Reactor Performance Comparison

This document compares the performance of two different approaches to flow reactor temperature control. One is the Vapourtec system using forced convection, the other is a competing system in which temperature control is by conductivity from a temperature controlled aluminium component.

Before looking at the performance, here is a brief summary of the different requirements for the temperature control system on a flow chemistry system.

Minimal Steady State Error

This is perhaps the most obvious requirement. Put simply, when the system says it is at a given temperature, it should be. Where a system is designed such that the reactor lies on a heat path between a heated component and something cooler (i.e. ambient conditions), the reactor temperature will always be slightly different from that of the heated component, so if the system reports the temperature of the heated component, that will not be the reactor temperature. This will get worse as the target temperature gets further from the ambient temperature.
One way around this is to try to factor in some kind of correction, but unless it takes into account a number of factors it will only be a crude approximation.

No "jumping the gun"

A system should not prematurely report achievement of set-point. If the measurement of temperature is somewhere other than at the reactor itself, there is a chance that during transients, the system will report that a new set-point has been reached before it has. This means that the first reaction at the new conditions is potentially suspect (especially if it is part of an automated sequence in which case it will commence immediately).

Rapid Transients

There are two reasons why a rapid change between set-points is desirable. Firstly the overall duration of a set of experiments will be reduced if the time spent changing temperature is minimised. Secondly (and this applies particularly to cooling from high temperatures) the time spent returning to ambient conditions when the experiment is over can be a significant factor in how usable the system is. When a system is run at high temperatures (e.g. 200°C and above) solvents will need to be pressurised to avoid boiling. When the experiment is over, the system must be kept pressurised until the temperature has come down again, during which time the reactor cannot be disconnected to configure the next experiment. It is therefore desirable that the time taken to cool down is minimised.

Comparison of Two Systems

In this study, two comparisons were carried out. Both the Vapourtec homogeneous reactor heating system and heterogeneous (column) heating system were compared with those of a competitor.

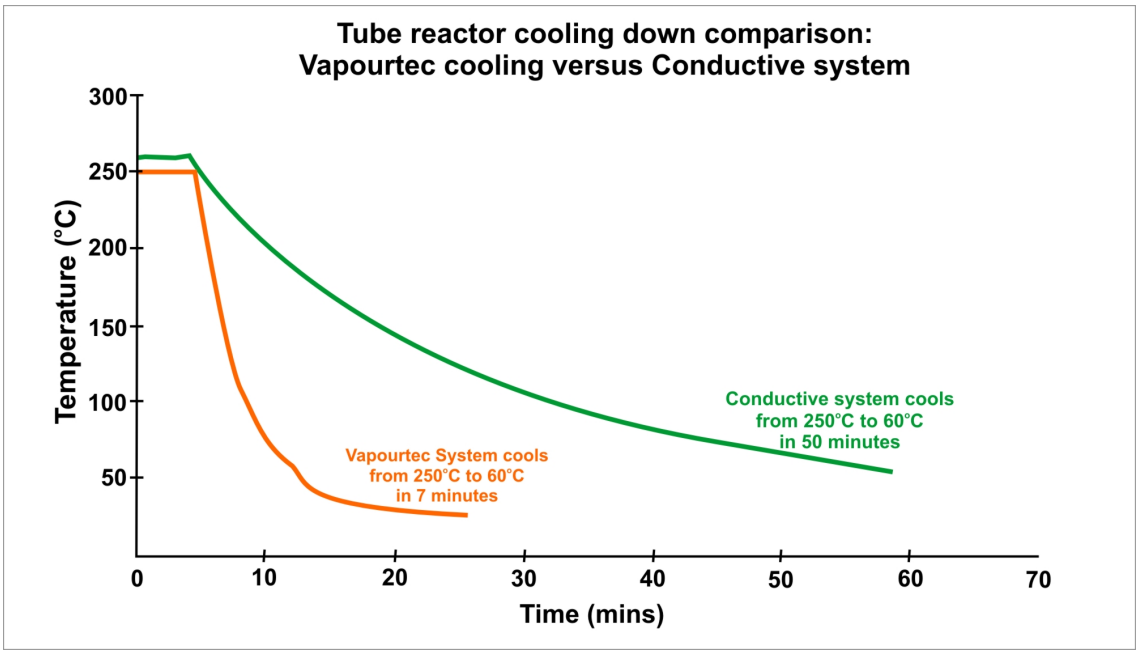
Homogeneous Reactor

For this comparison, high temperature reactors were used. Both systems use stainless steel tubing.

The Vapourtec system consists of a well insulated manifold inside which the reactor coil is wound on an open frame. Air is circulated rapidly inside the manifold and the temperature of the air is controlled so as to achieve target reactor temperature (measured by a probe in contact with the actual reactor tube.).
The competing system consists of a cylindrical mandrel onto which is wound the tube. The mandrel is itself fixed onto a temperature controlled central post which can be expanded to grip the bore of the mandrel. There is a glass cover which can be placed over the whole reactor. This serves not only to prevent accidental injury, but also has some assistive effect on the speed of heating up and the accuracy at higher temperatures.

Key observations are recorded below.

Vapourtec Forced Convection	**Tube wrapped round aluminium mandrel**
No “steady state” error	Error of up to 9 °C at very high or very low temperatures
Set-point only reported as achieved when actual reactor core at temperature	Set-point reported as achieved prematurely, when mandrel reaches temperature, causing first experiment at that temperature to be inaccurate.
Rapid cooling for fast transitions and safe handling soon after high temp reactions complete (< 10 mins)	High thermal inertia, slow cooling. Slow to move between different temperatures, long time before safe to handle (up to 1 hour).



As can be seen from the graph, the competing system takes a great deal longer to cool from a high operating temperature. This is perhaps not surprising as the Vapourtec system has a very low thermal inertia and rapidly circulating air.

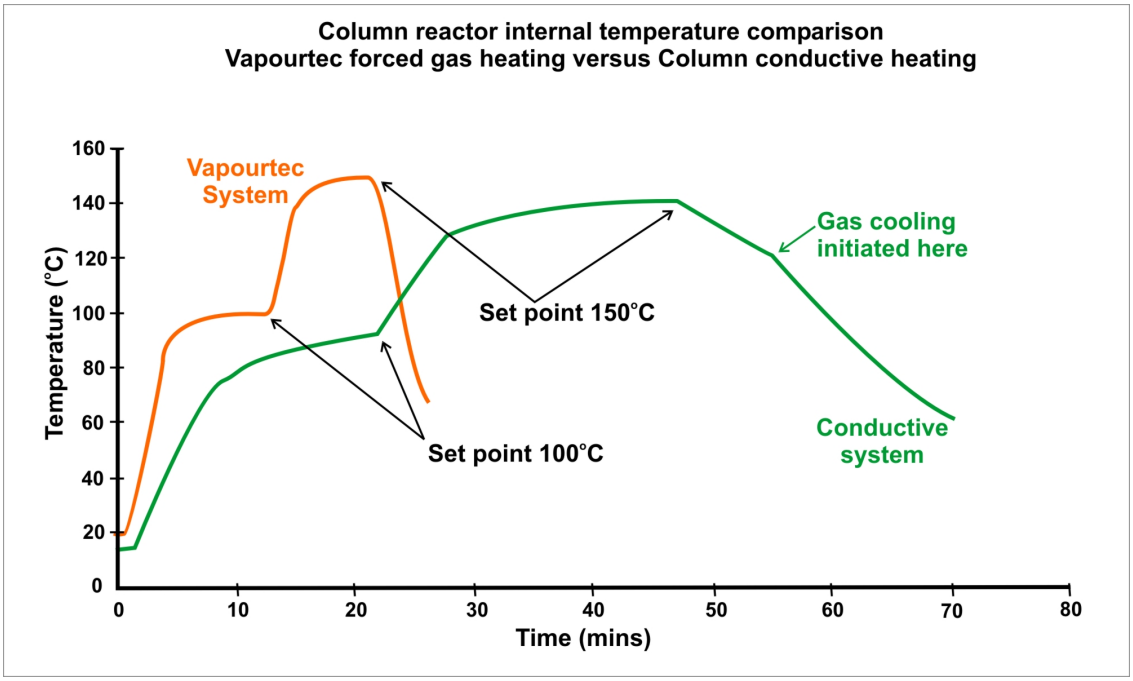
The competing system has a facility for gas to be blown through the reactor mandrel to speed cooling. In the experiment shown on the graph above, this was used for the cooling part of the graph. Without it the cool-down time would be longer.

Heterogeneous Reactors

The Vapourtec column heating system again consists of a manifold inside which air is rapidly circulated around the reactor. The entire column is visible.

The competing system consists of a 2 part shell which clamps around the column. One of the two clamp parts is temperature controlled, while the other is not. Where the shell is in contact with the column, the column is not visible.

Vapourtec Forced Convection	**Two part "shell" clamped around column, only one part temperature controlled**
High conductivity	Line contact, poor conductivity into column
Set-point only reported as achieved when actual reactor core at temperature	Set-point reported as achieved prematurely, when clamp reaches temperature, causing first experiment at that temperature to be inaccurate
No steady state error	Large steady state error as one half of clamp uncontrolled and drifts towards ambient temperature
Full column visibility	No visibility of column over controlled area. No temperature control over visible area.



Conclusion

The Vapourtec system offers an advantage in 3 areas

- Accuracy
- It does not report temperature set-point achieved prematurely
- Transients are far more rapid, saving time and enabling safe handling and reactor dismantling sooner after experiment completion.

More information about the Vapourtec system is available at

http://www.vapourtec.co.uk/products/rseriessystem