CO₂-catalysed aldol condensation of 5-hydroxymethylfurfural and acetone to a jet fuel precursor

Roland Lee, Jesse R. Vanderveen, Pascale Champagne and Philip G. Jessop*

Method
Single step dehydration, aldol condensation, and hydrogenation was carried out in a Parr 31 mL high pressure vessel (T316SS, Parr no. N4742, modified to 31 mL). Glucose/5-HMF, hydrogenation catalyst (Pt (nominally 50 %), Ru (nominally 25 %) on high surface area advanced carbon support supplied by Alfa Aesar (stock# 12100) and reaction solvents were added to the Parr vessel equipped with a stir bar. The vessel was then closed and heated in an oil bath to the required temperature and allowed to equilibrate for 30 min. Following equilibration, the reactor was pressurized with CO₂ and H₂ to operating conditions.

Following reaction (both for conversion of 5-HMF to aldol condensation product or direct conversion from glucose) and dilution, the samples were analyzed with the use of GC-MS (Perkin Elmer Clarus 680 gas chromatograph (GC)), using an Elite-SMS column (30 m, 250 µm i.d., 0.25 µm film of 5% diphenyl 95% dimethyl polysiloxane). Initially the temperature was held at 30 °C for 0 min, followed by a ramp to 125 °C at a rate of 2.5 °C /min held for 1 min, ramp to 260 °C at a rate of 20 °C /min held for 1 min and, finally, ramp to 300 °C at a rate of 20 °C /min held for 3 min. The injector temperature was held at 250 °C and the detector at 200 °C for the duration of the analysis. Carrier gas (helium) flow rate was maintained at 1 mL/min. Aldol condensation products were independently prepared by a literature method¹⁷ and utilized for calibration and determination of retention time. Chromatograms and data were collected precisely with the use of Perkin Elmer TurboMass, version 5.4.2.1617 chromatography software.