

## Vapourtec Adds Cooled Column Reactor System

In January 2010 – Vapourtec announced the launch of the cooled tube reactor module for the R Series Flow Chemistry System.

Now the cooled column reactor module is available.

### What can it do ?

The cooled column module can control the temperature of an Omnifit column used as part of a flow system, from ambient down to  $-40\text{ }^{\circ}\text{C}$ , with control accuracy of  $\pm 2\text{ }^{\circ}\text{C}$ .

And one Vapourtec R Series system can host up to 2 cooled column reactors simultaneously, which can each be **independently** temperature controlled.

Each cooled column reactor system includes

- a pre-cooling zone for each of 2 reagents to ensure they are at target temperature before mixing
- a cooled mixer
- a cooled Omnifit column

### Why would I want this ?

This enables both reactions which need to be run at low temperatures **and** reactions that are run close to ambient but which are expected to be very exothermic.

### How does it work ?

The standard Vapourtec reactor heating system controls temperature by rapidly circulating air around the reactor within the reactor manifold. Hot air is used to raise the reactor temperature, ambient air is used to cool it.

The cooled column module is based on the same principle as the cooled tube reactor module. Gas is circulated just as described above but that gas is very cold. A dry ice heat exchanger is used to generate chilled nitrogen which is used to cool the column down to the low temperatures required. No external recirculating chiller is required, and the reactor temperature set point is completely programmable, under control of either the simple front panel controls or the Flow Commander™ control software.

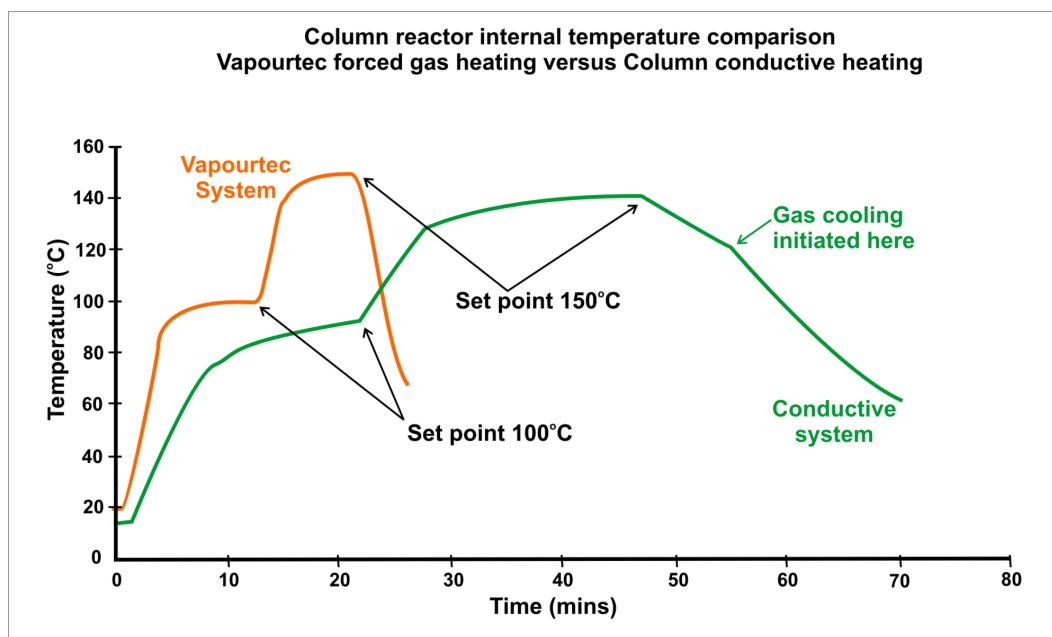
The forced convection temperature control system is clean, reliable and provides extremely accurate temperature control. Unlike other methods of controlling column temperature, the reaction is visible, while the entire wall of the column is kept at the required temperature at all times.

## How well does the “forced convection” principle work ?

Scientists are often sceptical at first.

However, this technology is more accurate than (for example) clamping a column between temperature controlled aluminium components.

The graph below shows a comparison between the Vapourtec *heated* column and another system based on the “heated clamp” method.



Each system was instructed to go to the next target temperature after it had reported reaching the previous target. Temperature was measured inside the column, in the actual liquid.

There are several key differences

1. The Vapourtec system moves between target temperatures far faster, saving time.
2. Because the other system reports on the clamp temperature, not the column temperature, it reports arrival at target prematurely when the column is still changing temperature. Vapourtec systems do not.
3. The other system does not ever correctly reach the target. That is, it reports clamp temperature but the contents of the column continue to lag. The Vapourtec heated column system reports column contents temperature correct to  $\pm 1^{\circ}\text{C}$ . The worst case error with the other system is in excess of  $10^{\circ}\text{C}$ .
4. The contents of the Vapourtec column are visible at all times.

### What else will I need to use this new module ?

The cooled column reactor does not include the actual Omnifit column. A document explaining which Omnifit columns are supported is available on request.

You'll need access to dry ice, and a supply of dry nitrogen.

Your Vapourtec system may need upgrading to be compatible with the new module (see below) if it is older than Jan 2010.

No additional recirculating chiller, water supply or other cooling equipment is required.

### FAQ

**Q** *Why does it not use an external recirculating chiller ?*

**A** Feedback from users indicated that such chillers are often too bulky and can represent an inconvenience in a crowded lab. They can also represent a significant investment in a small group. Furthermore, if there are several cooled reaction stages to be cooled at different temperatures, that would require multiple recirculating chillers.

**Q** *How cold can the column temperature be set ?*

**A** Anything from ambient down to -40 °C

**Q** *How accurately does the system control temperature ?*

**A** +/- 2°C

**Q** *Will I still be able to see my reaction in the tube reactor ?*

**A** Yes, there is a viewing window in the reactor housing insulation.

**Q** *-40 °C is fairly cold. Doesn't it ice up ?*

**A** The reactor itself is surrounded by dry gas, all contained within a double insulated manifold. There will be some ice present at the cold gas entry point near the top but this is minimal.

**Q** *How many cooled column reactors can I use at the same time ?*

**A** Three of the four positions on the R4 reactor module can accept a cooled column reactor. Up to two cooled columns be used simultaneously.

Q **Are all the Vapourtec column manifolds available cooled ?**

A The standard *heated* column reactor manifold is available in three sizes, small/medium/large. However, the *cooled* manifold is only available in the medium and large sizes, which means that 6.6mm Ø x 50mm long Omnifit columns are not catered for.

Q **Can the Flow Commander™ software control cooled column reactors the same way it controls other reactors ?**

A Yes. If you need, for example, to optimise a reaction over a range of sub ambient temperatures (or characterise the reaction kinetics) it works just the same as with temperatures above ambient. An upgrade will be required for FlowCommander™ software but this is free.

Flow Commander™ will show how hard the cooling system is working, too, which enables you to predict dry ice usage.

Of course, simple manual front panel control is available too – Flow Commander™ software is not obligatory.

Q **I already have a system. Can it be upgraded ?**

A Systems that left the factory before the start of 2010 will need to be returned to base for an upgrade for either cooled column **or** tube reactors.

An onsite upgrade is not possible, unfortunately. Systems supplied after that date but before November 2010 (or which have already been upgraded during 2010 for cooled tube reactor use) may need a firmware upgrade in order to use cooled columns, but this can be done by the user onsite using a simple software utility and a supplied cable.

Q **What exactly will I need ?**

A If your system does not need a back to base upgrade, but you have no cooled reactor equipment yet, you will need:

- one chilled gas regulator system (simply plugs into the R4)
- a single dry ice reservoir and gas tubing (stands on the bench)
- a single cooled column manifold (snaps onto the front of the R4 like any other reactor manifold) and a suitable Omnifit column.

If a 2<sup>nd</sup> reactor is to be cooled, the user can then add

- a second reservoir & gas tubing
- a second cooled column reactor manifold

If, however, you already have a cooled tube reactor(s), you will just need the cooled column manifold.

Q ***So can I queue up a sequence of cooled reactions in Flow Commander™, load up the dry ice reservoir and hit start ?***

A Yes, just like with heated reactions, the system will move to the next temperature set-point, and then start the reaction.

Q ***Can I use liquid nitrogen for the cooling rather than dry ice ?***

A Not at present, though it is an option that may be considered for the future.

Q ***What does the cooled column module cost ?***

A Please contact Vapourtec for a precise quotation. The cooled column manifold costs less than the cooled tube reactor manifold.

Q ***What does the back-to-base upgrade cost ?***

A This only applies to R4's shipped prior to January 2010. The cost depends on your location, as the price includes 2 way shipping for the R4. Please contact Vapourtec for a price.

Q ***When is it available ?***

A The cooled column system will be available from November 2010