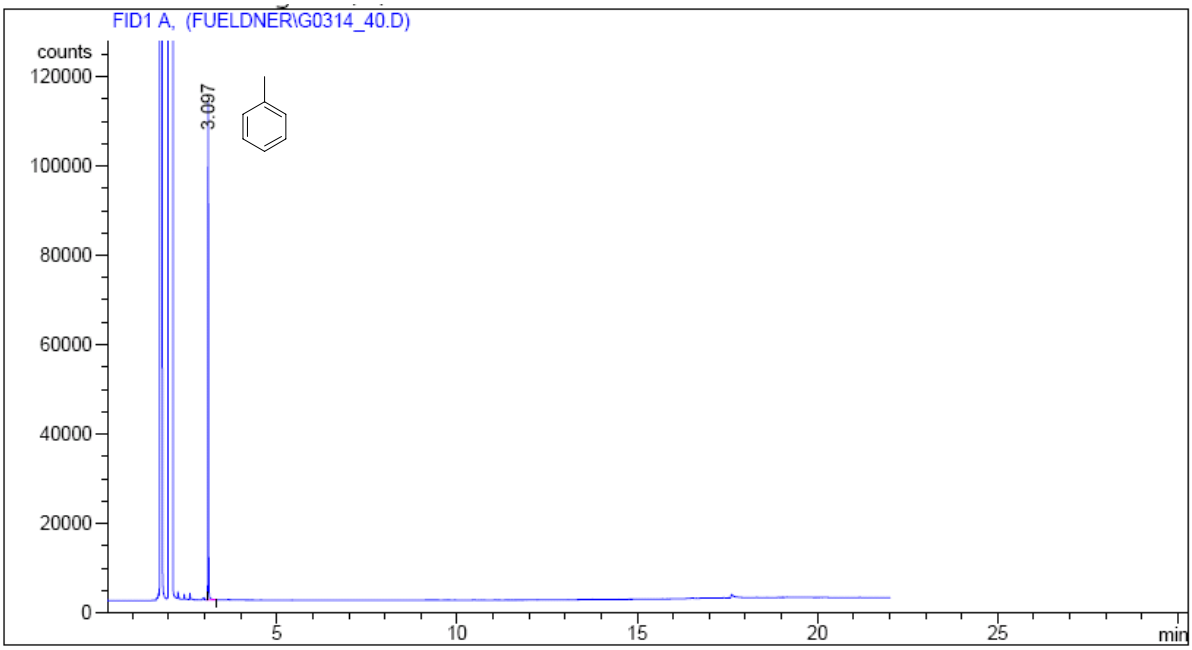

Fig. S7 Chromatogram for the reaction of 4-iodoanisole with *n*-butyl acrylate in D-mannose/DMU after aqueous work-up with 3 mL EtOAc (aqueous phase was extracted with EtOAc (3x20 mL) and analysed).