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Continuous Catalytic Friedel-Crafts Acylation in Biphasic Media Ionic Liquid / Supercritical Carbon Dioxide

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Detailed description of the apparatus in Fig. 1

All connections were made from commercial stainless steel fittings (Hoke/Germany) or glass fittings of the GL series (Schott/Germany). CO₂ Reservoir 1: The high pressure reservoir is part of the infrastructure of the institute and is supplied by Hofer/Germany. Pressure reducer (2): The pressure reduced is of the RHP-Series and supplied by Hoke/Germany. Mass flow controller (3): L1, Bronkhorst supplied by Mättig Mess-und Regeltechnik /Germany. High pressure piston pump (4) is supplied by Gilson/Germany. As substrate reservoir (5) a modified buret (glass-blowing workshop of the institute) was used to enable handling of the substrate mixture under argon atmosphere. As reactor (6) a custom built (workshop of the institute) autoclave of 10mL volume equipped with glass windows. Heating and stirring by means of a teflon coated magnetic stirring bar (Komet, VWR/Germany) was achieved by a magnetic stirring plate (IKA/Germany). As back pressure regulator (9) either a commercially available SFC module (Jasco/Germany) or a custom built pneumatic valve (Sitec/Switzerland) embedded into a heating block (mechanical workshop of the institute) was used. Cooling traps and gas washer (10) (glass-blowing workshop of the institute) were cooled with dry ice /acetone and toluene was used as solvent. Pressure transducers from Wika/Germany and thermocouples type K (Thermocoax, RS Electronics/Germany) were used. Data acquisition and control was carried out by LabView (National Instruments/Germany).

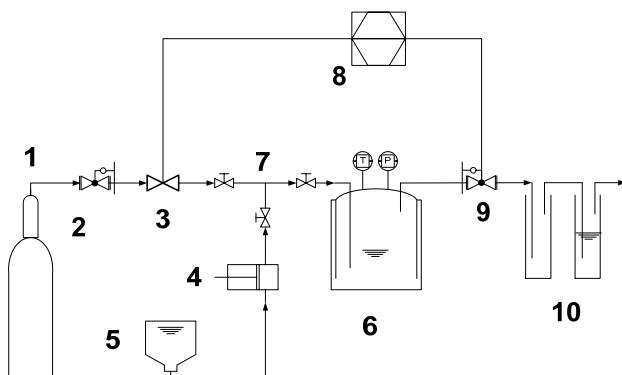


Figure 1: Reactor for batch and continuous flow Friedel-Crafts acylation. CO₂ Reservoir 1, Pressure reducer 2, Mass flow controller 3, high pressure piston pump 4, substrate reservoir 5, stirred tank reactor with pressure temperatur transducer 6, valves 7, analog/digital interface unit 8, back pressure regulator 9, cooling trap and gas washer 10.