Reaction Calorimetry

H·E·L Hazard Evaluation Laboratory

SIMULAR

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A reaction calorimeter is a tool that allows for the precise simulation and thermal study of a chemistry and associated plant, typically at the one litre scale. Subsequently, important safety, viability and optimisation data can be obtained prior to scale up.

Safe process scale-up

The most common application of a reaction calorimeter is the determination of heat released during a process. From this the subsequent cooling duty required for scale up can be calculated. In addition the potential adiabatic temperature rise can be calculated. Combined with other techniques, a basis for safety can therefore be determined.

Further information available includes the determination of reagent accumulation and the impact of variables such as agitation and catalysis upon the reaction rate. By changing feed rates and batch temperature, the optimum operating conditions for both efficiency and product quality, can be determined.

Flexibility without compromise

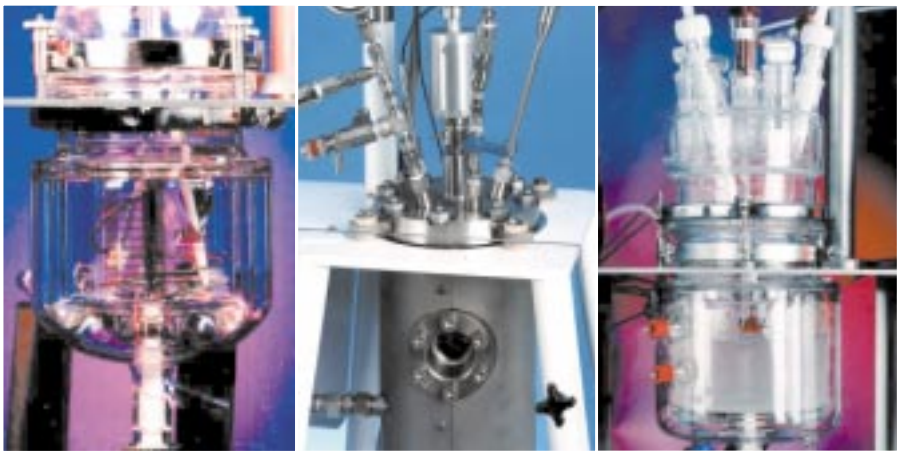
SIMULAR is built around our unique modular electronics and the latest in-house developed Windows software, to provide you with a tool that meets your requirements, both now, and into the future.

Select the components that most closely match your plant conditions, then combine them to have a dedicated, simple-to-operate tool that simulates your plant and process. This includes temperature range, agitation, speed, reagent feed rate and even reactor geometry - *find out why we offer the best, most flexible, reaction calorimeter for your application.*

Multiple calorimetric methods

Unlike most leading reaction calorimeters, SIMULAR is designed to be used by research chemists and safety specialists alike. This is made possible by power compensation calorimetry, unique to the SIMULAR. This allows accurate determination of heat release without the need for time-consuming calibration or complex calculation procedures.

For more conservative users, the classical heat flow calorimetry method is also supported as standard.



...an essential tool for Process R&D

Specify SIMULAR around your chemistry...

Turn-key systems

For safe and efficient scale-up, it is essential that your tools simulate your plant as closely as possible.
In addition it is important that as much information and insight about the process be obtained.
As such the options, upgrades and flexibility offered by the SIMULAR are almost limitless, and we put them all together in a turn-key and dedicated solution.

Reactors

The standard reactor is a 1 litre glass, atmospheric pressure system. Optional volumes range from 0.2 to 20 litre in glass, stainless steel or resistant alloy, with pressure ratings up to 200 bar. Specialist geometries and custom designed vessels are available to simulate real plant conditions.

Feeds

Reagent Feeds - Gas, liquid and solids feeds are all available using a variety of application dependent techniques. Standard liquid feeds are pump and balance combinations, but options include syringe pumps for low flow rate feeds and pressurised vessels for faster rates and/or highly volatile reagents.

Gas feeds can be controlled via constant reactor pressure, bottle/balance combinations or even mass flow control. Finally automated solids addition is also made possible using a screw feed system developed exclusively by HEL.

Operating Range

Pressure - HEL offer a range of options for the study of reactions under pressure including 6 and 12 bar glass reactors, 60 bar stainless steel with a number of other alloys and specialist material options. We even produce systems that operate at up to 200 bar reactor pressure.

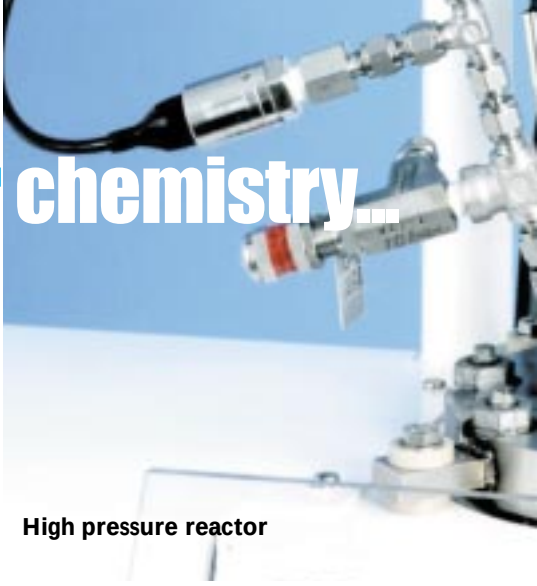
Temperature - By using commercially available heater/chiller systems, we can offer you the temperature range you need, between -80 and over 350°C, with precision of better than +/-0.1°C. These systems also offer a small foot print and emergency cooling is available when required.

Controls & Sensors

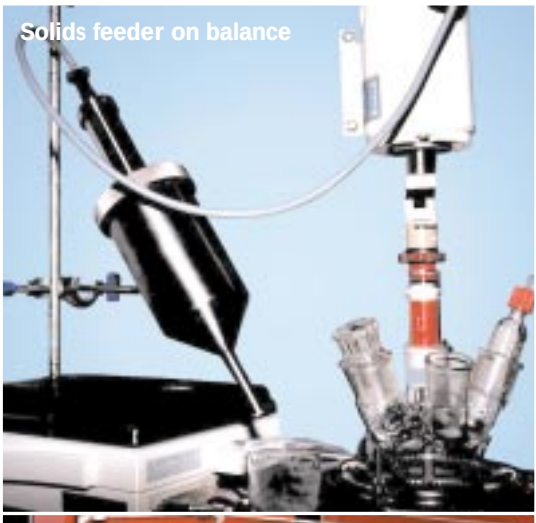
Software and electronics allow a variety of inputs to be logged and if necessary, used for feedback control. This includes pH, turbidity, conductivity, FTIR, particle size etc.

Sampling

HEL have developed tools for automatic sampling and dilution during chemical reactions. A portable sampler, software driven, is available as an option.

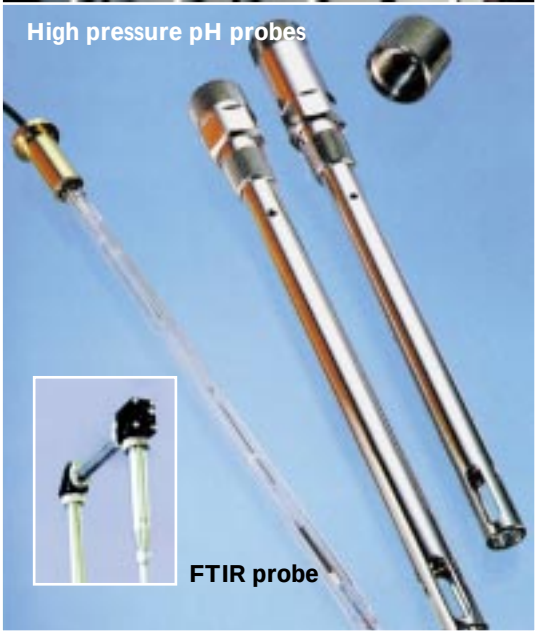


High pressure reactor



Solids feeder on balance

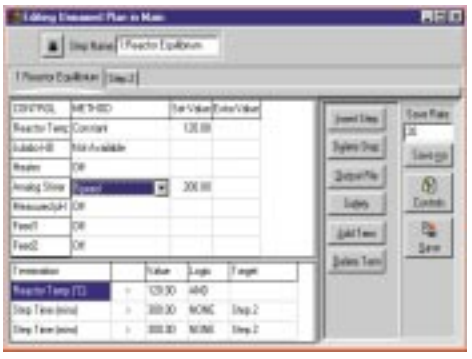
Four feed pumps



High pressure pH probes

FTIR probe

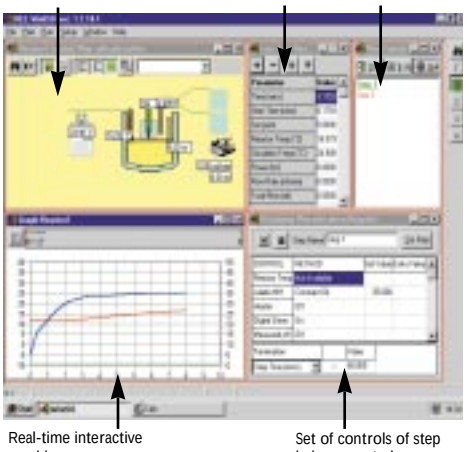
Graphical overview of a plan window



Mimic with display of key data

Numerical data display

List of experimental steps



Real-time interactive graphics

Set of controls of step being executed

The PC software package, makes operation simple and easy to survey.

Graphical overview of iQ

Calorimetric methods

Only SIMULAR offers you three different methods of reaction calorimetry.

HEAT FLOW is the most widely used method, and is standard on all SIMULAR systems. Heat generated by the reaction is removed by the oil jacket, and by using precise calibration heaters, pre and post reaction, the energy evolved can be determined.

REFLUX CALORIMETRY allows for the determination of heat flow even during large changes in reflux temperature and reactant volume.

POWER COMPENSATION is a method that bypasses the need for the lengthy calibration steps involved in heat flow.

ADVANTAGES:

- ▲ Reduced experiment time
- ▲ Intuitive 'live' results
- ▲ Improved temperature control.

Running experiments

winISO has been developed in-house to provide a logical and simple interface for describing multiple step processes.

Using input screens, arranged in a series of “recipe cards”, the operator simply indicates the required set point for each peripheral. Each step is then terminated on any selected conditions, (eg temperature, time, feeds completed), or by a series of logic statements.

During operation, all conditions are displayed on a mimic, numerically and on a trend graph. All steps can also be modified during operation. Changes are fully documented in the output file.

Information obtained

All process variables such as pressure, temperature, pH, feed rate etc, can be stored on disc for off-line analysis as well as being displayed in real-time.

Formal calorimetric data is obtained by off-line calculation using our proprietary iQ software package. This includes reaction enthalpy, heat release rate, heat transfer rate etc.

WHY SIMULAR?

Flexible

Whether it is the wide temperature options, multiple reagent feeds or reactor specifications, no other system covers such a wide range of chemical conditions and variables.

Powerful

SIMULAR is the only reaction calorimeter to offer Heat Flow, Power Compensation and Reflux Calorimetric techniques. Each of which offers specific advantages depending on your chemistry, and all are available regardless of reactor specification.

Expandable

The modularity of SIMULAR systems allows you to add on to your system - feeds, sensors etc - in the future simply and without major cost implications.

Ease of ownership

The system will fit into existing fume hoods and usually needs only existing power supplies. We also have the most innovative and user friendly software, and use the standard laboratory components that you are already familiar with.

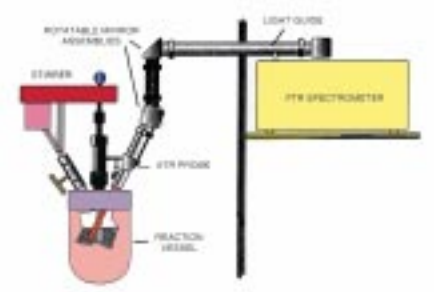
DON’T CONFIGURE YOUR CHEMISTRY TO SUIT YOUR EQUIPMENT - SELECT THE SIMULAR TO SUIT YOUR CHEMISTRY.

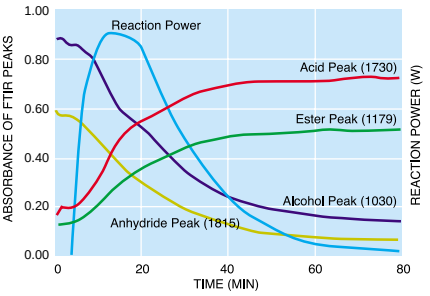
Process Optimisation
Yield, batch time

Optimisation of chemical reactions – to maximise yield, minimise side product etc – sometimes needs complex combinations of measurements and control. SIMULAR is uniquely flexible in being able to take inputs from a range of sensors – FTIR, turbidity, particle sizing devices – and then allowing the data to be used to gain a better understanding of the process.

The diagram shows the simultaneous use of FTIR and calorimetry – the appearance of product and disappearance of reactants, seen in the IR spectra, is supplemented by heat flow data.

SIMULAR is unique in being able to combine different sensor inputs and allow adjustment of control parameters to influence the reaction. Control based on model predictions is also possible – the model can run in parallel with the SIMULAR software.



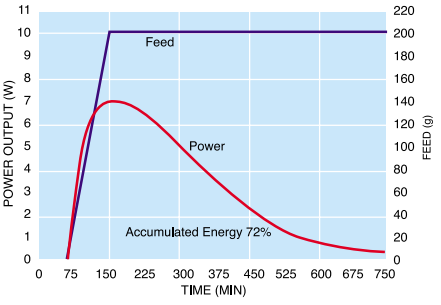


Process Safety
Reagent accumulation

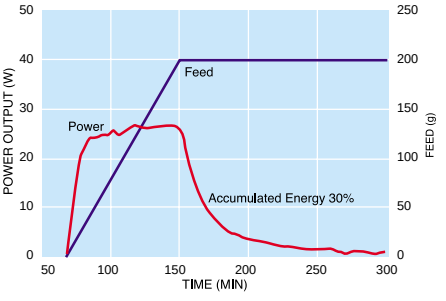
Most reactions are now run in a semi-batch manner: one or more reagents are held back and then dosed at a controlled rate. If the reaction rate is slow at the selected operating temperature, the dosed component can accumulate in the reactor – the amount of accumulation being a function of the feed rate.

The example shows a reaction where over 70% of the dosed chemical has accumulated. By introducing a catalyst, this is immediately reduced to 30%.

SIMULAR data can be used to estimate the accumulation and evaluate the different ways in which it can be reduced. The influence of a catalyst on the reaction is illustrated below.

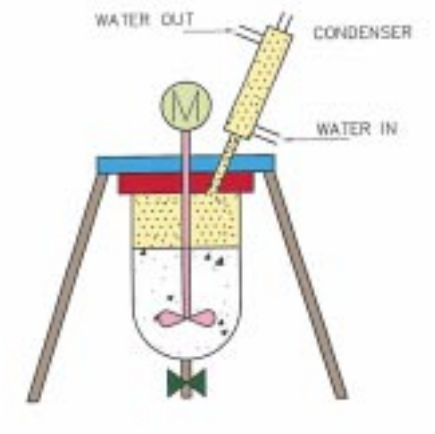


UNCATALYSED OPERATION



INFLUENCE OF CATALYST

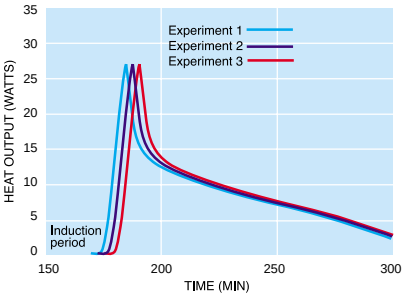
Scale-up Data
Heat transfer, cooling duty

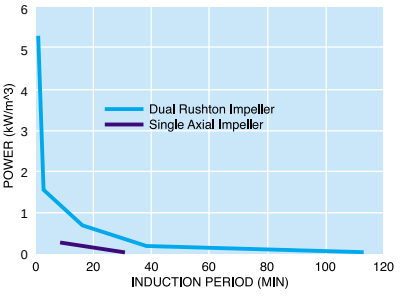


Chemical engineering information necessary for the scale-up of reactions is readily obtained from the SIMULAR. This includes the cooling duty (Q) for either jacketed reactors or reflux condensers and heat transfer rate (UA).

For multi-phase reactions where mixing is crucial, aspects of agitation can also be studied.

Here is a case where a reaction involving three liquid phases was performed under reflux. Not only did we obtain the condenser duty but also the influence of agitation on the induction time.





Thermal Safety Parameters

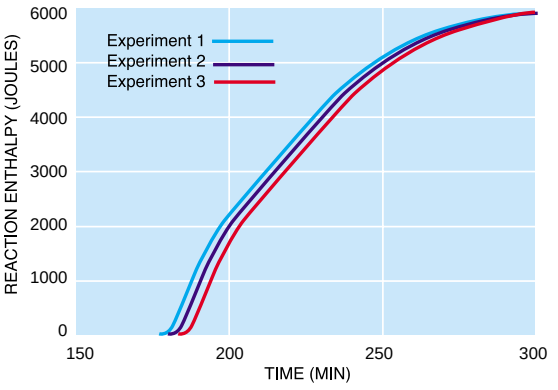
Reaction heat, potential temperature rise

Reaction calorimetry can provide the total heat evolved by the process being evaluated, $\triangle H_r$ in Joules per gramme of mixture. This can then be converted into the reaction enthalpy.

Knowledge of the total heat output can be used to predict the potential temperature rise, $\triangle T_{ad}$, under adiabatic conditions.

The specific heat of the reacting mixture, Cp, can also be estimated using SIMULAR.

Knowledge of the maximum temperature can be used to assess the potential hazard – for example it can be compared with the decomposition temperature of a component in the mixture.

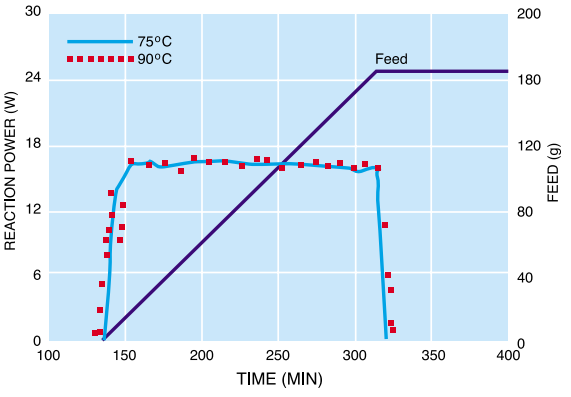


Reaction Kinetics

Feed rate or kinetically controlled reaction

Knowledge of the heat output rate, as a function of time, is a direct reflection of the global kinetics and thermodynamics. This information can be used to quickly develop detailed thermo-kinetic mathematical models of reactions.

If this level of sophistication is not required, the data can without any detailed evaluation reveal key features such as a feed rate or kinetically controlled reaction. The attached data shows a classic dose controlled reaction, revealed both by the sharp rise and fall of heat output with feed and by the lack of dependence on temperature.

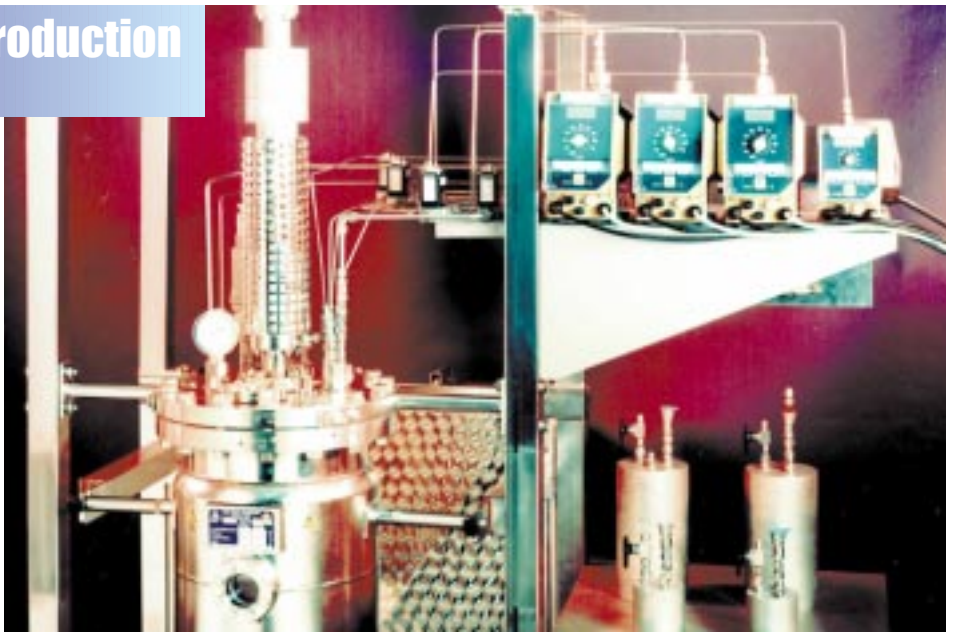


Regulatory Compliant Production

FDA compliance

SIMULAR can be used as a small scale sample production unit. As a highly controlled reactor that keeps a complete record of the process recipe and all process data during the run, it is in keeping with the very best industrial production plant.

If necessary, we can also supply versions of winISO that meet the FDA requirement CFR21 Rule II. This relates to limiting the access to SIMULAR to authorised staff and assuring integrity of the data generated.



Hazard Evaluation Laboratory

HEL offer a comprehensive range of products and services for the modern process chemistry and process safety laboratory. Our computer controlled reactor systems and calorimeters are designed for the safe and efficient development and optimisation of chemical processes. The acknowledged technical strengths of HEL are complimented by Consultancy Services that include specialist testing and advice on a confidential basis.

Consultancy & Testing Services

Process & Reaction Hazard Evaluation
Process Research and Development
Reactor Relief Systems/Vent Sizing
Fire & Explosion Testing
Continuing Education
Process Safety Consultancy

Other Products

auto-MATE — Parallel Synthesis: Miniature Multiple Reactor Systems

AUTO-LAB — Automated Reactor and General Control Systems

AUTO-LAB junior — Automated Batch Reactors

Chem-SCAN — Rapid Chemical Reaction Scanning

Duet — Parallel Synthesis Workstation

PHI-TEC — Adiabatic Calorimetry

TS^{U} — Thermal Screening

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