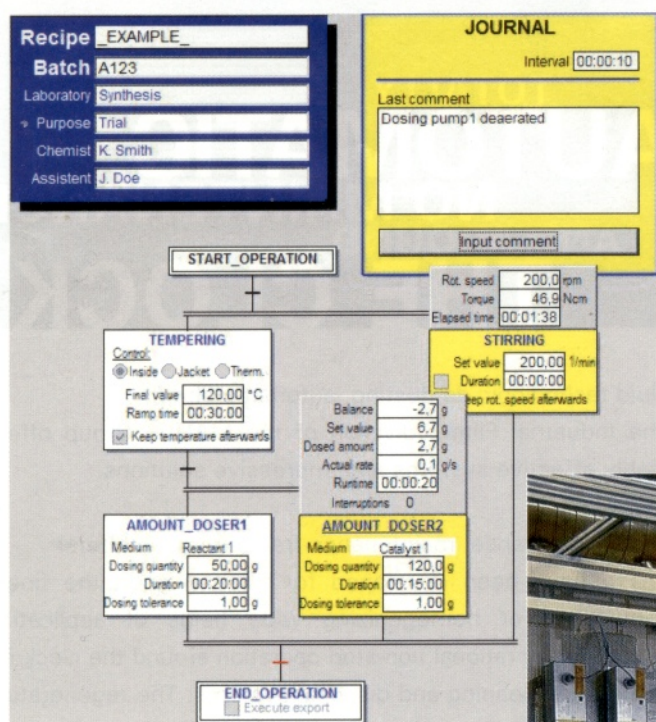


# Automated laboratory

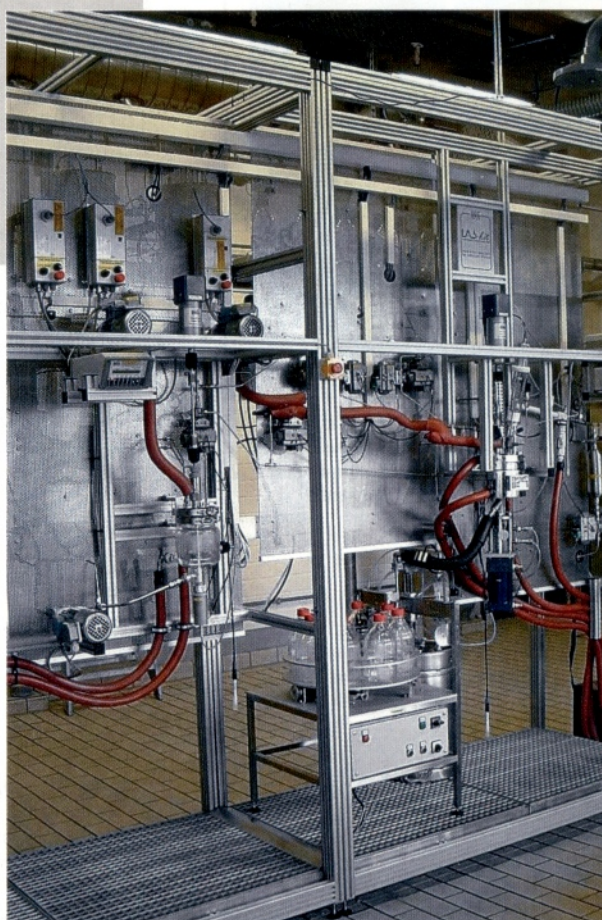
## Recipe control for research reactors

Recipe control is a prerequisite for efficient process control and easy handling of batch operations in R&D. At the same time, manual operation must still be possible, however. The functionality of a recipe control software program specially designed for R&D is described here, taking the example of LabVision with HiBatch, the Namur NE 33 compliant recipe control system.



Example of a simple recipe: the unit operations are picked from the library with the mouse, placed on the editor and connected

Pilot plant for polymerisation under pressure



The automation of reaction plants is a prerequisite for safe and reproducible operation. Data from automated research reactors can be used to scale up the process. Automated laboratory equipment nevertheless has to be adapted to the specific requirements of the laboratory. Firstly, the staff has no specific knowledge of process automation. Secondly, R&D demands a high degree of flexibility when it comes to handling the system. Automation systems developed for production are not able to deliver this. Namur Worksheet NA 27 states that it should be permissible for research and development parameters, and even the system configuration, to change during operation and that the process control system should be designed to enable staff without automation expertise to perform these modifications.

## Recipe editor

Calls for easy handling and high flexibility are satisfied by the HiBatch recipe control system for batch plants. The process is controlled by a sequence of so-called unit operations according to Namur Recommendation NE 33. Unit operations are basic laboratory operations like dosing, tempering, stirring, distillation, crystallisation, etc. They are combined either serially or in parallel in a graphic editor. When connected together to build a recipe, they represent and document a distinct reaction process. Laboratory staff are thus able to program a complex synthesis procedure in a few minutes. The recipe, for example, heats the reactor at a predefined rate while simultaneously stirring. Two reactants are then dosed. The recipe is complete when dosing has finished. Every 10 minutes or on request the actual values are entered in the report. Unit operations can also be used several times in one recipe. After the recipe has been written, the desired values for the temperatures, heating rates, dosing rates, pressures and so on are assigned to it. The recipe and parameters are stored separately from one another. Different sets of parameters can therefore be stored for each recipe. If-then decisions, conditional branches and counter-loops are likewise implemented with graphical elements. A unit operation library meets all the requirements of daily work in the laboratory. Customised unit operations can be programmed if required and incorporated in the library.

## Plant visualisation

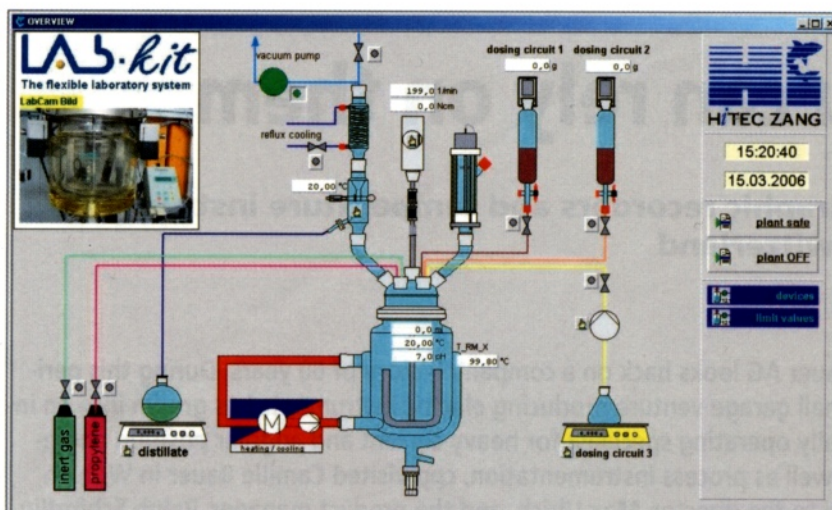
The progress of the recipe can be monitored in the recipe plan or on the plant operation console. This user interface is designed for manual control, e.g. when starting the plant, as well as for monitoring the actual plant status and measured values. In parallel reactor systems, one recipe can be assigned to any number of reactors. Each reactor can work with different reaction parameters or different recipes at the same time. The process control hardware is the HiTec-Zang LAB-manager. LAB-manager is a stand-alone programmable controller characterised by a high level of reliability.



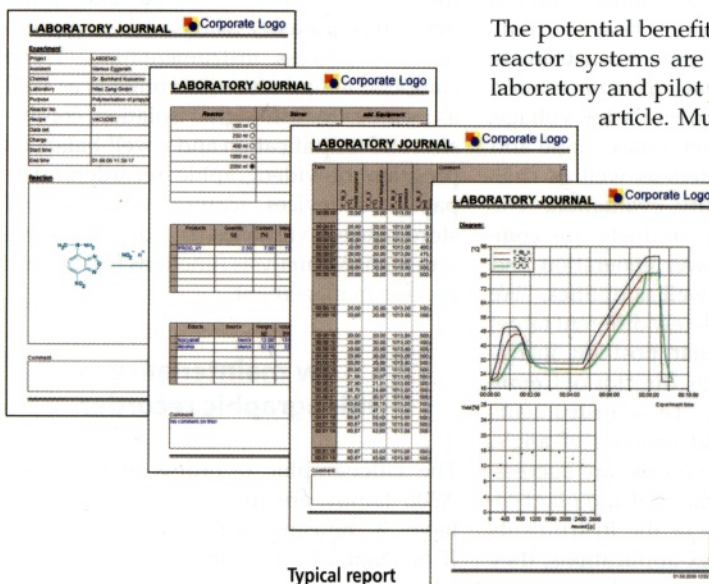
This compact device contains the complete electronics of a conventional control cabinet and can be mounted close to the reactor or even in the fume hood. LAB-manager was developed in close collaboration with R&D laboratories on the basis of the recommendations contained in Namur NE 28. Connections to laboratory equipment are realised with standardised plug connectors. Almost any sensor, actuator or laboratory device can be connected using these plugs. A PC with Windows XP and LabVision process control software is supplied for visualisation and handling and as a mass storage and data analysis device. This task sharing principle combines the performance of a PC with the reliability of an automation system. The LabVision KalDas software module supports reaction calorimetry. Heat flow and balance calculations can be carried out. Reaction calorimeters based on LabVision are not restricted to calorific experiments but hold the full potential of an automated laboratory reactor.

## Instrumentation

HiTec-Zang additionally offers a wide range of equipment for laboratory reactors. GraviDos, the gravimetric liquid dosing system, consists of a sample vessel suspended on a load cell and a solenoid valve. Due to its simple setup, it is inexpensive and robust compared to systems comprised of a balance and a pump. ViskoPakt is a series of measurement stirrers with a maximum torque value between 10 and 110 Ncm. The precision torque measurement stirrers in the ViskoPakt rheo series feature a high torque resolution – even fractions of the water viscosity are resolved. Both series are also exceptionally compact. Reliable and precise dosing of solids is one of the most challenging problems in a laboratory. The SoliDos solids dosing device doses powders and granulates reliably even in condensing atmospheres. Liquid phases can be isolated, for example when draining the reactor, with the aid of the PhaDec phase interface detector. PhaDec differentiates between the heat conductances of the liquids. As a result, the phases need be neither con-



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



Typical report

ducting nor transparent. Dosing small volumes or automatic sampling from the reactor are possible with the AutoSam liquid handling system. AutoSam is available for sample volumes between 1 and 2000 ml. Other options, such as sample preparation, in-situ molecule spectroscopy and product work-up, can be used to construct complex, automatic laboratory or pilot plants, including the complete range for R&D.

The potential benefits for recipe controlled reactor systems are demonstrated by the laboratory and pilot plant described in this article. Multiple reactor systems

with sequential reactions are the most complex plants in batch or semi-batch operations. In the pilot plant illustrated here, 13 dosing systems operate on two cascaded reactors. For some of these systems the reactant vessel, the pump and the piping needed to be heated. The plant also integrates the

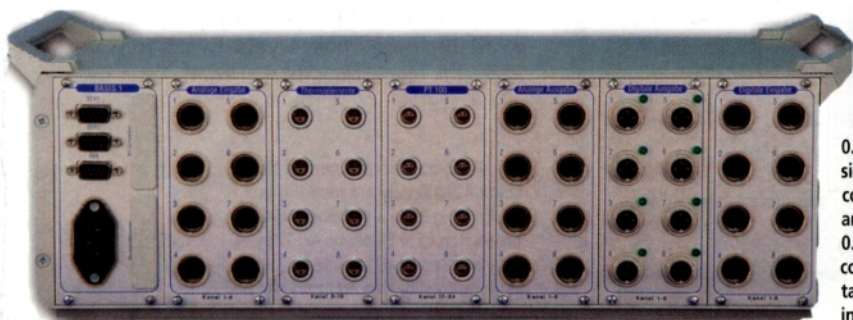
following functions: inert gas supply with three pressure levels, Coriolis mass flow measurement, distillation under increased and reduced pressure, pressure and heat-resistant endoscope camera for the main reactor and pH control. The plant automatically executes up to eight subsequent reactions and collects the entire product in the sampler. It is cleaned – likewise automatically – after each reaction.

The large pipeline network has to be shared by different unit operations. Central management of the resources was therefore essential. As soon as a unit operation is requested, the pipework parts needed to implement it are assigned without any manual intervention. All other unit operations then wait until the prioritised operation releases the pipework sections concerned again.

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LAB-manager with serial interfaces for laboratory equipment, 0...10 V or 0...20 mA analogue signals, thermocouples, Pt100 resistance thermometers, 0...10 V/0...20 mA control outputs, digital outputs, digital inputs