

Vapourtec Launches -70 °C Cooled Reactor System

January 28th 2010 – Vapourtec are proud to announce the launch of the cooled reactor module for the R Series Flow Chemistry System.

The R Series has up until now been able to control reactions from ambient to 250 °C , but with the new module, reactions can be cooled down to -70 °C.



R Series shown with two cooled reactors, running at two different temperatures.

What can it do ?

An R series system can have up to 2 cooled reactor positions. These can be **independently** controlled at any temperature from ambient down to -70 °C, with control accuracy of +/- 0.5 °C.

Each cooled reactor position includes

- a pre-cooling zone for each reagent to ensure they are at target temperature before mixing
- a cooled mixer
- a cooled reactor
- a cooled post mixer for inline quenching if required

Reactors are available in 2, 5 and 10 ml sizes and all reactors are fully resistant to corrosive reagents (for example fuming nitric acid !).

How does it work ?

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

With the new cooled reactor module, a dry ice heat exchanger is used to generate chilled nitrogen which is used to cool the reactor 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.

(New users are often strangely sceptical of the forced convection temperature control used by Vapourtec - until they have used it. It is clean, reliable, and provides extremely accurate temperature control. Because the reactor tube is surrounded on all sides by the temperature controlled gas, conductivity is actually better than the line contact achieved by winding a reactor tube around a metal mandrel. And the new cooled reactor module is no exception.).

“Pre-cooling” and Quenching

Often cooling is required to slow down what would otherwise be a very rapid reaction. But there is no point in cooling a reactor if the reagents have already been mixed (and started reacting) upstream of the cooled region, because the reaction will already have been partially completed by the time it enters the cooled area. Furthermore, rapid exothermic reactions can dissipate a significant amount of heat in the actual mixer, which needs to be removed.

For this reason, Vapourtec have included the mixer that precedes the reactor within the cooled housing, along with the actual reactor, so the reagents are already in the cooled zone when they first meet. Not only that, but lengths of “pre-cooling tube” before the mixer ensure that the reagents are already at target temperature before they are brought together.

There is also provision for a second mixer **after** the reactor, enabling inline quenching to occur before the reaction products leave the cooled zone.

What else will I need to use this new module ?

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).

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. **A two stage chiller capable of cooling a typical reaction to -70 °C would cost approximately 5 times what the Vapourtec cooled reactor module costs.** Furthermore, if two reaction stages are to be controlled at two, different temperatures, that would require two recirculating chillers.

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

A Down to -70 °C

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

A +/- 0.5°C (verified in tests by measuring the actual fluid in the reactor).

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, see picture below.

Q **-70 °C is pretty cold. Doesn't it ice up ?**

A The reactor itself is surrounded by dry gas, all contained within a double insulated manifold.

Ice build up is not a problem.

However, as you can see if you look closely in the picture (right), at -70 °C, the reactor viewing window does start to fog up a bit, but a wipe with a soft cloth does the trick.

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

A The R4 reactor module with cooling module added now offers:

- Two positions capable of -70°C to ambient **or** ambient to 150°C
- One position capable of ambient to 150°C
- One position capable of ambient to 250°C

Q **Can the Flow Commander™ software control cooled reactors the same way it controls heated 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.

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. An onsite upgrade is not possible, unfortunately. Systems supplied after that date can be upgraded by the user, simply by purchasing the cooled module.

Q **What does a user upgrade entail ?**

A A standard “cooled reactor” module includes

- 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 reactor manifold and a single reactor coil (snaps onto the front of the R4 like any other reactor manifold)

If a 2nd reactor is to be cooled, the user can then add

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

Q **How long does the system take to move between different temperatures ?**

A Tests at an ambient temperature of 25 °C show that

- from ambient to -40°C takes approximately 7 minutes.
- from ambient to -70°C takes approximately 16 minutes.

Q **What is the typical dry ice consumption ?**

A The harder the cooling system has to work, the more will be consumed. To give some idea, at -40°C, approx. 0.2 kg of dry ice / hr (note that the reservoir can be topped up during a reaction). The reservoir accepts between 1.5 and 2kg of dry ice depending on granule size, so up to 8 hrs at -40°C is possible unattended. With colder temperatures, or a significantly exothermic reaction, the consumption will be higher, of course. If you wish, two reservoirs can be used in series to give a far longer unattended runtime.

Q ***Can the cooled reactor system be used in conjunction with the acid resistance announced in November 2009 ?***

A Absolutely. The cooled reactor tubing and fittings are all acid resistant.

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 ***Is there a cooled system for column reactors ?***

A Not yet, but watch this space. More announcements are expected later in the year.

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

A Please contact Vapourtec for a precise quotation. The cost is approximately 15% of the overall R Series system price for the first cooled reactor position, and about half as much again for the 2nd position.

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

A This depends on your location, as the price includes 2 way shipping for the R4. Please contact Vapourtec for a price.