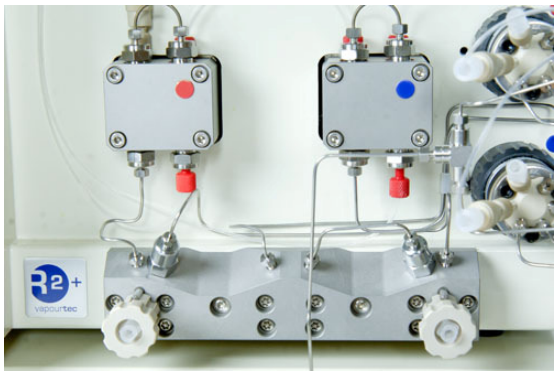
New High Pressure Pump Module Released

March 2012 –
Vapourtec Announce new High Pressure Pump Module

Vapourtec have announced a new addition to the range of pump modules available for the R-Series™ system. In addition to the standard and the acid resistant version (each available with or without sample injection loops, in one or two pump configuration) there is now a special high pressure version.



Vapourtec High Pressure System, (above) featuring the new R2+HP Pump Module, and a close-up of the new pump manifold (left)

What Exactly Can It Do ?

The new module has all the same functionality as the standard pump module except that

- it has a maximum pressure limit of 200 bar
- all fluid tubing and the pump manifold are made of stainless steel
- there is no strong acid resistant version
- a high pressure pump module can be added to an existing two pump system for increased capability

Why ?

In what situations are such high pressures useful ?

There are at least four situations where high pump delivery pressures enable more flow chemistry to be explored.

- High temperatures with volatile solvents
- Continuous flow polymerization
- “Flash chemistry”
- Synthesis of ionic liquids

These are explained in more detail below.

Volatile Solvents / High Temperatures

There are several situations where either volatile solvents, volatile reagents or dissolved gases are involved, and the permissible temperature is effectively limited by the available pressure. In these instances, a higher pressure capability permits the user to make the most of the available temperature capability.

For example

- Dichloromethane at 250°C requires > 79 bar to prevent it from boiling in the reactor
- Ethanol at 250°C requires > 66 bar to prevent it from boiling

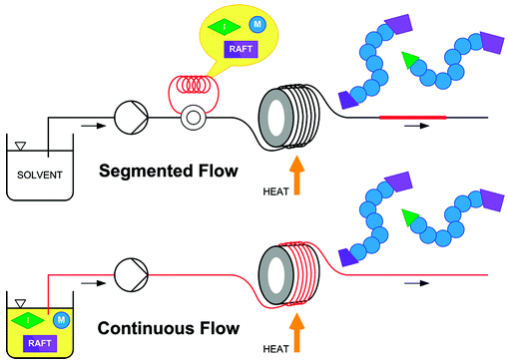
Vapourtec offer a free chart showing what pressure is required for each solvent to ensure that it does not boil at a particular temperature.
It can be found at :

http://www.vapourtec.co.uk/bpchart

Continuous Flow Polymerization

In recent years, scientists in the field of "living" polymerization have demonstrated continuous flow processes that give great control over the final product. But as polymers form, the viscosity increases, and long residence times are typically required.

The combination of long reactors and high viscosity results in significant back pressures, and in some cases a higher available pump pressure will permit a longer reactor and hence a higher throughput.



RAFT polymerization in flow, from recent paper

http://dx.doi.org/10.1021/op1003314

More information at

http://www.vapourtec.co.uk/applications/polymerization

Very Fast Reactions

This area has become known as “flash chemistry”, and involves reactions that may require residence times of the order of seconds. Often such reactions are significantly exothermic, and it is necessary to ensure rapid mixing or else the extent of the mixing becomes a key reaction parameter that does not scale well.

When a fast reaction is exothermic, it may not be sufficient to rely on the relatively thick wall of a glass chip to transport heat away, otherwise significant temperature spikes can occur. In these cases, the optimum reactor may be a stainless steel capillary, which offers

- excellent heat transfer
- better diffusion mixing (small radial distances) resulting in reduced overall axial dispersion and lower deviation in residence times

However, stainless steel capillary reactors generate more back pressure than larger bore tubing or chip type mixers and as a result, higher pump pressures are required.

For an analysis of mixing, dispersion and the effects of varying reactor cross sections, see the following paper:

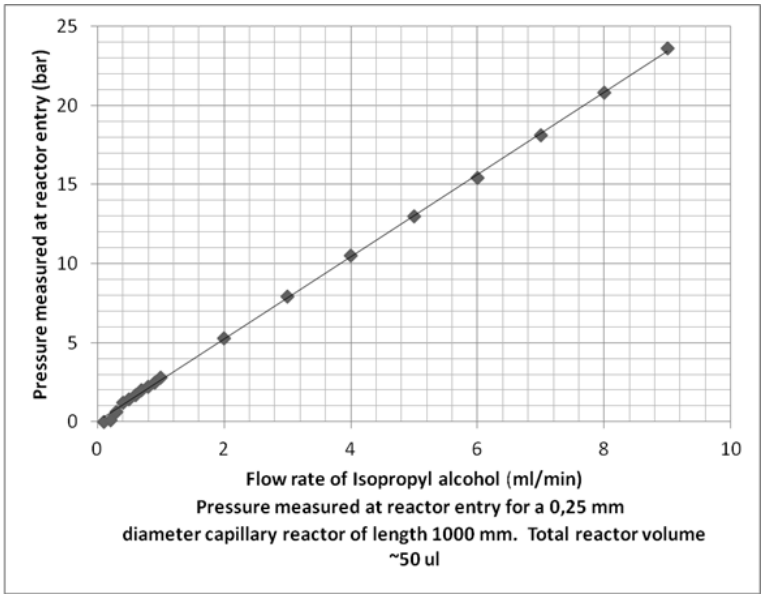
Mixing and Dispersion in Small-Scale Flow Systems
Kevin D. Nagy, Klavs F. Jensen, Bo Shen, Timothy F. Jamison
Novartis-MIT Center for Continuous Manufacturing, MIT, USA
http://dx.doi.org/10.1021/op200349f

The graph (right) shows typical back pressure from a 0.25mm (0.01”) bore stainless steel capillary reactor.

1m of this tube gives a 50µl reactor volume.

In a reactor of this size, a 1ml/min flow rate equates to a 3 second reactor residence time.

It is therefore clear that at greater flow rates (and hence with longer reactors) the pressure required will be significant.



Pressure measured at reactor entry for a 0,25 mm diameter capillary reactor of length 1000 mm. Total reactor volume ~50 ul

Synthesis of Ionic Liquids

There is a growing interest in the use of ionic liquids (ILs). These can exhibit a wide range of tunable solvating properties, depending on the components used in their synthesis, and have been termed “designer solvents”. Their negligible vapour pressure and their ability to be re-used has made ILs of great interest in the push for “greener” chemical processes.

ILs are expensive, however, mostly because of the challenges of the synthesis route. As the initial synthesis step can be highly exothermic, the process is often carried out at very low temperatures (to slow the reaction) and with some dilution solvent. (Purity is key, and any hotspots in the reaction vessel may compromise this). The upshot of these constraints is that reaction times may be as much as 24 hrs and a solvent removal step is inevitable afterwards. This contributes to the high cost.

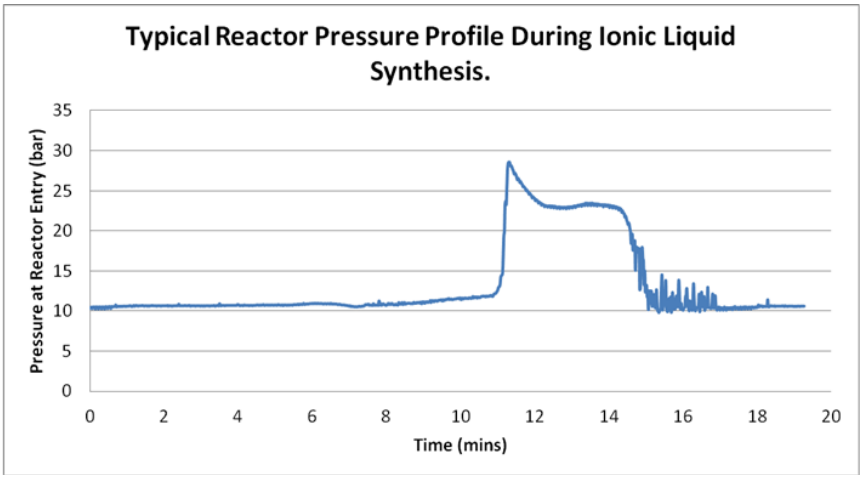
Synthesis in flow, however, offers a very different scenario. In a flow system with good heat transfer and active heat removal, it is possible to run IL synthesis with neat reagents at higher temperatures. This speeds the reaction, giving residence times of perhaps 10-30 minutes, there is no solvent removal required afterwards, and consistent temperature conditions across all the output result in a high purity.

More information (including an application note) at

http://www.vapourtec.co.uk/ionicliquids

There is another important property of ILs to consider, however. They often exhibit high viscosity, so during IL synthesis, the viscosity will change throughout the reactor. The graph below shows the change in back pressure measured on entry to a single reactor during IL synthesis when the system switches from solvent to reagents and back.

It can be seen that a pump capable of delivering over 25 bar is required in this example.



For this reason, a capability for high pump pressure is useful if IL’s are to be synthesized at higher throughputs, (ie with longer reactors).

In Conclusion

There are now several applications of growing importance in the continuous flow field for which a capability for higher pressure pumping is essential.

The new Vapourtec pump module is ideally suited to these applications.

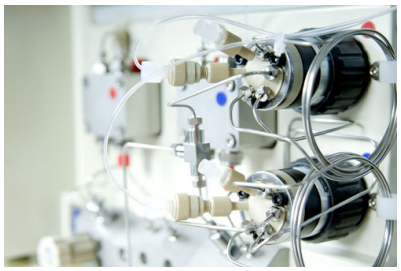
FAQ

Q When is this new pump module available ?
A Immediately.

Q Do I need to buy special stainless steel tubing ?
A The high pressure system comes with all tubing, Back Pressure Regulators (BPRs) and fittings to make it ready for use. (see right)

Q Is a Hastelloy® version available ?
A Not at this time.

Q Is it possible to use sample injection loops with the high pressure system ?
A Yes.



Q Can a 4 pump system be constructed featuring a mixture of high pressure pump modules and standard pump modules.
A Yes

Q What reactors are available for use at these elevated pressures ?
A PFA reactors cannot be used at such pressures, of course. Vapourtec now offer both standard temperature range (ambient to 150°C) and high temperature range (ambient to 250°C) reactors in stainless steel with suitable high pressure connectors BPRs and other fittings.

Q Are there capillary reactors available for “flash chemistry” ?
A Yes, please contact Vapourtec to discuss your application