EQUILIBAR
PRECISION PRESSURE CONTROL

For immediate release: **June 2015**

At University of Stuttgart, Equilibar regulator provides stable pressure control for Gas Chromatography column used in plasticizer research

At only 0.5 bar, the dome-loaded back pressure regulator provides stable gas by-pass around the Gas Chromatography column downstream of catalytic reactor.

Background

The Institute of Chemical Technology at Germany's University of Stuttgart is leading research efforts to find improved catalysts for the production of phthalic anhydride, a primary precursor for the production of plasticizers. Previous research in this area has been limited by the need to maintain an extremely low concentration of the o-xylene reactant (<1%) in the catalytic reactor to avoid hot spots and explosive mixtures. Stuttgart researchers have developed a new method that is able to safely incorporate higher concentrations of o-xylene, which provide more predictive results. This method successfully utilizes an Equilibar back pressure regulator to provide stable low pressure control despite high temperatures and a wide range of conditions.

Challenge

In this application, Stuttgart researchers use a Gas Chromatography column downstream of the oxidation reactor to monitor reaction efficiency. This process requires back pressure control in parallel with the Gas Chromatography to allow excess flow to pass around the column while also maintaining a stable upstream process pressure. In addition, the application requires a range of set-point pressures from 0.5 to 2.0 bar(g), with a gas flow ranging from 200-2500 ml/min. Finally, the valve must be maintained at temperatures as high as 300C to prevent condensation of fluid components.

The researchers wanted to find a simple, economical pressure control solution that could handle these challenging conditions. They first tried a needle valve to maintain the upstream pressure. This static valve failed to maintain the process at stable pressure, with significant pressure swings of 0.5 bar over a 20 minute period of time. They then considered a control valve solution but found the complexity of providing an automated controller to accompany the valve to be undesirable and expensive.

Looking for a more innovative approach, Stuttgart researchers contacted Equilibar to determine the feasibility of using a dome-loaded back pressure regulator. Equilibar regulators are designed to use a flexible, frictionless diaphragm as the only moving part, thereby taking a dramatically different approach compared to traditional back pressure regulators. They are able to accurately maintain pressure over a wide range of flow rates and can be made from a wide range of materials to accommodate aggressive chemistries and temperatures.

Solution

Working with Equilibar engineers, the Stuttgart researchers selected the EB1LF1 dome-loaded back pressure regulator, coupled with a simple air pressure regulator to supply the 1:1 set-point pressure. A thin polymide diaphragm and FFKM Kalrez o-rings were selected to handle the higher temperature conditions.

The EB1LF1 demonstrated its ability to control the GC inlet pressure precisely, even with the temperature specifications. Over several weeks of testing, the pressure deviation was observed to be within 0.01 bar of set-point. Very few adjustments were required once the flow rate was established, and no further adjustments of the set-point were required despite varying reactor temperatures.

Process Details

As shown in Figure 1, the process can be separated into two sections: 1.) dosage and 2.) reaction with analysis.

In the dosage process, two MFC s control the flow of synthetic air while the o-xylene is pumped into an evaporator. After the evaporated o-xylene is mixed with the air, it is passed to a four-way valve.

The four-way valve represents the first part of the reaction and analysis section. The valve controls the gas flow through the reactor or the bypass. A pipe leads from the valve to a split in the process, with one path leading to the GC and the other leading to the Equilibar back pressure regulator. This way the sample loop of the GC is constantly flushed and does not restrict the gas flow through the plant.

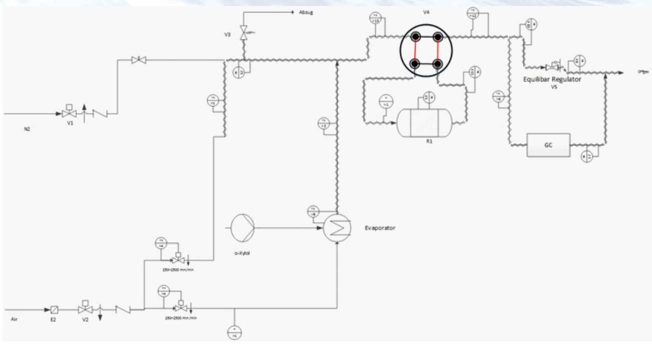

Figure 1: R & I diagram

Figure 2: Equilibar EB1LF installed

Through another fitting downstream, the gases from the GC and the pressure regulator are combined to continue to the flue gas treatment. The flue helps to oxidize all the hydrocarbons into CO2 and H20 to eliminate harmful gasses leaving the process.

After evaporation, all pipes and valves are heated to 250°C or above to make sure that the o-xylene does not condense inside the plant. The main product has a boiling point < 280°C and needs to be in the gas phase for analysis. If the gas is condensed or re-sublimated, it could cause failure by blocking the flow paths in the system and causing down time. The system, therefore, requires a pressure regulator that can withstand temperature cycling and that can be maintained at an elevated temperature.

The figure shows the experimental set-up using the Equilibar EB1LF unit. The regulator is located inside a heated aluminum block that is further insulated with mineral wool. .

Objectives of phthalic anhydride Research

EPhthalic anhydride is the main precursor in the large-scale production of plasticizers, which are used to make plastics and other materials durable and flexible. Plasticizers can be found throughout industry-- in automotive applications, medical devices, electrical cables and more. Their breadth of use underscores the importance of research to find improved manufacturing methods for these compounds.

Research into the partial oxidation of o-xylene has been conducted since the early 1900s. During early stages, a vanadium-oxide catalyst was used and over the years reaction networks have been created while increasing selectivity and activity of the catalyst. As the oxidation reaction is highly exothermic, it is not possible to reach isothermal conditions in a packed bed because this causes hot spots that influence the reaction kinetics. When working below the explosive regime (<1 % Volume oxylene in air) the influence on reaction kinetics is small. As soon as higher o-xylene concentrations are used, severe hot spots are formed.

The University of Stuttgart's research team has found a method where even at higher concentrations of o-xylene, isothermal conditions can be achieved. This allows a reliable prediction of reaction kinetics during partial oxidation of o-xylene in the explosive regime. With these results, it should be possible to describe the reaction under conditions that were, until now, not under investigation. Looking into the future, this development could help industry develop more effective processes in their facilities. .

About the Institute of Chemical Technology at the University of Stuttgart

The University of Stuttgart is a member of the TU9 Association of German Institutes of Technology, with a current enrollment of around 25,000 students. The Institute of Chemical Technology is part of the faculty of chemistry and is actively involved in collaborative endeavors with scientists in other disciplines, including scientists at the nearby Max Planck Institute.

The Institute's research group is responsible for numerous projects in the development of solid catalysts such as zeolites, metal organic frameworks, and supported metal nanoparticles. The group also works on heterogeneous catalysis with a focus on photocatalysis, electrocatalysis, energy-related catalysis, selective (de)hydrogenation and oxidation reactions. Operando techniques in catalysis including solid-state NMR, IR, and XRD are further areas of inquiry.

Stuggart's research team has investigated reaction engineering of selectivity/conversion optimization, space-time yield, photon efficiency maximization, kinetic measurements and basic engineering/scale-up.

Contact Equilibar

Based in North Carolina, Equilibar provides unique and innovative pressure control solutions for applications around the world. . Their back pressure technology is used in a wide array of processes including catalyst, petrochemical, supercritical and other industrial applications. For more information please contact an Equilibar applications specialist at www.equilibar.com or 828.650.6590.

Philipp Eversfield / PhD Student
University of Stuttgart – Institute for Chemical Technology., Pfaffenwaldring 55, 70569 Stuttgart Germany. Mr. Eversfield can be reached by email at philipp.eversfield@itc.uni-stuttgart.de

Tony Tang is a Senior Engineer at Equilibar, LLC, a provider of high precision pressure control solutions. He has worked as an applications and development engineer at Equilibar since 2010. He has received his M.S. and B.S. from North Carolina State University. Mr. Tang can be reached at tonytang@equilibar.com or 828-650-6590.