Fauske & Associates Brings ChemiSens to North America

By Jim Burelbach, Ph.D.

"No, it's not a new washing machine," I replied. My son was examining the ChemiSens fliers on the back seat of my car, and he suspected there was a surprise anniversary gift in the works. This struck me as a reasonable conclusion, given the practical compact design of the CPA202 with its stainless steel cabinet and back-lit viewing window (Fig. 1). OK, then what is it?

The CPA, or Chemical Process Analyzer, brings Reaction Calorimetry to the next level. It is a complete, pre-calibrated, precision tool for analyzing chemical processes. The CPA202 retains the versatility of a laboratory reactor yet facilitates all the techniques and measurements that are essential to effective process development. The absolute heat production from the process is presented on-line in real time without subjective interpretations of calibration pulses or unknown baselines. Everything from stirrer torque to condenser power is accounted for to give ***the truest measure of heat flow***.

Figure 1.
The CPA202
Thermostat footprint is only 40 cm by 60 cm.

Reactor Specifications

The heart of the CPA system is the reactor. The standard reactor (Fig. 2) is good from vacuum to 20 bar (290 psi) and -50°C to 200°C, with power resolution to 10 mW and double glass sidewalls so the reaction mixture can be directly observed. Reactor volume is 250 ml, with a usable (and continuously variable) volume from 10 to 180 ml. For high-pressure applications an available all-metal reactor is good to 100 bar (1450 psi) and 250°C, with a sealed magnetic stirrer drive to ensure leak-free operation. There is also a new HighSens reactor design that gives enhanced sensitivity (say for crystallization, adsorption/desorption, miscellization, etc.), achieving power resolution down to 0.1 mW. Operating limits for the HighSens reactor are up to 10 bar (145 psi) and from -40°C to 150°C.

Figure 2.
Standard reactor
(FV to 20 bar).

Stirring is controlled (and variable) between 50 and 2000 rpm with a variety of impeller designs and a baffle insert. Ports are provided in the lid and the base for auxiliary probes such as pressure, pH, IR, UV, gas flow meters, etc. Other auxiliary devices include a fixed basket for solid catalysts, ion exchange resins, etc. During operation the reactor is positioned in a precisely controlled thermostatic "bath," which also acts as a safety shield.

The Chemical Process Analyzer gives you the "True Heat Flow" Difference

The CPA202 from ChemiSens works on a unique principal called "True Heat Flow" that makes the CPA202 significantly different from an ordinary heat flow calorimeter. In the CPA202 the heat flow from the reaction process is directly measured using a constant-area thermopile comprising hundreds of temperature sensors. This "True Heat Flow transducer" is ***pre-calibrated at the factory - no additional calibrations are required***. All of the heat flow occurs at the well-defined bottom of the reactor (the rest of the reactor being insulated). Unlike a conventional reaction calorimeter, the measured heat flow is not sensitive to changes in heat transfer coefficient or wetted heat transfer area. Also, there is no error introduced by asymmetry in the liquid surface (say due to vortex distortion by instrument probes). This means that accurate measurement of evolved (or consumed) ***power is directly available on-line*** without any tedious calibration or post processing.

The temperature of the process reactants is controlled using a sophisticated Peltier element which acts as a reversible heat pump to transfer heat to (or from) the thermostating liquid that is maintained at the same temperature as the process reactants. The True Heat Flow transducer is sandwiched between the Peltier element and the bottom of the reactor vessel. The thermostatic bath provides a large controlled heat sink for thermal stability and safety.

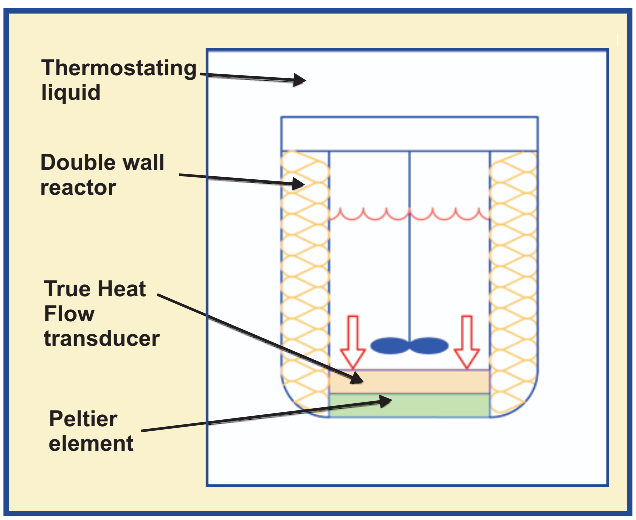

Figure 3.
Schematic of True Heat Flow design.

Dosing Made Simple

In most reactors the dosing of chemicals causes a thermal disturbance in the system, which needs to be compensated for in the system heat flow balance. However, the thermostatic bath of the CPA202 makes it ***convenient to thermally equilibrate process fluid streams*** before they enter the reactor vessel, eliminating transient thermal effects and simplifying data interpretation. For example, a coil of injection line tubing is typically submerged in the thermostat fluid so that when the dosed fluid enters the reactor it is already at the reactor temperature.

The CPA reactor accepts a number of simultaneous lines for controlled addition of solid, liquid, or gas. A companion Dosing Controller (VRC202) provides a universal interface with dosing devices (and other sensors). Thus, dosing can be dynamically controlled based on measured quantities such as reactor pressure, mass flow rate, or the reading from an electronic balance.

A particularly convenient means for controlled liquid injection is the optional MSC202 dosing syringe (Fig. 4). Each device is designed to inject up to 50 ml against up to 100 bar (1450 psi) and gives low controlled flow rates (from 0.001 to 10 ml/min) that are pulse free, accurate, and stable. The syringe has a fixed seal through which the piston is driven (so there is an annular gap between the piston and cylinder). This intelligent design simplifies cleaning and maintenance, since the syringe can be easily flushed using valves on either end of the cylinder body. The motor drive is protected by front and back limit switches, and the control system is tied into the automatic safety shutdown system.

Figure 4.
MSC202 precision drive unit
for Motorized Syringe Control.

Dosing from multiple syringes can be prescribed ahead of time, or controlled as the output from a user-specified control loop, say to maintain a process variable such as True Power at a specified value. This option is particularly intriguing for those interested in ***on-line process optimization*** (see ProFind software discussion below).

Easy Solids Addition

Dosing of solids is accomplished by either batch-wise or continuous operation. The batch injector (Fig. 5) is particularly ingenious, for it ***allows a solid charge to reach thermal equilibrium before being remotely released into the bulk liquid***. The device is mounted beneath the reactor lid and is opened using a slight internal pressure. The loading chamber can be inerted, thus avoiding contact with oxygen or moisture. A continuous solids dosing device is also available. For continuous dosing the solid is gravity fed through a rotating disk "valve" which turns at a pre-programmed speed. In the standard reactor the mixing of the reactor contents can be visually observed.

Figure 5. Batch injector for solids, pastes, and powders.

Cooling Systems

The Peltier element has a cooling (or heating) capacity of approximately 30 Watts. For special applications additional instrumented cooling capacity can be achieved using an optional internal cooling coil (Fig. 6). A fully instrumented overhead condenser can also be added to the system to allow for operation under reflux conditions.

Safety by Design

The CPA202 has a variety of active and passive safety features. The 13 liter volume of the thermostatic bath surrounds the reactor, providing a large heat sink that can be supplemented by the above-mentioned internal cooling coil. The reactant volume

Figure 6. Internal cooling coil.

is typically in the range from 10 to 180 ml, substantially smaller (and thus inherently safer) than most traditional reaction calorimeters now in service. Keeping the quantity of chemicals to a minimum while still simulating the chemical process naturally means less waste, less potential for exposure, and less risk from an uncontrolled reaction.

The CPA 202 has safety instrumentation that automatically stops all dosing and maximizes cooling power if user-specified temperature or reactor power limits are reached. In the event of a communications or computer failure, a separate "watch dog" circuit initiates emergency cooling, and a "panic button" is available to manually activate maximum cooling.

Software Operations

The CPA202 can operate in a variety of "thermal modes," including isothermal, isoperibolic, adiabatic (up to 4°C/min), and scanning mode (up to +/- 2°C/min). The system is run using the ChemiCall application on a PC (desktop or laptop), and a network interface between the PC, thermostat, and reactor is provided by a standalone CPA202 control unit (Fig. 7). A separate VRC202 dosing controller interfaces with almost any type of syringe pump, gas flow regulator, electronic balance, condenser, etc., and is also used for auxiliary sensors such as pressure and pH. All graphic captures (data plots), operator notes, and event logs are stored for later use in the ChemiCall report generator. Raw data and the complete report can be exported to other standard programs such as Excel and Word

Included ProFind software provides an additional graphic interface for advanced automated operation. ProFind allows you to automate experiments by building a procedural workflow chart using intuitive drag-and-drop building blocks. Sophisticated conditionals, parallel execution, and nested parallel processes are possible. The CPA202 can provide up to 50 measured parameters, most of which can be used on-line for conditional logic in automated pre-programmed experiments. Thus, for example, the automated on-line control of dosing profiles is possible, and a reaction process can be controlled so it follows an "ideal accumulation profile" based on continuous determination of the actual thermal conversion. This application is nicely illustrated in a recent NATAS publication [1].

Final Words

Fauske & Associates is extremely pleased to represent ChemiSens in North America. I recently visited the ChemiSens offices in Lund, Sweden, and was very impressed with the quality of their products and their applied knowledge of reaction calorimetry (visit www.chemisens.com). Interestingly, their first reaction calorimeter was built almost 30 years ago, about the same time that Fauske & Associates went into business. By the time you read this we should have received our own CPA202 unit, which will be available for demonstration at our laboratory in Burr Ridge, Illinois (outside Chicago). We welcome you to visit us and see this great instrument for yourself!

And regarding the anniversary gift, I went with the barbeque grill.

Selected References

1. Lamanna, P., Nilsson, H., and Reuse, P., On-Line Reaction Calorimetry Optimization of Safety Parameters, 35th Annual Conference of the North American Thermal Analysis Society (NATAS), East Lansing, Michigan, 2007.
2. Widell, R., and Karlsson, H. T., Autocatalytic Behavior in Esterification between Anhydrides and Alcohols, Thermochimica Acta 447 (2006) 57-63.
3. Zogg, A., Stoessel, F., Fischer, U., and Hungerbuhler, K., Isothermal Reaction Calorimetry as a Tool for Kinetic Analysis, Thermochimica Acta 419 (2004) 1-17.

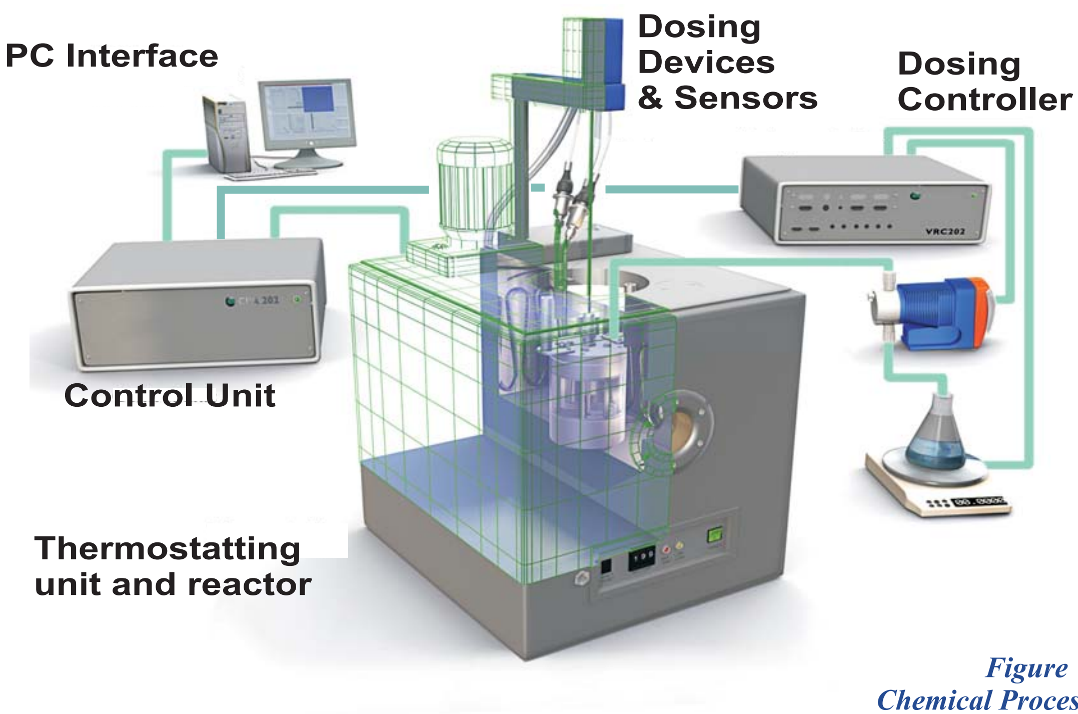

Figure 7.
Chemical Process Analzyer CPA202 and Accessories