### VSD006

### Installing Vapourtec Cooling Module and cooled reactor(s)



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#### 1 TOOLING / EQUIPMENT REQUIRED

- Dry Nitrogen or Dry Compressed Air, minimum pressure 2 bar, minimum flowrate 35 litres / min.
- Dry Ice approximately 2 kg
- Adaptor(s) to connect dry Gas / Air supply to a 6mm OD x 4mm ID pneumatic hose
- Vapourtec cooling module equipped with pressure regulator and insulated umbilical (Part No. 50-1140)
- Vapourtec cooled reactor (Part No. 50-1141)
- 3mm Allen Key

#### 2 BACKGROUND / METHOD

The Vapourtec R-Series R-4 reactor heater / cooler can be equipment with either one or two cooled reactors. Cooled reactors can be used in positions 1, 3 or 4. Position 2 in NOT available for mounting cooled reactors. For E-Series system, both positions can be used for cooling. Cooled reactors of two different types can be used;

Reactor type	Temperature Range
Cooled Columns	- 40° C
Cooled tube reactors	- 70° C
Cooled UV-150 Photochemical reactors	- 40° C
Cooled glass chip microreactors	- 40° C

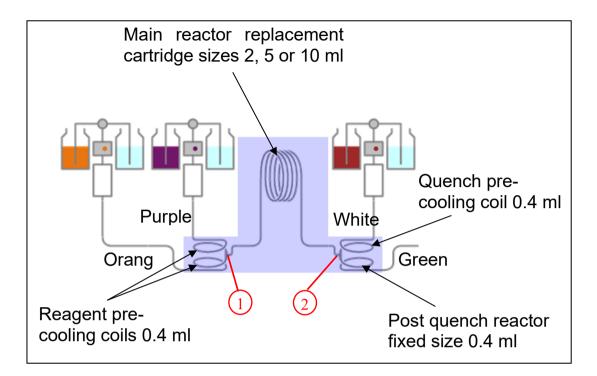
The cooling function is provided by bleeding dry nitrogen gas or dry compressed air at low pressure through a bed of dry ice. The resulting mixture of nitrogen (or air) and carbon dioxide is cooled to a temperature close to -78° C and is fed intermittently via a pinch valve into the cooled reactor assembly. Within this assembly the cooled gas mixture is recirculated around the reactor assembly to provide heat transfer between the gas and the reactor. The cooled tube reactor assembly is equipped with the features for the following functions:

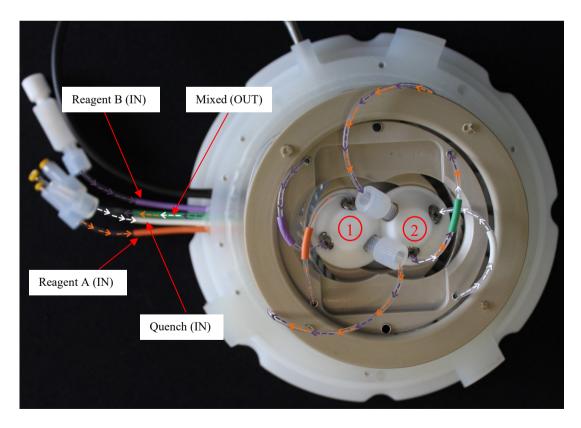
Feature	Description
Pre-cooling reagent A	Orange connection line, 0.4 ml pre-cooling coil
Pre-cooling reagent B	Purple connection line, 0.4 ml pre-cooling coil
Reagent mixing	Tee piece mixer, port size 0.8 mm DIA
Single Core Main Reactor	Replaceable standard sizes 2, 5 or 10 ml
Pre-cooling Quench solution	White connection line, 0.4 ml pre-cooling coil
Mixing the quench solution	Tee piece mixer, port size 0.8 mm DIA
Post quench reactor	Fixed volume 0.4 ml
Outlet port	Green connection line
Dual Core Reactor	2 Coils of different sizes totalling 10ml

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#### 3 SCHEMATIC DIAGRAM OF REACTOR ASSEMBLY AND FLOW PATH

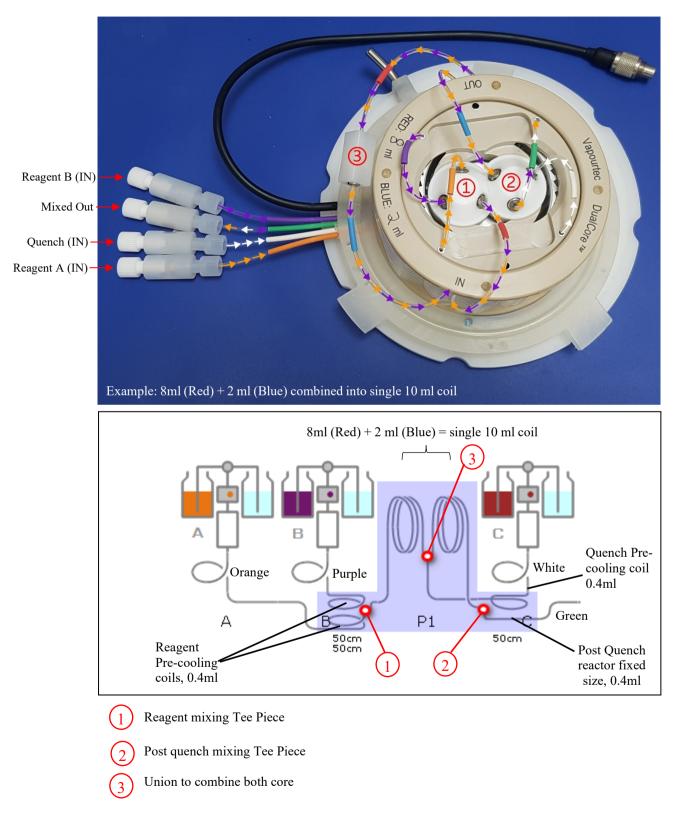
#### 3.1 Single Core Reactor Flow Path





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#### 3.2 Dual Core Reactor Flow Path



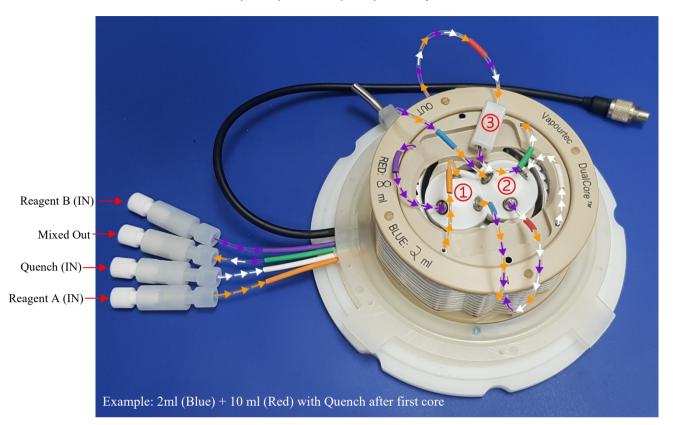
3.2.1 Dual core 8ml (Red) + 2 ml (Blue) combined into a single 10 ml coil

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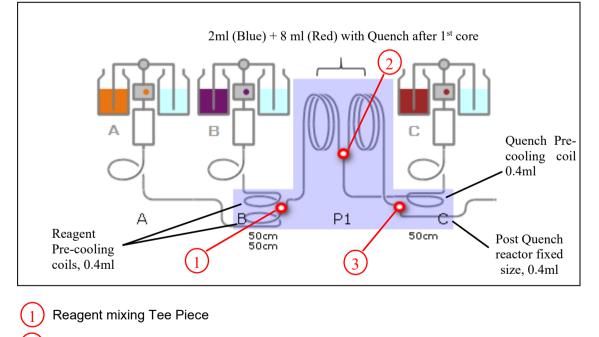
#### INO Vapourtec () TE. a Ш BLUE: DualCore Reagent B (IN) -Mixed Out 4 3 Quench (IN) Reagent A (IN) Example: 8ml (Red) + 2 ml (Blue) with Quench after first core 8ml (Red) + 2 ml (Blue) with Quench after 1st core Α В Quench Precooling coil Orange Purple White 0.4ml Green Ρ1 50cm 50cm 50cm Reagent Post cooling Pre-cooling 3 reactor fixed coils, 0.4ml size, 0.4ml Reagent mixing Tee Piece Quench solution mixing Tee Piece Union to exit line

#### 3.2.2 Dual core 8ml (Red) + 2 ml (Blue) with quench after first core

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#### 3.2.3 Dual core 2ml (Blue) + 8 ml (Red) with quench after first core



Quench solution mixing Tee Piece

3) Union to exit line

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#### 4 SAFETY INFORMATION

The symbols shown below will be used throughout this document to draw the reader's attention to important information.

$\triangle$	Attention. Important notes.	601	Note.
$\oslash$	Not permitted. Misuse may cause damage.		Isolate equipment from mains

#### 5 SETUP AND OPERATION OF THE COOLING MODULE

Cooled reactor should only be used when the Vapourtec cooling module (50-1140) is connected to the system. The cooling module utilise dry ice to cool a flow of nitrogen or compressed air, this cooled gas is then directed to the reactor via an insulated umbilical tube. A pinch valve is used to control the amount of cooled gas circulating inside the reactor to achieve the set target temperature.

Only dry ice should be used in the cooling module. Using solvents or other liquids will **CAUSE DAMAGE** and may create a potential hazard.

For an efficient cooling, the cooling module requires a minimum inlet gas pressure of <u>2 bar</u> and minimum flowrate of <u>35ml/min</u>. If this requirement is met, the cooling module shall cool the reactor from ambient temperature of 25 °C down to:

- a) -40°C in approximately 7 minutes.
- b) -70°C in approximately 16 minutes.

The dry ice consumption depends on how hard the cooling system has to work. At -40°C, approximately 0.2 kg of dry ice/hr is consumed (note that the reservoir can be topped up during a reaction). The reservoir accepts between 1.5 to 2kg of dry ice, depending on granule size. Thus, up to 8 hrs unattended operation at -40°C is possible. With colder temperatures, or a significantly exothermic reaction, the consumption will be higher, in this case, two reservoirs can be used in series to give a far longer unattended runtime.

The size of the dry ice has a direct impact to the cooling efficiency:

- Chunky dry ice will have less contact surface area
- Fine dry ice (snowy form) will create high back pressure for the gas flow and reduce the gas flow rare

Dry ice in granular form with approximately 1cm diameter x 2cm length is recommended for efficient cooling. See Figure 1.



Figure 1

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#### 5.1 Assembly of the Cooling Dewar parts



**Step 1** – Fit the regulator and bracket in the threaded holes using the 4 x M4 screws provided (See Fig 2). If being used with an E-Series module, the E-Series bracket also needs to be fitted (See Fig 3).

**Step 2** – Fit the pinch valve tube by screwing into the threaded hole. Finger tight is sufficient (See Fig 4). Attached the opposite end (larger of tube) into the push fitting on the regulator, pushing in until you can't release by pulling (See Fig 5).

**Step 3** – Fit the Pressure Relief valve into the threaded hole on the side. Finger Tight is sufficient. (See Fig 6)

**Step 4** – Remove one of the blanking plugs and fit the insulated umbilical cord into the threaded hole positioned opposite of the regulator. Leaving the second hole blanked. (See Fig 7)

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Figure 2

Figure 3

Figure 4

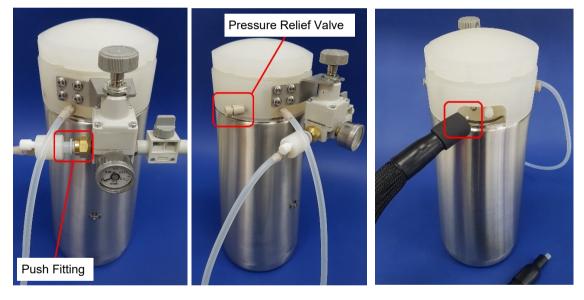


Figure 5

Figure 6

Figure 7

#### 5.2 Connecting gas supply

Dry nitrogen gas or dry compressed air is required for the cooling module. The maximum incoming gas pressure from the laboratory supply is 8 bar. High pressure gas supply could cause damage to the cooling module. A gas regulator is recommended to further reduce the gas pressure to 2 bar.

The E-Series or R-Series system should have an inert gas line installed. User can cut the blue gas tubing and split the gas line with the supplied black tee connector.

If no inert gas line is installed on the system, use the supplied gas connection kit (orange and blue tube) to connect the nitrogen/compressed air from the laboratory supply. See figure 8 and figure 9.

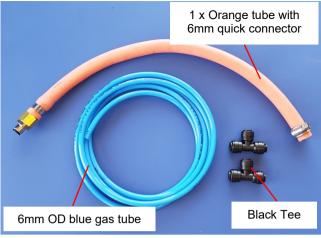


Figure 8

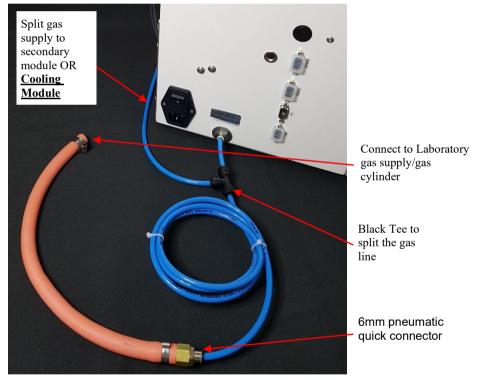


Figure 9

#### 5.3 Sitting and operation of the cooling module



The cooling module is normally sited to the left-hand side of the R-Series or E-Series pumping module in close proximity to the three pinch valves on the side of the R4 module. Refer to Fig 10.



Figure 10

To integrate the cooling module to the R-Series or E-Series:

- A pinch valve is used to control the cooled gas flowrate. To connect the pinch valve, insert the silicone tube into the pinch valve as shown in Figure 11.
- For R-Series, cooling is available on position 1, 3 and 4. If the reactor is to be used in position 1 then use the upper pinch valve. If the reactor is to be used in position 3 then use the middle pinch valve. If the reactor is to be used in position 4 then use the lower pinch valve.
- For E-Series, cooling is available on both positions. Depending on the reactor position used, insert the silicone tube into the respective pinch valve.

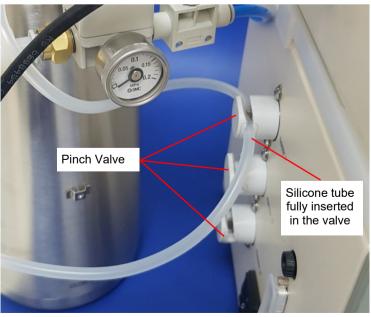


Figure 11

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- Connect the 6mm OD / 4mm ID pneumatic tubing (blue gas tube) to the pressure regulator mounted on the cooling module.
- The inlet nitrogen/compressed air (from the laboratory supply) should be set to 2 bar pressure which shall create approximately 0.5 bar pressure inside the cooling module. The regulator on the cooling module is pre-set (0.5 bar) and should NOT be adjusted.
- An ON/OFF valve located on the cooling module is used to switch on/turn off the gas flow into the cooling module. See Figure 12.

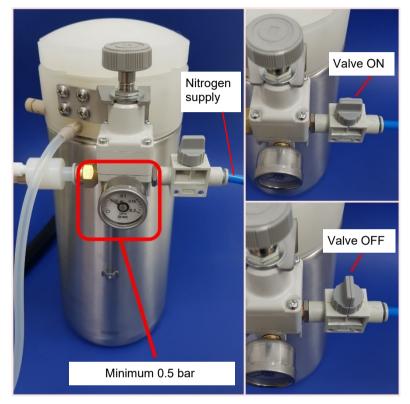
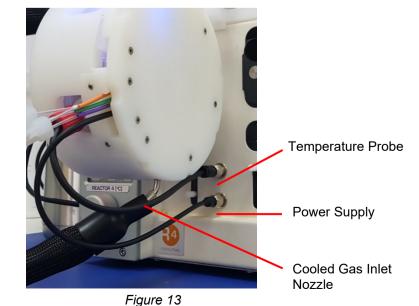


Figure 12

- Install the cooled reactor into the appropriate location on the R4 module. Once the reactor body is installed, connect the temperature sensor and power supply to the R4 module as shown in Fig 13.
- Connect the insulated umbilical onto the cooled gas inlet nozzle located below the cooled reactor (Fig 13).



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#### 6 OPERATION OF THE COOLED REACTOR



Before proceeding, ensure that the Nitrogen of compressed air supply is turned off.

Only dry ice should be used in the cooling module. Using solvents or other liquids will CAUSE damage and may create a potential hazard.

- Remove the lid of the cooling module and fill completed with dry ice. Replace the lid.
- Ensure that the reactor tubes do not contain any liquids that will freeze at the temperature at which the reactor is to be operated.
- Turn on the R4 power using the rocker switch on the lower left-hand side. You will hear the pinch valves open for 12 seconds to confirm function.
- Turn the nitrogen or compressed air supply on.
- Using the manual control knob or the control software, SET the temperature at which you wish to operate the reactor.
- Using the manual control knob or the control software, turn the reactor ON. The pinch valve will open and gas will be heard exhausting from the reactor. After a few seconds the reactor temperature will begin to reduce. Once the desired temperature has been reached the pinch valve will close and the sound of the exhausting gas will stop.

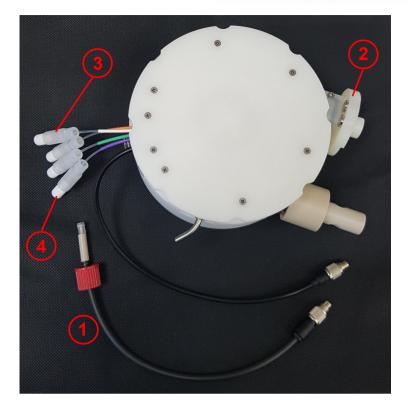
The system will now maintain the reactor temperature at the set temperature by repeatedly opening and closing the pinch valve.

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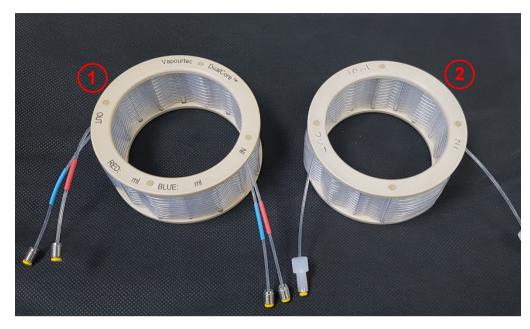
#### 7 SPARE PARTS



No	Part Number	Description
	50-1140	Vapourtec Cooled Module – Complete Assembly
1	40-1314	Umbilical
2	30-3340	PRV Check valve
3	50-1381	Cooling Module Regulator



No	Part Number	Description
	50-1141	Cooled Tube Reactor Assembly
1	50-1010	Temperature sensor for tube reactor
2	40-1288	Outlet Mounting top adaptor cooled
3	40-1924	Union – PFA
4	40-1313	PTFE Blanking Plug



No	Part Number	Description
1	50-1197	Replacement Dual Core PFA tube cartridge for cooled tube reactor
	50-1146	2ml replacement PFA tube cartridge for cooled tube reactor
2	50-1145	5ml replacement PFA tube cartridge for cooled tube reactor
	50-1144	10ml replacement PFA tube cartridge for cooled tube reactor

#### 8 VAPOURTEC CONTACT DETAILS

If you experience any issues or need assistance please contact using the email address below.

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