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ILS-Integrated Lab Solutions Uses Equilibar to Provide Flow Control for High Temperature Vinyl Acetate Monomer Reactor

Researchers face challenges when making flow control decisions for processes involving extremely high temperatures. Conventional mass flow controllers for the low flow rates used at the laboratory scale cannot often be used at high temperatures. ILS-Integrated Lab Solutions in Berlin has found a successful solution to this problem by using an Equilibar® dome-loaded multiple orifice back pressure regulator in combination with a high-temperature mass flow sensor to provide reliable flow control despite extremely high temperatures.

Background

ILS is a Berlin-based fabricator of high-throughput to pilot-scale catalyst testing workflows. ILS researchers frequently use Equilibar's dome-loaded multiple orifice regulators for pressure control of particularly complex applications. Recently, ILS also used Equilibar technology to provide flow control for an application involving extreme temperatures that were too high for traditional mass flow controllers.

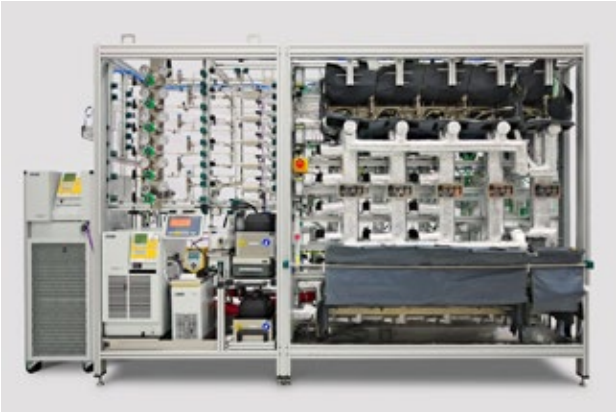

Figure 1: ILS parallel test reactor system for VAM Vinyl-Acetate-Monomer catalyst R&D

The Problem

ILS constructed a parallel test reactor for a major supplier of Vinyl Acetate Monomer (VAM) catalysts. VAM is an industrial chemical used in the production of various polymers.

Its synthesis is particularly challenging for the following reasons:

- An explosive mixture of O2/Ethylene is used in the feed
- Acetic acid in the feed can result in corrosion issues, especially if it liquefies
- The feed vapors must be maintained in the vapor-phase, which requires a temperature of 200°C.

Unfortunately, most commercial thermal or Coriolis mass flow controllers suitable for operation at the low flow rates used in laboratories are not able to work at temperatures above about 70°C. Therefore, this application required the selection of both a flow sensor and a control valve capable of handling both the high temperature and the corrosive acid.

The Solution

For this complex scenario, ILS chose to use a high-temperature mass flow meter from Intek in combination with an Equilibar dome-loaded multiple orifice pressure control valve. Together, the components of this unit were able to ensure superior flow control despite the extreme conditions.

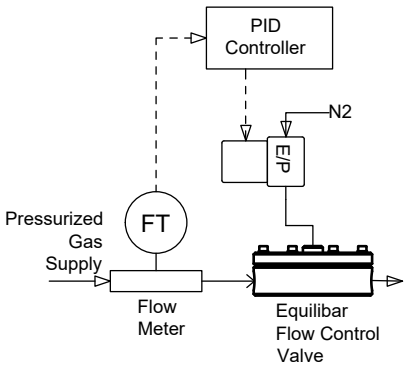

Figure 2: Schematic representation of an Intek flow meter in combination with an Equilibar to create flow control valve setup

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Specifically, ILS used a Siemens process-control system to control the nitrogen dome pressure head on the Equilibar via an electronic pressure controller. With this setup, it was possible to control flow via the dome pressure of the Equilibar, which was operating in a feedback control loop between the electronic controller and the Intek flow meter.

Equilibar LF Series

Equilibar Research Series units are designed to meet the demanding requirements of catalyst research and high pressure reactor control applications. These products hold stable pressure across very wide flow ranges and can handle liquids, gases, or multi-phase mixtures. They are commonly used with highly aggressive chemicals and can perform at temperatures up to 450 °C.

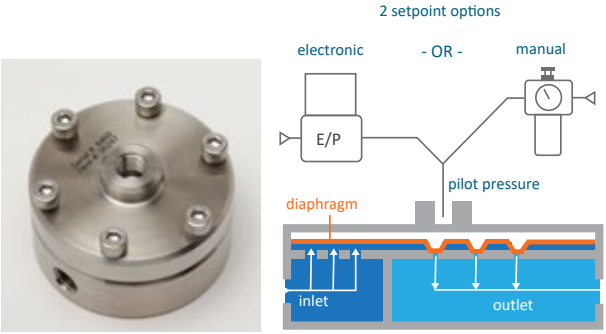

Figure 3: Equilibar® LF Series photo and schematic of how it works

Equilibar Research Series products are 1:1 dome-loaded, meaning that a pilot pressure must be supplied to the dome of the regulator equal to the desired setpoint. This pilot pressure can be supplied by a manual gas regulator or by an electronic pressure regulator for computer automation.

The LF Series is part of Equilibar's patented multiple orifice design, in which more orifices open up as needed to accommodate increasing flow requirements. This design offers the highest possible precision in multi-phase flow applications.

Intek Rheotherm®

The Intek Rheotherm® measurement method employs two resistance temperature detectors (RTDs) to measure flow. One RTD measures the fluid temperature and the other RTD measures the temperature of a constant low-power heater which is cooled by the flowing fluid. The temperature differential between the heated and unheated RTDs provides the primary flow signal as shown in the diagram.

The challenge for most thermal meters in high-temperature applications is the gradient that occurs when there is a large difference between the temperature of the process fluid and the ambient environment. This thermal gradient significantly impacts the accuracy of the sensor. While similar in design to other thermal mass flow meters, the Intek Rheotherm sensor incorporates several design attributes that enable it to be used for high-temperature applications.

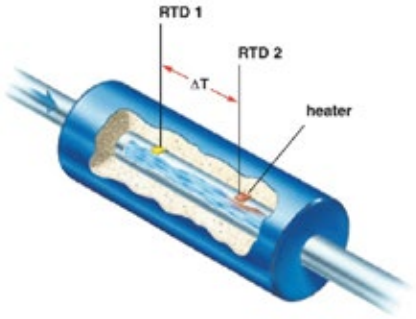

Figure 4: Intek Rheotherm® low flow meter with two RTDs

Intek's design incorporates several heat shields and insulation that mitigate the impact of the thermal gradient on the measurement, enabling the sensor to accurately measure the delta-T required to determine the flow rate.

Unexpected Event Demonstrates Robustness:

Not only was the Intek/Equilibar combination able to handle the application's temperature extremes, an unexpected event demonstrated that the unit could handle even more challenges than the original process involved.

"Unfortunately, the client had a serious problem with the unit in which a small, isolated oxygen/ethylene flame formed in the upstream feed section," said Dr. Anton Nagy, ILS founder. "While this was never a serious safety concern because it involved only a small section of ¼-inch piping, it resulted in the formation of a huge amount of rust that was transported to the vapor feed-control section. "

Figure 5: Rust formation due to flame in upstream feed

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"In these circumstances, a normal mass flow controller would have broken immediately due to blockage. To our amazement, the Intek/Equilibar combination continued to work after just flushing the system with nitrogen and a small amount of water."

This easy repair was possible because the large orifice size of the holes in the Equilibar makes them easy to clean and less prone to blockage. "Because of this, the feed-section of the unit could be purged and immediately started back up," Nagy said. "It wasn't even necessary to change the stainless steel diaphragm."

According to Nagy, the unit has been operating virtually non-stop for approximately two years at the client's facility and is a key component of their catalyst R&D development work flow.

Contact Equilibar

Equilibar is a provider of unique and innovative pressure control solutions based near Asheville, North Carolina. The patented back pressure technology is used in a wide array of processes including catalyst, petrochemical, supercritical and other industrial applications. For more information contact an Equilibar application specialist at inquiry@equilibar.com or 828.650.6590.

Anton Nagy, founder and CEO of ILS-Integrated Lab Solutions, has worked previously as a research chemist at Avantium, Shell and Bayer. Dr. Nagy can be reached at +49-302639669100 or anton.nagy@integratedlabsolutions.com. ILS-Integrated Lab Solutions Gmbh, Barbara-McClintock-Strasse 11, 12489 Berlin, Germany.

Jeff Jennings, P.E., is founder and president of Equilibar, LLC, a provider of high precision pressure control solutions. Prior to Equilibar, he worked as a process development engineer for 23 years. He is a licensed professional engineer in North Carolina and holds several patents. Jennings can be reached at jeffjennings@equilibar.com or 828-650-6590.

Intek, Inc., based in Westerville, OH, has been engineering and manufacturing flow measurement solutions for over 40 years. Specializing in low-flow applications, Intek offers unique solutions for challenging measurement problems including compatibility with most industrial liquids and gases. Learn more at intekflow.com/flow-measurement-instruments/ or contact them at 888-LOW-FLOW (569-3569).