Selective hydrogenation in flow over Pd nanoparticles supported on hierarchical silica monoliths as efficient microreactors

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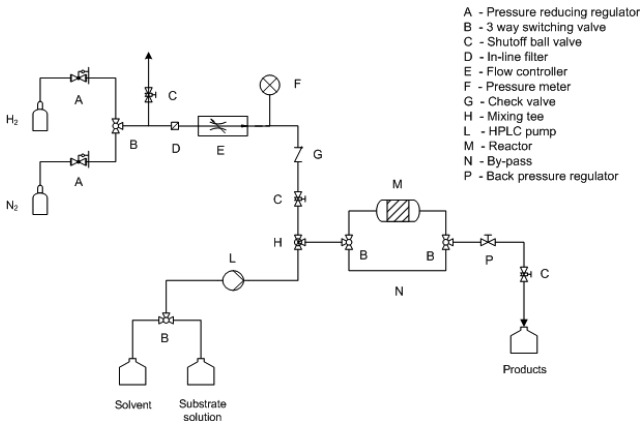
Supporting informations



Scheme S1. Schematic view of continuous-flow microreactor system.

Catalytic flow hydrogenations were carried out using the continuous-flow reactor system constructed at Istituto di Chimica dei Composti Organo Metallici, Firenze (Italy) shown in Scheme S1. The system was designed to allow for a simultaneous flow of substrate solution and hydrogen gas (up to 40 bar pressure) through a reactor tube containing the heterogeneous catalyst. The reactor was completely inert, as all wet parts were made of PEEK, PFA or PFTE. The flow of the substrate solution was regulated by an Alltech® model 426 HPLC pump in PEEK. A constant flow of hydrogen gas was adjusted by a flow controller BRONKHORST HI-TEC model F200CV-002-RGD-11-V-MFC. The hydrogen pressure in the reactor was monitored by a BRONKHORST HI-TEC P502C-AGD-11-V-6K0R-EPC meter. The concurrent flows of gas and liquid were driven through a T-shaped PEEK mixer to ensure efficient gas dispersion. The mixed hydrogen-substrate solution stream was introduced in the reactor through a 6-port Rheodyne mod. 9060 switching valve in PEEK. The Pd-MonoSil catalyst (typically 6 mm diameter, 2 cm length), cladded into a heat-shrinkable PTFE tube together with two glass tubes (4 mm inner diameter) at each end, was connected in a top-down arrangement to the system by PFA Swagelok fittings and Chemraz® O-rings. At the outlet of the reactor, the product solution was collected for GC analysis and the excess amount of the hydrogen gas released to the atmospheric pressure. Commercially available H_2 (99.995%) was used as received.