Hydrogenation reactors learn new tricks

Customized solution

The hydrogenation laboratory of a Swiss pharmaceutical manufacturer found itself constrained by the capabilities of its existing reactors. The complex, prolonged experimentation and testing needed to determine optimal and safe process parameters for new production processes could not be efficiently achieved with the existing reactor setup, mainly due to the lack of process automation. Instead of writing off the investment in the existing reactors, they decided to have SYSTAG upgrade the existing reactors with a flexible and programmable recipe, control and logging system, based on SYSTAG FlexyConcept.





Initial situation

Constant change

Products, markets, feedstocks, equipment, regulations; they all keep changing, and they often lead to new or changed production processes. Laboratories are under constant pressure to validate and optimize new processes and recipes, and provide the production plant with reliable and thoroughly proven parameters for safe operation to produce a high quality product.

Excellent reactors, but not very smart

A Swiss pharma manufacturer was confronted with demands that could not economically be met with the existing - otherwise perfectly satisfactory - hydrogenation reactors in their lab facility. The testing of new processes required more prolonged round-the-clock operation of 4 reactors, with constant adjustment of gas dosing, stirring and heat input based on continuous measurements. Not to mention the need to stay within safe limits to avoid a calamity. With 24/7 attendance and manual logging being prohibitively expensive, it seemed that the only solution was to replace the existing reactors with a fully automated setup with comprehensive data logging. The Chemist Group Leader Hydrogenation asked Swiss laboratory system integrator SYSTAG whether there was an equivalent, but less expensive solution, that did not entail scrapping the 4 reactors.

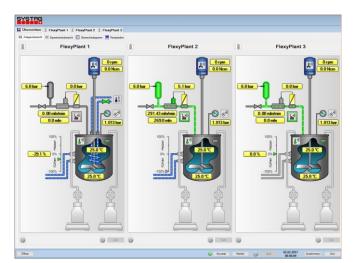


Chart 1: Unser interface-main screen



The solution

Intelligence through upgrade

SYSTAG proposed to upgrade each of the four reactors with a FlexyPAT (process automation technology) control system, a component of SYSTAG's FlexyConcept lab automation platform. SYSTAG Project Leader Erwin Telser: "Apart from installation of the FlexyPAT controllers and retrofitting the reactors with suitable sensors and gauges, there were additional requirements. We needed to develop some custom software algorithms, and the customer also wanted two different measurement techniques for hydrogen consumption, based on pressure differential and the thermal mass flow method. Moreover, they also requested placing a backup controller on permanent standby in the same cabinet as the 4 active control units, for rapid changeover in case of failure. Despite the limited space, we managed to solve this."

There were also downtime constraints for performing the upgrade, as the reactors are in almost constant use. Erwin Telser: "Thanks to good preparation and planning by our team, we were able to install, test and commission the upgrade within just 2 weeks!"

The lab's existing hydrogenation reactors and control devices (pumps, valves, agitators, etc.) are now directly controlled from the user-friendly interface of a single PC workstation running configurable recipe software. Equipment state and process parameters are visualized graphically. All process parameters and measurements are continuously logged by the e-Lab journal. Operations are safeguarded from critical situations around the clock; emergency cooling and/or shutdown are automatically activated if necessary, with designated personnel alerted by e-mail.

≙ æ ≵ ⊫⊾ ®. Ø ⇔ × r	* = = = x		11 M 188 47 1
2 14 10 14 18 17 (00 14) 2 14 10 10 14 18 17	12 14 14 AP AP 1	ने को बने 📫 📑	
\$ 1 +3 1 7 1 4 1 1 1 1 4 1 1			· · · · · · · · · · · · · · · · · · ·
Phase 1 Inertisation	Gas Dosage 1		×
Phase 2 Loop 5x			
* Phase 3	Control:		
A*) Temperature control		i Hold	Stop
Control mode Reactor End value 80.0 *C Ramp time 5.0 min	Control mode:	Bypass	
Batch mode Batch	End value:	50.000 bar	50.000 bar
* Phase 4	Ramp time:		0.0 min
Gas Dosage 1	Gradient:	24.494 bar/min	
Control mode Bypass End value 50.000 bar Ramp time 2.0 min	MFC Constant Flow:	50.00 In/min	
MFC Constan Beset integrat.	Pressure reactor:		1.013 bar
Batch mode Batch	MFC values:		
Phase 5 Actual flow:		0.00 In/min	
L ⁺¹ Stirrer control	Actual volume integrator:		0.00 In
Control mode On End value 500 rpm Bamp time 0.0 min	Total volume integrato	r:	0.00 ln R

Chart 2: Change recipe "on-the-fly"

Outlook

Better lab performance with more safety

A year and a half after the upgrade, the hydrogenation lab continues to meet and even exceed the expectations of the Hydrogenation lab's Group Leader. The lab now has significantly enhanced safety, flexibility and productivity, and the automated e-Lab documentation provides extensive data for improved and easier analysis and process design. Given the proven success of the FlexyPAT hydrogenation reactor upgrades, this customer is now exploring more opportunities for the use of automated Lab Reactors from SYSTAG in their other development laboratories.

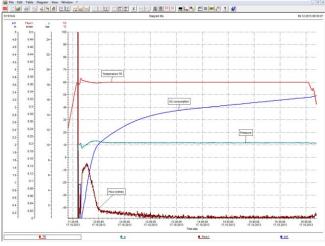


Chart 3: Online trend with gas consumption

Automated sampling system

For improved reaction tracking, the contamination-free sampling system enables automated sampling under pressure of up to 4 samples per reactor.



Chart 4: Sampling system

Functionality

Reactor layout

- 2x 2lt SS316 reactor, up to 100bar
- 2x 500ml SS316 reactor, up to 100bar
- Reactor with el. heating jacket and built in cooling coils, additional cooling coils (cold finger) inside the reactor
- Stirrer motor with gas stirrer
- Automatic sampling system
- Liquid dosing by HPLC pump (shared infrastructure for all reactors)

Temperature control

- Temperature control by Jacket or Reactor mode
- Electronically heated reactors
- Additional internal cooling coil for fast / efficient cooling

Gas Dosing / pressure control system

- Hydrogenation feed by Mass Flow Controller
- Pressure control by Hydrogen
- Constant gas flow control
- Logging of Hydrogen consumption by MFC or pressure difference method
- Automatic gas exchange by inert or active gas

Software Interface

- Single Software platform to control 4 independent reactors with individual functionality
- Recipe control system
- On-line trend and MS-Word based e-lab Journal
- E-Mail alert system
- Configurable emergency alarm concept
- Automatic data transfer to e-Lab Notebook
- Temperature controlled liquid dosing by HPLC pump (shared infrastructure for all reactors)

For more information please contact:

SYSTAG, System Technik AG Mike Mandlehr, Marketing Director Mike.Mandlehr@systag.ch +41 44 704 54 42 systag.ch/lab-reactor-systems



More highlights



ePAT

Automate your jacketed reactors

*e*PAT symbolizes simple but highly efficient laboratory automation for various reactors, so that you can focus on faster, cost-effective and more robust process development. *e*PAT in process development also means seamless data recording, on-line trend with all measurement data - anytime clearly displayed.

FlexyPAT

Automated Lab Reactor

FlexyPAT enables optimal automation of conventional jacketed reactors, whether it is for research and process development or for use in the scale-up lab. The range extends from small volumes up to several litres. For vacuum or pressure applications, the modular FlexyPAT automation technology and PC-based control allow you to reproduce and fully document all chemical processes.



FlexyCUBE

Parallel Synthesis Workstations for R&D

FlexyCUBE is a new concept for the chemical development lab to meet all process optimization needs. FlexyCUBE as Parallel Synthesis Reactor enables the development of chemical processes, APIs or intermediates faster, cheaper and more robust. Up to 6 reactors are monitored and monitored via a PC.

Technical details are subject to change



Copyright by SYSTAG (2005 - 2009, 2010, 2013)

Hydrogenation application

SYSTAG, System Technik AG

Bahnhofstr 76 Tel:+41 44 704 54 54 Email: infos@systag.ch CH-8803 Rüschlikon Fax:+41 44 704 54 55 www.systag.ch

System Technik Deutschland GmbH

Rodheimerstr 63D-61191 RosbachTel:+49 6003 93 50 50Fax:+49 6003 93 50 52Email: infos@systag-deutschland.dewww.systag.ch