

Micoreactor Systems

Using Teledyne ISCO Syringe Pumps

Overview

Continuing research at the United States Environmental Protection Agency's National Risk Management Research Laboratory in Cincinnati, Ohio focuses on the development of process-intensified and green reaction strategies for pharmaceutical relevant reactions. Teledyne ISCO high precision syringe pumps have been invaluable to this work in demonstrating organic chemistries in the Spinning Tube-In-Tube (STT[®]) reactor. These synthetic chemistries include the high-conversion synthesis of esters from acid anhydrides and alcohols, of imidazole-based ionic liquids, and of dipyrromethanes.

This fundamental research work has been carried out in a reaction system comprised of two Teledyne ISCO Model 100DX [see note] syringe pumps with the STT[®] Magellan[™] microreactor. The key feature of this microchemical reactor system is the high ratio of surface area to volume. As a result, mass transfer, heat transfer, and mixing are vastly more efficient. Other benefits of this type of system include:

- Increased throughput
- Minimized unwanted by-product formation
- Safer chemistries
- Solventless or decreased solvent usage
- Minimal environmental footprint
- Smaller physical footprint

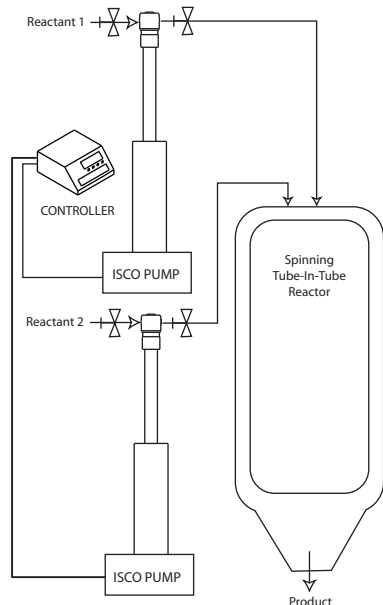


Figure 1: STT Reactor Setup with two ISCO pumps operating independently and simultaneously from one controller, in constant flow mode

Setup

Figure 1 shows the configuration of an STT[®] 1.2mL microreactor with two separately connected Teledyne ISCO syringe pumps, each with a manual valve for the inlet and outlet. As shown, each pump is connected separately to the reactor with a manual valve on each inlet and outlet.

Based upon the chemistries employed, the required residence times in the reactor vary from 10 seconds to 5 minutes. This requires the syringe pumps to operate at flow rates from 0.100 mL/minute to up to 10 mL/minute. In order for the reactions to yield the desired conversion and selectivities while minimizing unwanted reaction by-products, the concentrations of the reactants must be precise and kept constant.

Further automation, as seen in Figure 2, can be achieved with a dual-pump, continuous flow system, where reactions can be produced indefinitely.

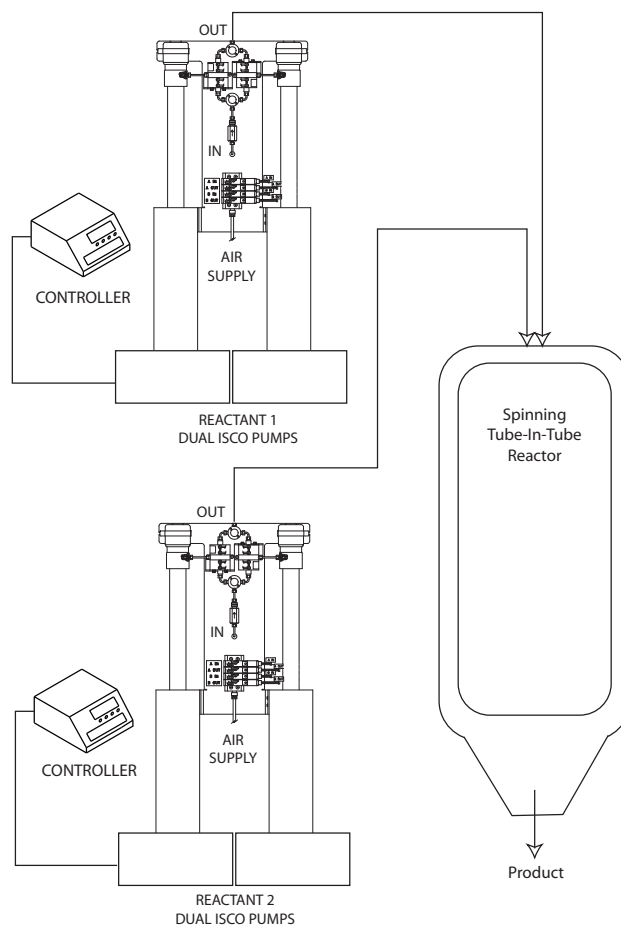


Figure 2: STT Reactor Setup with two dual pump systems for continuous flow

Why Teledyne ISCO Pumps?

Teledyne ISCO pumps have a flow rate accuracy of $\pm 0.5\%$ or better, and flows are pulse-less. Pulse-less flow means fluid pressure and density are constant, without fluctuation in solvating properties or chemical potential. The pumps can be operated in either constant flow or constant pressure mode, as a stand-alone unit, or via external control. Teledyne ISCO syringe pumps also have a flow rate and pressure limits alarm to alert the user to any change in conditions.

In addition, Teledyne ISCO Syringe pumps have much larger cylinder volumes (up to 1 L), eliminating the need for refills with most batch runs. For continuous operation, however, dual pump systems deliver pulse-less flow with the same accuracy and precision of single pumps, and can operate unattended.

Other pumps types, such as reciprocating pumps, produce a flow or pressure pulse that affects the quality of the product produced. For reciprocating pumps, the piston-swept volumes are typically small, and thereby require frequent refills, creating periodic pressure drops. Although dual piston reciprocating pumps produce smoother flows than single piston pumps, they cannot produce the pulse-less, accurate flows required for many high performance applications.

Recommended Pumps

Typically, chemical engineers choose to work with the Model 260x for low flow applications, 500x for most common applications, or the 500HLf pump for Hazardous Location environments. Single pumps are most often used in batch applications, while dual pumps are used in continuous flow.

Table 1: Teledyne ISCO Pumps

	1000x	500HLf	500x	260x	65x
Flow Range (mL/min)	0.100 - 408	0.001 - 204	0.001 - 204	0.001 - 107	0.00001 - 25
Pressure Range (psi)	0 - 2,000	0 - 3,750	0 - 5,000	0 - 9,500	0 - 20,000

STT® is a registered trademark of Kreido Biofuels, Inc.
(www.kreido.com)

REFERENCES

1) Gonzalez, M. Process Intensified Green Reaction Chemistries Performed in the Spinning Tube-In-Tube (STT®) Reactor. 2007 Spring National American Institute for Chemical Engineers (AIChE). Houston, TX. 22-27 April 2007.

2) Palusinski, O.A.; Vrudhula, S.; Znamirovski, L.; Humbert, D. August 2001, 97(8), 60-66. *Journal of Chemical Engineering Progress*. April 2008 <http://findarticles.com/p/articles/mi_qa5350/is_200108/ai_n21476192>

3) Rouhi, A.M. June 2004. *C&E News*. April 2008 <<http://pubs.ac.org/cen/news/8226/8226/earlysci2.html>>

4) Gonzalez, Michael, Ph.D, USEPA, Cincinnati, OH. Personal Interview. 5 June 2008.

Note:

The 100DX model pump, which was used during the original experiment, is discontinued. Current model 260x is the recommended replacement for the older 100DX model.

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Product model names have been updated in this document to reflect current pump offerings.*

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