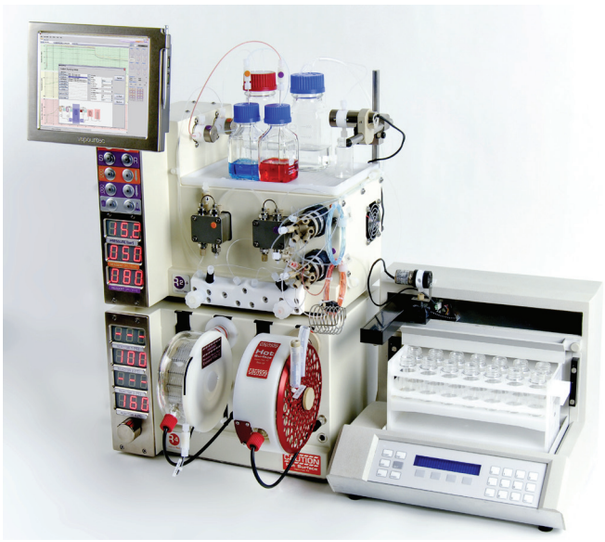
Rapid, Safe Investigation and Quantification of Reaction Exotherms

Continuous flow chemistry can offer distinct benefits over a batch approach in certain circumstances. Highly exothermic reactions are a prime example, where the very large surface area to volume ratio of a small bore tube reactor enables faster heat removal and can in some cases prevent thermal runaway.

When the scale of a suspected reaction exotherm is not known at all, it is usually necessary for safety reasons to secure calorimetric analysis before even attempting the reaction at any significant scale, with all the cost and delay that this implies.

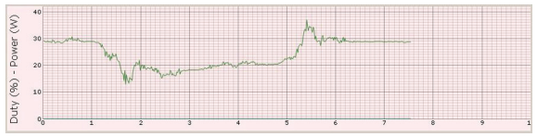


With the Vapourtec R Series flow chemistry platform, however, there is a faster approach. The Vapourtec system offers both active reactor cooling and a continuous digital readout of the power that is being used to maintain the reactor system at target temperature. This means that when a reactor is stable at the target temperature, and a continuous infeed stream of system solvent is replaced by a stream of reagents, there is an immediate and quantifiable change in the power input required to maintain reactor temperature, which is equal to the contribution from the continuous reaction exotherm.

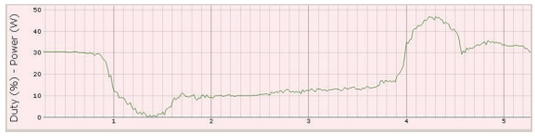
But before even attempting such a "steady state" reaction, the user can move up in steps from small discrete plugs of reagent mixture, noting the impact on reactor power at each step, to ensure that no reaction is ever attempted in the reactor which could exceed the total heat removal capacity of the reactor system.

Typical tube reactor coil, surface area to volume ratio 50x that of a round bottomed flask

Nitration Example



Experiment 1.
4 ml/min,switch to reagent causes drop in heater power of ~ 10W



Experiment 2.
8 ml/min,switch to reagent causes drop in heater power of ~ 20W

Example.
In this actual test (carried out during a 1/2 day customer visit) a nitration reaction was investigated.

Nitration
In each of the graphs, the real time onscreen readout of reactor power consumption (in Watts) can be seen dipping when the infeed is switched from solvent to reagent mixture, hence giving a value for the reaction heat output. Despite the rapid response of the system to such changes, the actual temperature of the reactor contents was maintained within 1 degree of target at all times.

About Vapourtec
Vapourtec, based in Suffolk, UK, develop and manufacture the R Series flow chemistry system, which offers a flexible development platform for mg to scale reactions. Over 120 systems are in use worldwide in pharmaceutical, fragrance, fine chemical and petrochemical labs, as well as in continuous polymerisation research.

More information at **www.vapourtec.com/heat**