Teledyne LABS

Hydrogenation with Teledyne ISCO Syringe Pumps

keywords: syringe pump, hydrogenation

Abstract

Teledyne ISCO syringe pumps are ideal for intermediate-scale hydrogenation reactions. Such reactions could be for production of medicines for clinical trials or for evaluation of industrial processes. Syringe pumps are ideal for continuous flow processes. Scaling up a process is easily done by adding more syringe pumps to produce material in parallel.

The examples shown here use hydrogenation to create bio-based solvents, catalytic conversion of ligno-cellulose compounds, and production of hydrogen peroxide in a trickle-bed reactor. All examples in this application note use a syringe pump to deliver the liquid reactants.

Why use a syringe pump for hydrogenation?

Teledyne ISCO syringe pumps provide needed precision delivery of catalysts and reagents so that they can be reliably dispensed. These pumps can deliver your pumped substance pulse-free at a low, precise flow rate. Setting up gradient dispensing will allow delivery of different amounts of substance at preselected flow rates for the required length of time. Teledyne ISCO syringe pumps can be metered with great accuracy, without any pulsation or flow anomalies. Also, ISCO syringe pumps with a temperature control jacket will allow any pumped substance inside pump cylinder to be heated up or cooled down from –30 °C to +100 °C. And finally, the special fittings that are used in these syringe pumps prevent leaks and ensure safety at any pressure levels.

Some safety features of Teledyne ISCO syringe pumps include:

- User–set pressure limits in the pump controller to make sure pump will not go into overpressure.
- A mechanical sheer key that will break if the pump reaches over 1.5 times the maximum pressure of the pump.

Some examples

Hydrogenation of esters to ethers

Bio-based solvents have the advantages of reduced toxicity, improved worker safety, and decreased environmental impact. Examples of bio-based solvents include alcohols, glycols, diols, esters, and hydrocarbons, but the use of these solvents is limited by poor stability such as such as ester hydrolysis, reactivity from hydroxyl groups, or limited solvation of hydrocarbons. Ethers possess stability and solvation properties that make them useful with other organic and aqueous solvents.

The hydrogenation of propyl acetate to make 1-ethoxy propane used a Teledyne ISCO 100DX[1] syringe pump to study site requirements, particle size effects, and turnover rates as a function of reactant concentration.[i]

Propyl acetate was tested because it is representative of esters recovered from biomass and was an example of an unsymmetrical ether whose synthesis was otherwise difficult.

1 The Teledyne ISCO 100DX and 500D syringe pumps are no longer produced. The SyriXus 260x and 500x, respectively, are recommended for this application.

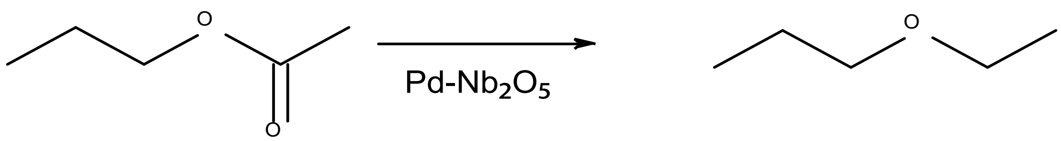

Figure 1. Catalytic hydrogenation of propyl acetate to 1-ethoxy propane

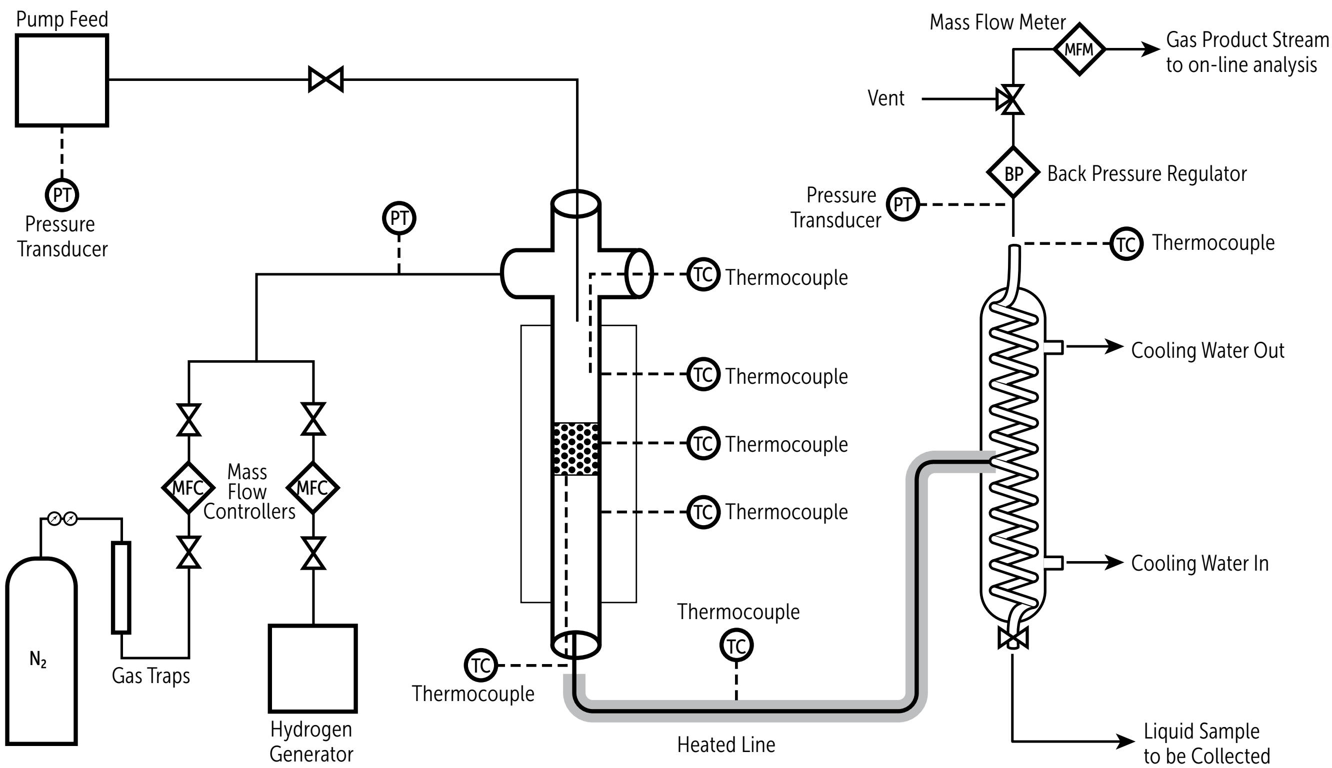

Figure 2. Schematic of packed bed flow reactor system used for reduction of lignin surrogates. (Runnebaum et al.[iii])

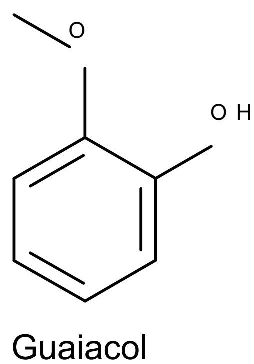

Figure 3. Guaiacol

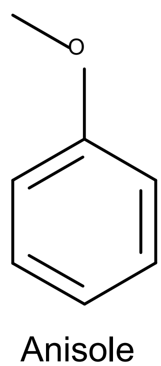

Figure 4. Anisole

Lignocellulose catalytic conversion

Lignocellulosic biomass is valuable as a renewable resource as fuels and chemicals because it is abundant and inexpensive. However, the oxygen content of this material renders it corrosive, unstable, and insoluble with existing fuels such as gasoline and diesel. Both of the following examples use different compounds as models for lignin for hydrogenation[ii,iii].

A Teledyne ISCO 500D pump was used to load the anisole into the packed-bed flow reactor, which was Pt/y-alumina powder containing 1% Pt.

Trickle-bed reactor

Hydrogen peroxide is an environmentally compatible oxidant with several applications in the paper, textile, and wastewater treatment industries[iv]. A 500D pump was used to feed the liquid phase of a trickle-bed reactor (TBR) with a temperature-controlled syringe barrel.

The syringe pump was used to deliver a methanol liquid phase.

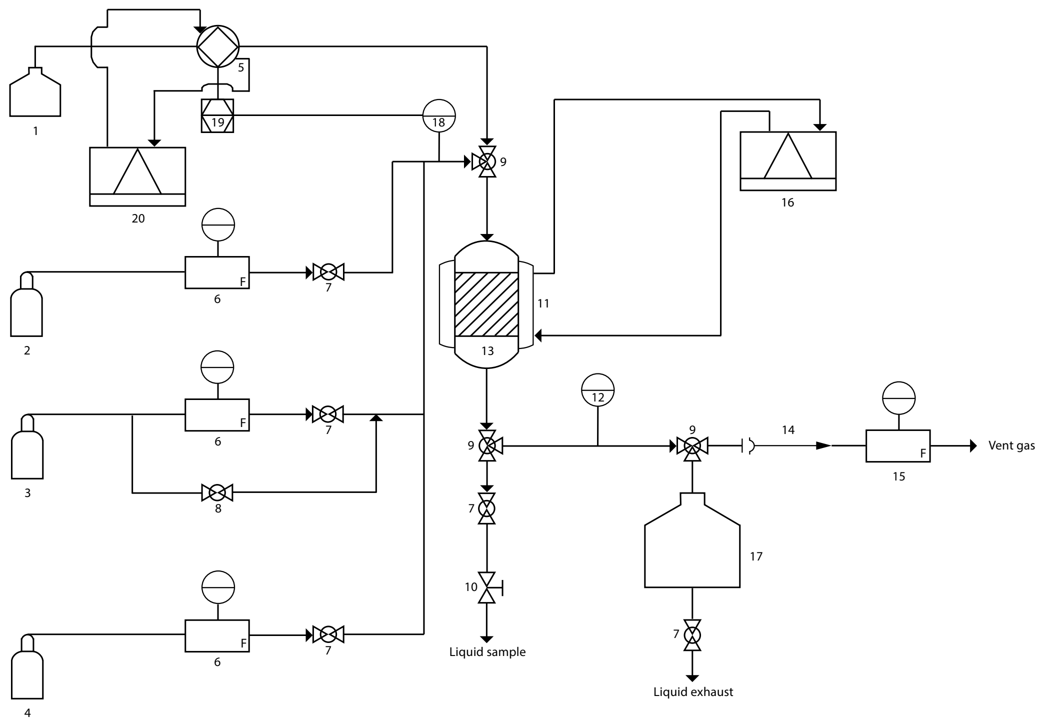

Figure 5. Trickle-bed reactor status. Schematic of the experimental apparatus. (1) Liquid solvent supply. (2, 3, 4) Gas bottles: N_2 CO_2/H_2 (97.5/2.5%), and O_2 respectively. (5) Syringe pump. (6) Mass flow controller (MFC). (7, 8) On/off and bypass valves. (10) Liquid sampling. (11, 16) External cooling wiht chiller. (12, 18) Pressure gauges. (13) Trickle bed reactor (TBR). (14) Rupture disk. (15) Back pressure controller (BPC). (17) Liquid collection. (19) Computer. (20) Chiller. (Biasi et al.[iv])

Conclusion

Teledyne ISCO syringe pumps can be used for a variety of continuous flow reactions. This application note listed some hydrogenation reactions, including from a trickle-bed reactor. The pumps delivered the reactants to be reduced in liquid matrices. The syringe pumps provide precise flow based on a programmed flow rate or pressure so that exactly the correct volume of reactant is fed to the reaction to prevent waste or side products.

Notes

i Pathways for Reactions of Esters with H2 over Supported Pd Catalysts: Elementary Steps, Site Requirements, and Particle Size Effects. Claudia E. Berdugo-Díaz, Yang Sik Yun, Melissa T. Manetsch, Jing Luo, David G. Barton, Xue Chen, and David W. Flaherty. *ACS Catalysis* **2022** *12* (15), 9717-9734 DOI: 10.1021/acscatal.2c02129

ii Catalytic Conversion of Guaiacol Catalyzed by Platinum Supported on Alumina: Reaction Network Including Hydrodeoxygenation Reactions. Tarit Nimmanwudipong, Ron C. Runnebaum, David E. Block, and Bruce C. Gates. *Energy Fuels* **2011**, *25*, 8, 3417–3427.

iii Conversion of Anisole Catalyzed by Platinum Supported on Alumina: The Reaction Network. Ron C. Runnebaum, Rodrigo J. Lobo-Lapidus, Tarit Nimmanwudipong, David E. Block, and Bruce C. Gates. *Energy & Fuels* **2011** *25* (10), 4776-4785 DOI: 10.1021/ef2010699

iv Hydrogen Peroxide Direct Synthesis: Selectivity Enhancement in a Trickle Bed Reactor. Pierdomenico Biasi, Federica Menegazzo, Francesco Pinna, Kari Eränen, Paolo Canu, and Tapio O. Salmi. *Industrial & Engineering Chemistry Research* **2010** *49* (21), 10627-10632. DOI: 10.1021/ie100550k