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Automation of Batch Synthesis in Laboratory Miniplants

High Reproducibility, Secure and Flexible

In synthesis R&D laboratories automation is still rarely used. If at all, its mostly used for relatively simple, repeated procedures, like liquid handling. But synthesis labs may also profit from advanced automation. Besides of time and work economies the reproducibility and the traceability of automated experiments are invaluable.

Automation in laboratories differs from production automation. While in production uniformity of the educts and the process is ensured, the requirements in the lab are ever changing. As a result, a lab automation system needs a much higher flexibility. This concerns the hardware as well as the software of the system.

Reasons for Synthesis Automation

Several reasons are stated for automation of processes. Regarding the recommendation of R&D laboratories, the relevant arguments differ from other fields of application. Mentioned are mainly:

- Reducing costs
- Gaining experimentation time
- Gaining more information
- Reporting
- Increasing reproducibility
- Security

At the first view, automation produces costs. The work in R&D laboratories requires a high degree of flexibility, the automation has to be designed to assure this flexibility. With automation the work in a R&D laboratory changes, but it is not necessarily less work. Because a automated reaction system is able to run overnight, the time gain is more plausible. As a result, the time and cost arguments are not dominant in R&D laboratories.

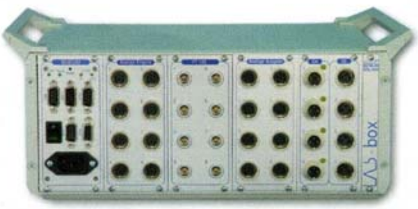

Fig. 1: Process controller with connectors according NAMUR. Connectors for Pt100, thermocouples, analogue inputs and outputs, digital inputs/output and serial interfaces RS232.

Data recording and storage as well as reporting are basic functions of a automation system. Data acquisition is complete and the data may be reanalysed whenever needed.

The most striking argument for automation is the gain in reproducibility. Humans never work as reproducible as machines, if apparatus and educts are the same, the automated reactor system will always produce the same results.

The security is also increased, because the staff will control the process from outside. Therefore the residence time in the dangerous area is significantly reduced. Very dangerous processes or unattended operation must be secured additionally with systems independent from the automation system.

Witch Degree of Automation?

Laboratory automation began with standalone controllers for parameters like temperature. A contact thermometer is a typical example for a basic on/off controller. They are able to keep a parameter at (or nearby) a setpoint without the need for a permanent surveillance.

Modern instruments include digital displays and pushbuttons for changing the setpoints, or they are able to control a couple of different workplaces for parallelisation, but the principle remains the same: control over one parameter without data storage and connection to higher level systems

Nowadays a couple of devices with a higher degree of automation are offered. Most of them are limited to relative simple, repeated procedures, often in parallel. They are derived from heating sys-

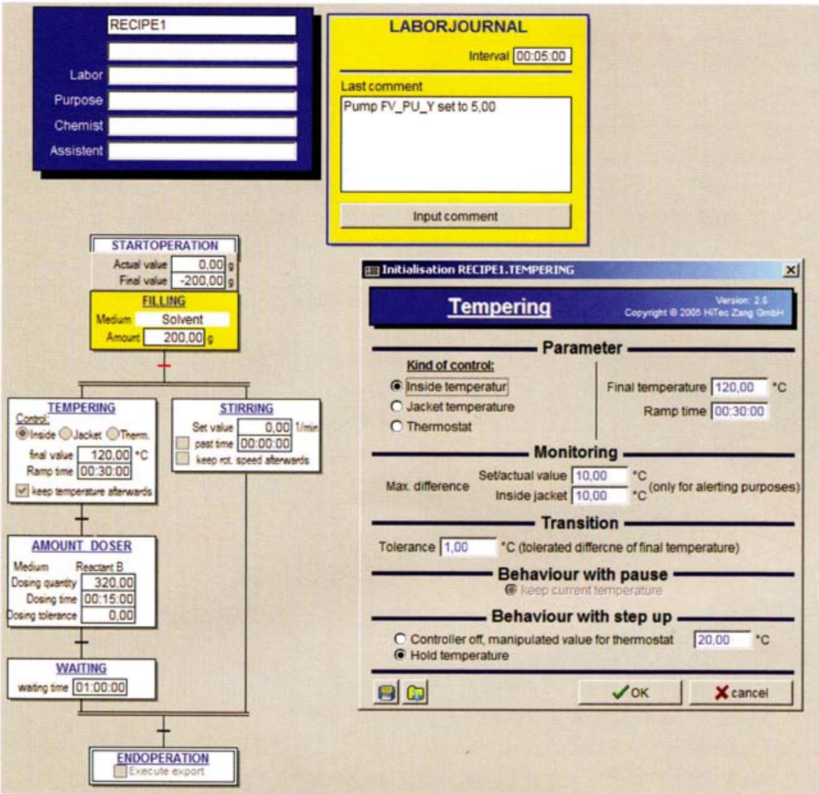

Fig. 2: Example of a simple recipe. Unit operation "Filling" is running, "Tempering" dataset open for editing. The recipe fills the reactor, heats up to 120°C while stirring, doses 320 g of reactant in 15 min., waits 1 hour and finishes afterwards.

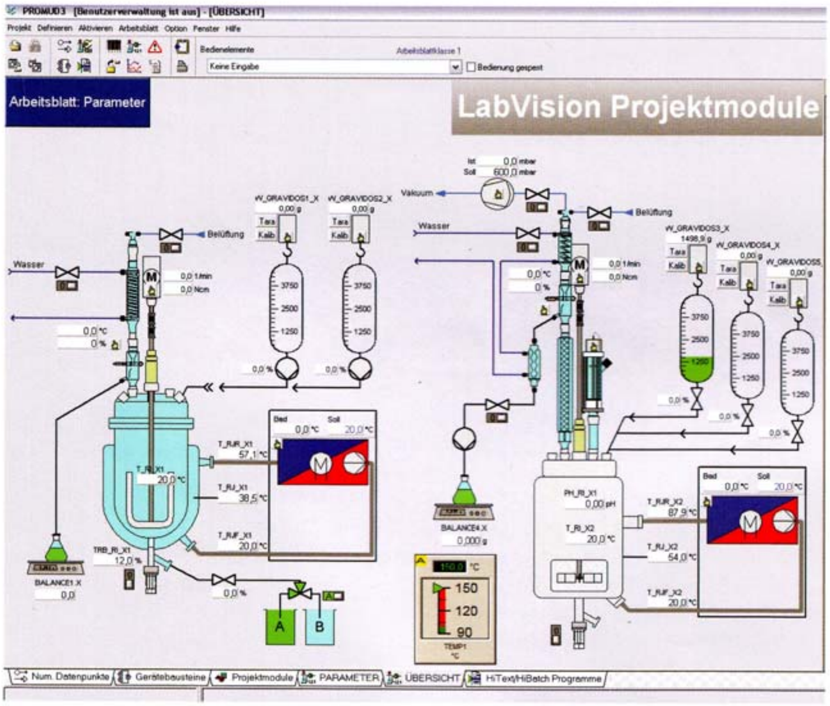

Fig. 3: User interface of an automated laboratory reactor system for polymerisation at low pressure. Equipment: 2 GraviDos gravimetric dosing systems, 1 dosing with balance and pump, SoliDos solids dosing device, distillation, gas dosing and vacuum pump.

tems and are able to stir the reaction mixture. They are useful for the fast optimisation of synthesis parameters, but not for more complex batch syntheses with different process steps.

Automated laboratory reactors offer a higher degree of flexibility. They include at least individual temperature control for each reactor, dosing possibilities and additional sensor inputs (pH, pressure...).

The term "automated laboratory reactor" (ALR) is diffuse, it may be a relatively simple device as well as a customised miniplant with serial reactors. Besides the complexity the main differentiator is: Is it limited to original manufacturer equipment or is it open for other devices. The following text treats ALRs with open architecture.

Automation Hardware

The demand for a wide working range together with a high precision calls for a high resolution measurement system. Hard-wired switch cabinets shall be replaced by smaller, standardised units with non-interchangeable connectors for the different signals. The NAMUR workgroup for measurement and control in the German chemical industry defined a standard for such R&D automation systems. The standard defines connectors for the commonly used process signals.

A remark to automation system based on computers alone: Computers aren't secure automation systems. Each process with a certain potential for danger shall be automated by reliable controllers, the computer is useful as a mass storage device and a human-machine interface that communicates with the controller.

Recipe Control – The Unit Operation Concept

While continuous processes only need a permanent control regarding fixed setpoints, batch operation is carried out as a sequence of actions depending on changing setpoints and actual values. The automation solution for those processes is called recipe control. A recipe is a sequence of actions leading from the educts to the products. These actions are organised as so called "Unit Operations". Unit Operations are preprogrammed steps representing basic laboratory operations, like dosing, tempering, stirring, distillation, crystallisation and others. These unit operations are combined in series or parallel with a graphic editor. Connected to build a recipe they represent and document a distinct reaction process.

Unit Operations may be used several times in one recipe. After compiling, the desired values of temperatures, heating rates, dosing rates, pressures and so on are assigned to the recipe. Recipe and parameters are stored independently

Fig. 4: Automated laboratory reactor with thermostat,. 5 liquid and one solids dosing, vacuum distillation with distillate balance and separate bottling of the fractions, pH control, liquid phase interface recognition and separate bottling of the phases, MFC, sample collector.

from each other. So, different sets of parameters (called Data Sets) may be stored for each recipe. Decisions, conditional branches or counter loops are also realised with graphic elements. A Unit Operation library fits all the requirements of the daily work in the laboratory. Customised Unit Operations may be programmed if required and incorporated in the library.

On parallel reactor systems a recipe can be assigned to different reactors. Each reactor may work with different reaction parameters or with different recipes at one time.

Process Visualisation

The progress of the recipe may be monitored in the recipe plan or on the plant operation console. This user interface is designed for manual control, e.g. when starting the plant, and to monitor the actual plant status and measured values.

This sheet contains usually only the essential information. Additional worksheets may be integrated for parametrising devices, administrate warning and alarm limit or for the control of external devices.

Example of a Laboratory Plant

The possibilities of recipe controlled reactor systems are demonstrated by the following laboratory pilot plant. Together with a similar parallel reactor system with four reactors the system is used for synthesis optimisation and scale up experiments.

Conclusions

Synthesis automation is a important prerequisite for the reliability of experiments. The work in a R&D laboratory needs special automation equipment designed for high measurement resolution and flexibility against changes.

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