

AMT TN-04

Aromatic Hydrogenation in the Coflore® MHR

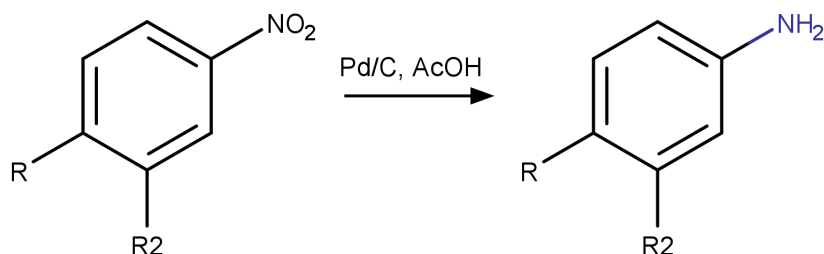


Figure 1: Reaction scheme outlining a typical aromatic nitro reduction reaction.

Introduction

Catalytic hydrogenation remains a difficult process to manage using conventional plant equipment, largely due to the safety concerns and costs associated with processing hydrogen at high pressures using large reaction vessels. Many safety concerns can be lessened or negated by switching to flow hydrogenation using a limited volume flow reactor, but this naturally brings about its own challenges. The Coflore® Multi-Phase Handling Rig (MHR) has been designed specifically to handle reactions such as metal-catalysed hydrogenations over extended run times at a high throughput. Consisting of the Coflore® ATR and SXV, the MHR can process up to 2,880 L of material per day for a reaction with a 5-minute residence time. Gas, such as hydrogen, is flowed through the reactor counter-currently against a mixture of catalyst and reactant. A gravity-assisted solids trap then allows for downstream separation of catalyst and product. Recovered catalyst can be recycled back into the MHR or reprocessed outside of the MHR system.

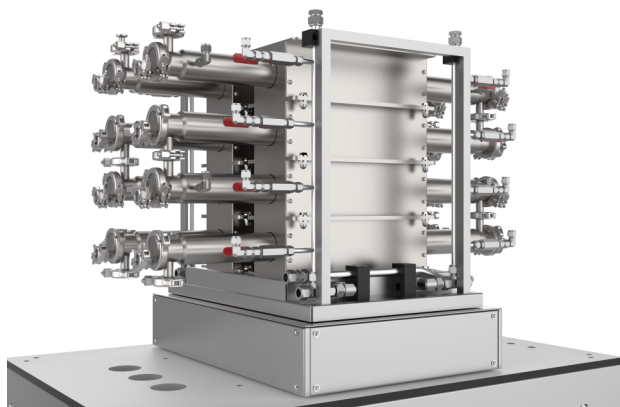


Figure 2: A bank of 8 Coflore® ATR reactor tubes.

Hydrogenation Equipment Setup

The peripheral process equipment required for particular reactions in flow can vary due to parameters such as the density, particle size and settling characteristics of solids, and the solubility of reagents and products. Catalysts such as palladium on carbon, or platinum on carbon, can be characterised as “fast-settling” solids which presents a particular challenge as to how to keep these solids well suspended in flow. The solids exchange valve (SXV) overcomes this issue and allows for dosing of fast-settling solids such as palladium or platinum on carbon at lower flow rates that would ordinarily block slurry pumps. Mechanical agitation in the ATR reactor tubes effectively prevents settling of the solid, which can then be separated from the reaction product via a gravity-assisted solids collection vessel to allow for catalyst recycling. Hydrogen is fed through the ATR counter-current to the reaction mixture.

Feasibility Case Study

A recent client feasibility study involved hydrogenation of an aromatic nitro compound to form the corresponding amino compound (Figure 1). The reaction was performed in a 700 mL ATR at 80°C and 8 bar with a 2:1 ratio of hydrogen to nitro compound and 5%w/v suspension of Pd/C. The reaction yielded the corresponding amino compound in a 90% yield at a 60-minute residence time, twice as fast as an equivalent batch process as reported in the literature.