

Vapourtec R-4 Flow Reactor Heater

User Manual

Original date: 31^{st} July 2008This revision:Rev 9 (2nd July 2020)

CONTENTS

1	Product Description				
2	Safety I	Safety Information			
3	Installin 3.1 3.2 3.3 3.4 3.5 3.6 3.7 3.8 3.9 3.10	g the R-4 flow reactor heater Unpacking Siting Flow reactors product range Overview of R4 Heater Assembly of column reactor into the heat exchangers Assembly of the glass heat exchangers onto the reactor Changing a reactor cartridge in a glass tube reactor Assembly of tube reactor onto the reactor Changing a reactor cartridge in a high temperature (HT) reactor Electrical connections	4 4 7 8 10 11 13 14		
4	The use 4.1 4.2	iser interface Description of the interface Display of data during heating			
5	Operatio 5.1 5.2 5.3 5.4	Operation5.1Setting the temperatures for R4 channels5.2Turning the heaters on5.3Turning the heaters off5.4Configuring the reactor for automatic operation			
6	Interfact 6.1 6.2 6.3 6.4	ing the R-4 for automation Overview of Serial Interface Switched I/O Interface Command Protocol 6.3.1 General 6.3.2 Serial Port Settings 6.3.3 Control Commands 6.3.4 Information Commands User serviceable parts 6.4.1 Replacing the fuse 6.4.2 Replacing the pinch valve	23 23 24 25 25 25 25 25 26 28 28 28		
7	6.5	Spare parts & Accessories listing	29		
1	I rouble	snooling	33		
8	Genera	Specifications	34		
9	Reactor	temperature and pressure range	35		
10	Vapourtec warranty 10.1 Standard limited warranty 10.2 Service contracts 10.3 Sample copy of EC declaration of conformity		37 37 38 40		
11	Vapourtec contact details 41				

vapourtec



1 PRODUCT DESCRIPTION

The Vapourtec R-4 is a flow reactor heater for applications in Flow Chemistry. It has 4 independently heated zones and 3 independently cooled zones (cooling module required). The temperature of each can be set independently in the range of:

Heating: Ambient to 250 °C for channels 2 & 4 Ambient to 150 °C for channels 1 & 3 Cooling: -70°C to ambient for channels 1, 3 & 4

The system has all the features necessary to ensure successful synthesis or reaction clean-up in flow chemistry. It has the ability to precisely control reaction conditions throughout the entire working length of the reactor.



Figure 1 R4 Reactor Heater

2 SAFETY INFORMATION

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



Attention. Important notes.



Not permitted. Misuse may cause damage.

Note.



Caution. Hot surfaces.



Isolate equipment from mains

3 INSTALLING THE R-4 FLOW REACTOR HEATER



Your Vapourtec R-4 Flow Reactor Heater can be installed by the User. Before the flow reactor heater is used this manual should be read.

3.1 Unpacking

Г	~ ^
	60

Carefully lift the R-4 out of the packaging and place on a firm surface. The glass heat exchangers are supplied in separate packaging to avoid damage.

3.2 Siting



For safety your R-4 must be sited within a fume cabinet or other suitably vented enclose. If it is decided to site the flow reactor heater in an open lab then the customer should undertake a thorough risk assessment prior to operation.



Do not store liquids on or above the reactor. Damage may result from spillage of liquids into the reactor. If spillage of liquids does occur, isolate the R-4 from mains.



Provide a firm surface for the reactor and check that the structure is adequate for supporting its weight.

Leave a minimum distance of 100 mm between the rear of the reactor and any solid objects. The R-4 requires this clearance to ensure adequate air flow through the reactor.



Do not block any of the ventilation panels. These are necessary to ensure adequate air flow through the reactor. Pay particular attention to the air intakes and exhausts located at the back of the reactor. These must not be covered.



If you need to move your R-4 Flow Reactor Heater caution should be exercised as it weighs 10 kg. To avoid damage, remove the glass heat exchangers before moving the R-4 Flow Reactor Heater.



For compliance with EN61000-3-3, the R4 must only be used in a location where the main electrical supply into the building is rated at > 100 Amps

3.3 Flow reactors product range



The reactors currently available range from small bore tube reactors through to large bore tube reactors, packed bed column reactors, microreactors & mixers, photochemical reactor (UV-150) and electrochemical reactor (ION). All the reactors are interchangeable on the R4 flow reactor heater.





Standard coiled tube reactor



- Excellent visibility of reactants
- Ambient to 150°C
- PFA, stainless steel or Hastelloy
- Strong acid resistance (with PFA)

Column reactor



Cooled column reactor



High temperature tube reactor



Micromixer chip reactor



- For heterogeneous reactions
- Excellent visibility of reactants
- Ambient to 150°C
- Sizes from 3ml to 20ml
- Glass and PTFE reactors
- Strong acid resistance
- Rapid temperature control

For heterogeneous reactions

- Excellent visibility of reactants
- -40°C to ambient
- Sizes from 3ml to 20ml
- Glass and PTFE reactors
- Strong acid resistance
- Rapid temperature control
- Double insulated for safety
- Ambient to 250°C
- Stainless steel or Hastelloy
- Acid resistance (with Hastelloy)
- Options for 50 bar or 200 bar
- For homogeneous reactions at small scale
- Excellent visibility of reactants
- -40°C to 150°C
- Seven reactor configurations available
- Use up to 8 reactors at one time
- Borosilicate glass reactor chips

Photochemical reactor



Cooled tube reactor



Heated mixing tube reactor



Either medium pressure mercury, low pressure Mercury or LED light sources

rtec

- Fully interlocked for safety
- -40°C to 80°C
- Up to 10ml reactors
- Wavelength filters available (for medium pressure mercury lamp)
- Strong acid resistance
- Good visibility of reactants
- -70°C to ambient
- PFA reactors
- Strong acid resistance (with PFA)
- Pre-cooling of 3 reagents
- Cooled mixers
- For temperature sensitive reagents
- Good visibility of reactants
- Ambient to 150°C
- PFA reactors only
- Strong acid resistance Pre-heating of 3 reagents
- Pre-nealing of 3 reage

20ml internal volume 3.2mm bore reactor Full length static mixers Strong acid resistant

Heated mixers

Large diameter tubular reactor for rapid mixing



ION electrochemical reactor



Options for integrated or standalone operation Ability to heat and cool the reactor -10° C to $+100^{\circ}$ C

Designed for bi-phasic liquid-liquid reactions

Fits into standard glass manifold Temperature range ambient to 150°C

- Operation at pressure up to 5 bar
- 20 electrode materials available

Flexibility in electrode spacing, electrode area and reactor volume



3.4 Overview of R4 Heater





Figure 2 R4 Reactor Heater (front view)



Figure 3 R4 Reactor Heater (side view)



Heating: Ambient to 150 °C for channels 1 & 3 Ambient to 250 °C for channels 2 & 4 (high temperature channel)

Cooling: -70°C to ambient for channels 1, 3 & 4 (with Vapourtec cooling module)

3.5 Assembly of column reactor into the heat exchangers



To avoid damage, the glass heat exchangers have been supplied within separate packaging. The column reactor is **preassembled** and ready to use.

nourtec

The procedure below is a guide for re-assembly the reactor if it has been taken apart for cleaning or to load catalyst into the column:

- 1. Remove the fixed and adjustable ends from the Omnifit HPLC column and unscrew the end caps.
- 2. Insert frits into the fixed and adjustable ends of the Omnifit HPLC column. Inspect and replace o-ring seals if necessary.
- 3. Place the Omnifit fixed end into the lower adaptor (see Figure 6) and screw onto the lower end of the column (see Figure 7).
- 4. Pack the column with solid support material (catalyst), if required.
- 5. Screw the upper adaptor onto the adjustable end and insert the adjustable end into the glass column (See Figure 8 and Figure 10).
- 6. Tighten the upper adaptor onto the glass column. Ensure that the adjustable end is screwed down to the correct position above the packing in the column. Turn the nut clockwise to move the adjustable end up and anti-clockwise to move the adjustable end down (See Figure 9).
- 7. Ensure the temperature sensor is removed from the glass heat exchanger.
- 8. Carefully slide the Omnifit HPLC column into the glass heat exchanger until the silicone retaining rings are both located within the glass heat exchanger.
- 9. Plug the temperature sensor into the socket provided to the right of the column position and turn to tighten (see photo), ensuring that the end of the sensor is located against the side of the Omnifit HPLC column.
- 10. Screw on the solvent connections as required to fixed and adjustable ends of the Omnifit HPLC column.



Please disconnect the temperature sensor <u>before</u> removing the Omnifit HPLC column from the heat exchanger to avoid damaging the sensor.



Figure 4 Column Reactor Assembly





Figure 5 Frits and O-rings



Figure 8 Screw upper adaptor onto adjustable end.



Figure 6 Fixed end in lower adaptor



Figure 9 Top adaptor and nut



Figure 7 Screw lower adaptor onto column



Figure 10 Insert upper adjustable end into column and screw down.



Figure 11 Column Reactor Complete Assembly



Remove the heat exchanger from R-4 before attempting to remove the Omnifit column from the heat exchanger to avoid damage to the glassware.



3.6 Assembly of the glass heat exchangers onto the reactor

Г	0 0
	60

- 1. Carefully locate the glass column reactor onto the front of the reactor by tilting the column and insert the air outlet tube (with magnet below it) into the exhaust blanking plate with one hand. While the air outlet tube is inserted, slightly push up the exhaust blanking plate.
- 2. Pull down the lever with the other hand and insert the air inlet tube. Gently release the lever to clamp the tube reactor in place.
- 3. Plug the temperature sensor into the socket provided to the right of the column position and turn to tighten (see Figure 12 and Figure 13).



Figure 12 Insert Column Reactor (a)



Figure 13 Insert Column Reactor (b)



Do not leave a temperature sensor connected when its heat exchanger is not located onto the front of the reactor heater.

Please disconnect the temperature sensor <u>before</u> removing the column from the heat exchanger to avoid damaging the sensor.



3.7 Changing a reactor cartridge in a glass tube reactor



To avoid damage, the glass tube reactors have been supplied within separate packaging. The procedure for disassembly (to change the reactor cartridge) is as follows:

- 1. If present, unscrew the temperature sensor from the side of the glass tube reactor.
- 2. Remove the large cap from the side of the glass tube reactor by unscrewing the small cap in the centre. See Figure 15 and Figure 16.
- 3. Carefully slide the reactor cartridge out of the glassware.
- 4. Slide a new reactor cartridge into the glass tube reactor, ensuring that the ends of the tubing are opposite the air inlet and outlet tubes on the reactor (see Figure 16).
- 5. Locate the large cap on the side of the reactor, using the glass pip for alignment. Take care to ensure that the tubing is not squashed against the glass (Figure 14).
- 6. Screw the location cap back onto the tube reactor.
- 7. Plug the temperature sensor into the socket provided on the side of the reactor, ensuring the end of the sensor is located against the reactor cartridge.



Figure 14 Tube Reactor Assembly



Figure 15 Unscrew the small cap and temperature sensor



Figure 16 Remove the large cap

vapourtec



Figure 17 Disassembled Standard Tube Reactor



Please disconnect the temperature sensor <u>before</u> removing the reactor cartridge from the tube reactor to avoid damaging the sensor.



When using PTFE or PFA reactor cartridges do not exceed the following temperatures/pressures:

Temperature	Pressure
Ambient - 40°C	40 bar
40 - 99°C	25 bar
100 - 150ºC	15 bar



Refer to section 9 for the temperature and pressure range for all the Vapourtec's reactors.

vapourtec

3.8 Assembly of tube reactor onto the reactor



To avoid damage, the glass heat exchangers have been supplied within separate packaging. The procedure for fitting the tube reactor to the R4 heater is as follows:

- 1. Carefully locate the glass tube reactor onto the front of the reactor by tilting the tube reactor and insert the air outlet tube into the exhaust blanking plate with one hand. While the air outlet tube is inserted, slightly push up the exhaust blanking plate.
- 2. Pull down the lever with the other hand and insert the air inlet tube. Gently release the lever to clamp the tube reactor in place.
- 3. Plug the temperature sensor into the socket provided on the side of the reactor and turn to tighten.



The air outlet tube is always at the top in the exhaust blanking plate.



Figure 18 Assembly of Tube Rector onto R4



Please disconnect the temperature sensor <u>before</u> removing the reactor cartridge from the tube reactor to avoid damaging the sensor.



3.9 Changing a reactor cartridge in a high temperature (HT) reactor



To avoid damage, the high temperature reactor has been supplied within separate packaging. The procedure for disassembly (to change the reactor cartridge) is as follows:

- 1. If present, unscrew the temperature sensor from the side of the glass tube reactor.
- 2. The reactor coil is attached on the reactor cover. To remove the reactor coil, unscrew the clamp knob at the centre to release the reactor cover. See Figure 20 and Figure 21.
- 3. Carefully remove the reactor cover (with coil attached) out of the glassware. See Figure 22.
- 4. There is a glass pip on the reactor glass body for coil alignment. Ensure that this glass pip is sitting properly inside the groove on the reactor cover before screw in the clamp knob. See Figure 22. Failure to do so might cause damage on the glass body.
- 5. Plug the temperature sensor into the socket provided on the side of the reactor, ensuring the end of the sensor is located against the reactor cartridge.



6. To locate the HT reactor onto the R4 heater, refer to section 3.8 above.

Figure 19 HT Reactor Assembly





Figure 20 Remove the temperature sensor and unscrew the clamp knob



Figure 21 Remove the reactor coil



Figure 22 Glass pip alignment for HT reactor



The R4 reactor heater shall heat the HT reactor from ambient to 250 °C in under <u>12 minutes</u>. If it fails to do so: 1) check the voltage supply to the R4, 2) check the fitment of the reactor coil and temperature sensor.



The recommended voltage for the R4 heater: 230V (+/- 10%), 50 Hz, **Or** 110V (+/- 10%), 60 Hz. If the incoming voltage is low, a step-up transformer is required.



If the reactor cover and temperature sensor fitment is incorrect, it will cause heat loss or temperature measurement error. This will reduce the R4 heating efficiency and take longer time (or unable) to achieve the target temperature. Ensure the glass pip is aligned correctly (refer Figure 22) before screw in the reactor coil (cover).



The hot liquid stream coming out from the HT reactor will be cooled down in the 1-meter cooling coil. It is safe to connect the standard PFA tubing to the outlet of the cooling coil.



It is recommended to fit the 250 psi BPR immediate after the 1-meter cooling coil to avoid boiling inside the HT reactor.

The PEEK 250 psi BPR (black BPR) has higher heat resistant than the acid resistant BPR (white BPR).

3.10 Electrical connections



Check the rating plate located to the rear of the reactor to ensure that the electrical supply you intend to connect to the reactor is suitable for your model of R4 heater.





For compliance with EN61000-3-3, the R4 must only be used in a location where the main electrical supply into the building is rated at > 100 Amps



The electrical connections should be made in accordance with the picture below. For detailed specifications of the serial and switch I/O connections please see the appropriate section in this manual.



When the R4 heater is used as part of an integrated R-series system, the connection to the R-series pump module is via the 9 pins RS232 interface.

~		~
6	ю	1
1	7	7

In an integrated R-series system, the R4 control knob can be used to control both the R-series pump module and the R4 heater. Refer to section 4.



Figure 23 R4 Heater side view





Figure 24 R4 connection interface



Figure 25 R2 to R4 connection

Vapourtec

4 THE USER INTERFACE

4.1 Description of the interface

The user interface is used for manual control of the R-4 (and the R2 pump module in an integrated R-series system, refer to R2 pump user manual). The control knob can be used to switch between three modes: OFF, SET and ON and is also used to set the required temperature for each channel.

4.2 Display of data during heating

On **ON** mode the display will show the actual temperature for each channel and not the target temperature set. The dots immediately below each display are cycling on and off to indicate the heating is turned on.

On **SET** mode, the display will show the current temperature setpoint with the active channel flashing.

<u>^ ^</u>	1
50	

On **OFF** mode, the display will show the current actual temperature if a temperature sensor is plugged in. The display will show '---', if no temperature sensor is present.

5 OPERATION

5.1 Setting the temperatures for R4 channels

Use the display for the reactors to switch ALL the channels between three modes; OFF, SET and ON. To achieve this control, the following operation is required:

One push of the control knob changes all displays to read SET.

Turning the control knob clockwise changes all displays to read ON.

Turning the control knob C-Clockwise changes the displays through SET to read OFF. The temperature of each of the 4 channels can be set independently in the range -70 °C to 250 °C (depend on reactor and channel used).

Caution. If at any time the control knob is pressed once but then no other action is taken the display will revert to actual temperature after 5 seconds.

Caution. Do not place anything other than the glass heat exchangers in the holes in the front of the R-4 as they may be hot.

From start-up the control works as follows:

- 1. After power-up all reactors start up displaying actual temperature (if a temperature sensor is plugged in) but behind this the system has remembered the previous set points.
- 2. Press the control knob all displays read SET.
- 3. Press the control knob again while SET is displayed to adjust the set point. If the heaters are on then they should remain on throughout the changing of the set point (unless a particular reactor is set to OFF) and will once again be ON after the set points are changed. If the reactor heaters were OFF before the set points were adjusted then after adjusting the set points the heaters will revert to OFF state.
- 4. Once the set points have been adjusted the displays will all revert to displaying the actual temperature. See illustration below.

5.2 Turning the heaters on

To turn the heaters on:

- 1. Press the control knob all displays read SET.
- 2. Turn the control knob clockwise until ON is displayed and then press.
- 3. The heaters are turned on and the displays show the actual temperature. In addition, while the reactors are changing in temperature the dots immediately below each display are cycling on and off. When the set temperature has been reached all three dots will flash in unison until the reactor has stabilised. Once a stable temperature has been reached the dots are not displayed at all. See illustration below.

5.3 Turning the heaters off

To turn the heaters off:

- 1. Press the control knob all displays read SET.
- 2. Turn the control knob C-clockwise until OFF is displayed and then press.
- 3. All heaters are turned OFF while the displays show actual temperature. See illustration below.

nurtec

Illustration of R4 control operation (Setpoint Input):

Caution. If at any time the control knob is pressed once but then no other action is taken the display will revert to actual temperature after 5 seconds.

pourtec

Caution. Do not place anything other than the glass heat exchangers in the holes in the front of the R-4 as they may be hot.

60/

Illustration of R4 control operation (Switching ON/OFF):

5.4 Configuring the reactor for automatic operation

The R4 reactor heater can be integrated with your flow chemistry system. The reactor heater is provided with serial data communication using RS232 protocols. With a suitable cable connected to the 9-way serial port the heater can be controlled remotely using the command set detailed in section 6.

When the R4 reactor heater is part of the R-Series integrated system, it can be controlled via the flow commander[™] software in manual mode or auto mode (with experiment setup). Refer to flow commander[™] user manual for details procedure.

nurtec

To operate the R-4 reactor heater manually using flow commander[™] software:

- 1. Press the connection button to connect the flow commander to the system. The button will turn green and shows 'Online'.
- 2. Press the box around the 'Reactor Temperature', this will bring up the input window.
- 3. Those R4 channels with no temperature sensor plugged in are greyed out. Input the temperature setpoint, click 'OK' to accept the new setpoint.
- 4. Click the 'Power' button to start the R-4 (and the R-series pump module if connected and turned ON).

	1) Turn 'Online' cy Stop	Connection Columnation Online System I Pump A Pump B 30 30 30 30 30 30 30 30 30 30 30 30 30
2) Input temp. setpoint	System Pressure 1 Pump A Pump B System Pressure 2 Pump C	4) Power ON set limit bar to air
Set Reactor Temperature	×	System II Pump C Pump D
Reactor Conditions		20 40 20 40 20 40
Reactor 1 Temperature	40 ÷ C ⊠ Powered	
Reactor 2 Temperature	20 C Powered	2) Set temp.
Reactor 3 Temperature	30 🛉 C 🗆 Powered	air
Reactor 4 Temperature	40 € C Powered	Pump Speed set Pump A: 0.000 ml/min Pump B: 0.000 ml/min Pump C: 0.000 ml/min Pump D: 0.000 ml/min
	Cancel OK 5	Reactor Temperature set 20/20 /20 24/off /40 /off /off /off /off

Figure 26 R4 control using Flow commander

rtec

6 INTERFACING THE R-4 FOR AUTOMATION

6.1 Overview of Serial Interface

The R-4 is equipped with two different interface options for communication between upstream processes or serial communication using RS232 protocols. The command sets for these interfaces are described in this chapter.

The RS-232 interface uses a 19200 Baud rate, 8 data bits, 1 stop bit and no parity for data transmission. The lead length connecting the RS-232 devices can be up to 3 m long but should be kept as short as possible to ensure reliable data transfer.

The cable requires a 9-pin female 'D' connector at both ends with all connections wired straight through.

Connector Description		Signal type	Requirement
9 way / 1 Connected to 4 & 6		Not Used	Not Used
9 way / 2 Transmit data		Output (RS-232)	RS-232 levels
9 way / 3 Receive data		Input (RS-232)	RS-232 levels
9 way / 4 Connected to 1 & 6		Not Used	Not Used
9 way / 5 Common		Ground (OV)	NA
9 way / 6 Connected to 1 & 4		Not Used	Not Used
9 way / 7 Connected to 9		Not Used	Not Used
9 way / 8 Connected to 7		Not Used	Not Used
9 way / 9 Not Used		Not Used	Not Used

The electrical requirement for this connector is given in the table below.

6.2 Switched I/O Interface

The cable requires a 25-pin female 'D' connector at both ends with all connections wired straight through.

Va

Connector	Description	Signal type	Requirement
25 way / 1	Input 1 +ve	Positive Input	10 – 15 V relative to Input 1 -ve
25 way / 2	+12V	12V @ 100 mA supply	
25 way / 3	Input 2 +ve	Positive Input	10 – 15 V relative to Input 2 -ve
25 way / 4	+12V	12V @ 100 mA supply	
25 way / 5	Input 3 +ve	Positive Input	10 – 15 V relative to Input 3 -ve
25 way / 6	+12V	12V @ 100 mA supply	
25 way / 7	Input 4 +ve	Positive Input	10 – 15 V relative to Input 4 -ve
25 way / 8	+12V	12V @ 100 mA supply	
25 way / 9	Output 1 A	Volts Free Contact	
25 way / 10	Output 2 A	Volts Free Contact	
25 way / 11	Output 3 A	Volts Free Contact	
25 way / 12	Output 4 A	Volts Free Contact	
25 way / 13	Not used	Not used	Not used
25 way / 14	Input 1 -ve	Negative Input	
25 way / 15	GROUND	Ground	
25 way / 16	Input 2 -ve	Negative Input	
25 way / 17	GROUND	Ground	
25 way / 18	Input 3 -ve	Negative Input	
25 way / 19	GROUND	Ground	
25 way / 20	Input 4 -ve	Negative Input	
25 way / 21	GROUND	Ground	
25 way / 22	Output 1 B	Volts Free Contact	
25 way / 23	Output 2 B	Volts Free Contact	
25 way / 24	Output 3 B	Volts Free Contact	
25 way / 25	Output 4 B	Volts Free Contact	

The electrical requirement for this connector is given in the table below.

pourtec

6.3 Command Protocol

6.3.1 General

Commands and responses are specified as human readable text. However, they are designed to be easily parsed by software for integration purposes. Controlling software should wait for a response before transmitting the next command.

Every command and response are terminated with a newline, carriage return, or a carriage return / newline pair.

6.3.2 Serial Port Settings

The port settings are as follows:

Baud: 19200

Bits: 8

Parity: None

Stop Bits: 1

Flow control: None

6.3.3 Control Commands

ST: Set Temperature Target

Sets the target temperature and optionally the ramp rate.

Syntax:

STchan temperature [rate]

Example 1:

ST0 80

Set the temperature target on channel 0 to be 80°C. The maximum ramp rate will be used. If the heater is running, the temperature will begin to change immediately.

Example 2:

ST1 90 20

Set the temperature target on channel 1 to be 90°C. The ramp rate will be 20°C per minute. If the heater is running, the temperature will begin to change immediately.

PN: Power on heater

Begins heating on one or all channels.

Syntax:

PN[chan]

Example 1:

PN

Power on all channels. They will begin to ramp to their target temperatures at the desired rate. If any of the channels are already on, they are unaffected.

Example 2:

PN3

Power on channel 3. It will begin to ramp to its target temperature at the desired rate. If it is already on, this command has no effect.

PF: Power off heater

Stops heating on one or all channels. R-4 User Manual - Rev 9 Syntax:

PF[chan]

Example 1:

PF

Power off all channels. They will begin to ramp down to ambient at the maximum possible rate. If any of the channels are already off, they are unaffected.

Example 2:

PF2

Power off channel 2. It will begin to ramp down to ambient at the maximum possible rate. If it is already off, this command has no effect.

6.3.4 Information Commands

GT: Get current Temperature

Gets the current temperature.

Syntax:

GTchan

Response Syntax when heater is powered:

Xtemperature

X is one of:

- H The channel is heating
- C The channel is cooling (to a specified target)
- F The channel is at the set point, but the temperature is fluctuating
- S The channel is stable at the set point
- U The channel is unpowered and cooling to ambient

Example 1:

GT0

H49.6

Get the temperature target on channel 0. The response indicates that the channel is heating and is at 49.6°C.

GV: Get Firmware Version

Gets version information for the reactor heater firmware.

Syntax:

GV

Response Syntax:

-TBD-

Syntax:

OK

This is a positive acknowledgement of the last command. It was accepted.

ER: Error

Syntax:

ERcode [description]

There was a problem interpreting the last command. An error code is provided for software to decode, followed by human-readable description.

ER1: The channel is unavailable because the temperature sensor is not connected

R-4 User Manual - Rev 9

ourtec

- ER2: The specified channel number is out of range; channels are numbered 0 3
- ER3: The specified temperature is out of range
- ER4: The specified ramp rate is out of range
- ER100: The command was unrecognised
- ER101: The command was incorrectly formatted
- ER102: The command was too big
- ER103: The command was sent before the previous command had completed

vapourtec

vapourtec

6.4 User serviceable parts

6.4.1 Replacing the fuse

Isolate the equipment from mains before removing ANY covers.

Remove the fuse cover onto the side panel (see picture below). For description of fuse function and specification see General Specifications.

6.4.2 Replacing the pinch valve

Isolate the equipment from mains before removing ANY covers.

- 1. Locate the pinch valve that needs replacing. Each valve is secured with 2 screws, above and below. These operate a quick release clamp system allowing easy removal.
- 2. Loosen but <u>do not completely remove</u> the screws, 2 or 3 turns will be more than enough. This will release the clamping mechanism and allow the valve to be removed.
- 3. Slide the valve out of the pump module a gentle twisting may be required. Attached to each valve is an inline connector that allows easy disconnection. Gently pull the valve and connector so they are outside of the unit.
- 4. Disconnect the connector, making sure the loose end <u>does not fall back inside</u> the unit.
- 5. Connect the new valve and slide the new valve back into the pump module in a reverse of the steps carried out earlier. Take care not to trap the wires.
- 6. When the valve is in the correct position, tighten the retaining screws.

Figure 27 Release the pinch valve clamp

Figure 28 Replace the pinch valve

6.5 Spare parts & Accessories listing

Vapourtec Part No.	Description of Spare Parts and Accessories
50-1193	Serial cable assy R2 to R4 straight
N/A	12V fuse
30-3247	Pinch valve

No	Part Number	Description
Complete	50-1095	6.6 mm x 150 mm Standard Column Reactor Assembly incl Omnifit part
column assembly	50-1388	10 mm x 150 mm Column reactor complete assembly incl Omnifit part
	50-1380	15 mm x 150 mm Column reactor complete assembly incl Omnifit part
Complete column	30-3291	6.6 mm bore x 150 mm length Fixed/Adjustable Omnifit p/n 006BCC-06-15-AF
assembly <u>without</u>	30-3292	6.6 mm bore x 150 mm length Fixed/Fixed Omnifit p/n 006BCC- 06-15-FF
manifold and thermocouple	30-3297	10 mm bore x 150 mm length Fixed/Adjustable Omnifit p/n 006BCC-10-15-AF
(50-1005/ 50-1013)	30-3301	15 mm bore x 150 mm length Fixed/Adjustable Omnifit p/n 006BCC-15-15-AF
	50-1003	Replacement temperature sensor for column reactor
(50-1005	Column reactor manifold and thermocouple (50-1003) for 6.6mm bore columns
(2)	50-1013	Column reactor manifold and thermocouple (50-1003) for 10mm OR 15mm bore columns
3	40-1924	Union - 1/4-28 PFA Vapourtec design (for 6.6mm bore columns) * * use 30-3512 for 10mm and 15mm bore columns
(4)	50-1073	Single adjustable end fittings for 6.6mm x 150mm column
	50-1075	Single adjustable end fittings for 10 x 150mm column

R-4 User Manual - Rev 9

No	Part Number	Description	
4	50-1077	Single adjustable end fittings for 15 x 150mm column	
	50-1420	Column Plunger Adjustable 6.6mm Assembly (includes O-rings and frit)	
5	50-1422	Column Plunger Adjustable 10mm Assembly (includes O-rings and frit)	
	50-1424	Column Plunger Adjustable 15mm Assembly (includes O-rings and frit)	
	30-3474	Replacement glass 6.6mm/150mm	
(6)	30-3514	Replacement glass 15mm/150mm	
\smile	30-3513	Replacement glass 10mm/150mm	
\frown	40-1055	Column Adapter 6.6 Long	
(7)	40-1555	Column Adapter 10 Long Fixed (Gen2)	
\smile	40-1067	Column Adapter 15mm Long	
\frown	50-1421	Column Plunger Fixed 6.6mm Assembly (includes O-rings and frit)	
(8)	50-1423	Column Plunger Fixed 10mm Assembly (includes O-rings and frit)	
	50-1425	Column Plunger Fixed 15mm Assembly (includes O-rings and frit)	
9 30-3512 Column fixed nut blue		Column fixed nut blue	
\frown	50-1071	Simriz O-ring 014 pack of 2 for 15mm column kit	
(10)	50-1070	Simriz O-ring 010 pack of 2 for 10mm column kit	
	50-1069	Simriz O-ring 007 pack of 2 for 6.6mm column kit	
	30-3393	PTFE Frits (30 micron) for 6.6mm bore column (pack of 20)	
(11)	30-3516	PTFE Frits (30 micron) for 10 mm bore column (pack of 20)	
	30-3452	PTFE Frits (30 micron) for 15 mm bore column (pack of 20)	

vapourtec

vapourtec

50-1009 Standard tube reactor complete assembly (ambient to 150°C)

No	Part Number	Description
	50-1009	Standard tube reactor assembly (ambient to 150°C)
	50-1094	Standard Reactor Assembly with S/S Tube Reactor 10ml
	50-1263	Standard tube reactor - no coil fitted
	50-1010	Temperature sensor for tube reactor
2	40-1050	Tube reactor glassware
3	40-1040	Tube Reactor Bottom Adaptor
4	40-1903	Top adaptor
	50-1011	10ml replacement PFA tube cartridge
	50-1012	5ml replacement PFA tube cartridge
	50-1036	2ml replacement PFA tube cartridge
(\overline{c})	50-1079	10ml copper coil (150°C)
\odot	50-1091	5ml copper coil (150°C)
	50-1092	2ml copper coil (150°C)
	50-1228	2ml stainless steel 150°C reactor coil (coil only, potted)
	50-1229	5ml stainless steel 150°C reactor coil (coil only, potted)
	50-1230	10ml stainless steel 150°C reactor coil (coil only, potted)
	50 1222	2ml stainless steel 150°C reactor coil (coil only, potted) 100 bar (stainless
	50-1252	steel fittings)
	50 1233	5ml stainless steel 150°C reactor coil (coil only, potted) 100 bar (stainless
	30-1233	steel fittings)
	50-1234	10ml stainless steel 150°C reactor coil (coil only, potted) 100 bar (stainless
	00-1204	steel fittings)
	50-1250	25ml stainless steel reactor for use in 50-1009 150°C (2.5 mm bore)
	50-1251	20ml stainless steel reactor for use in 50-1009 150°C (2.5 mm bore)

		vapoa	000
No	Part Number	Description	

10ml replacement standard Hastelloy coil

50-1120 High temperature tube reactor assembly (ambient to 250°C)

No	Part Number	Description
	50 1120	High temperature tube reactor assembly (ambient to 250°C) – stainless
	50-1120	steel coil
	50-1471	HT Hastelloy Reactor Comp Assy, 10ml
	50-1087	Copper Reactor complete assembly High temperature
	50-1010	Temperature sensor for tube reactor
2	40-1050	Tube reactor glassware
3	40-1040	Tube Reactor Bottom Adaptor
4	40-1903	Top adaptor
5	50-1134	25 ml replacement stainless steel tube cartridge for high temperature reactor
	50-1121	10 ml replacement stainless steel tube cartridge for high temperature reactor
	50-1122	5 ml replacement stainless steel tube cartridge for high temperature reactor
	50-1123	2 ml replacement stainless steel tube cartridge for high temperature reactor
	50-1080	10 ml replacement copper coil cartridge for high temperature reactor
	50-1088	5 ml replacement copper coil cartridge for high temperature reactor
	50-1089	2 ml replacement copper coil cartridge for high temperature reactor
	50-1236	5 ml replacement Hastelloy reactor cartridge for high temperature manifold
	50-1157	2 ml replacement Hastelloy reactor cartridge for high temperature reactor
	50-1237	10 ml replacement Hastelloy reactor cartridge for high temperature reactor

(5)

50-1238

1

Janoi

-0

Part Number Description No 50-1292 high temperature micro bore reactor cartridge 1ml SS 50-1129 High Temperature Reactor (Hastelloy C) 2ml comp assy 5 50-1157 High Temperature Reactor (Hastelloy C) 2ml replacement High Temperature tube reactor (no coil) 250C 50-1127 6 30-3165 check valve flow in 3315 50-1127 High temperature tube reactor (no coil)

 $\mathbf{\nabla}$

7 TROUBLESHOOTING

Problem	Possible Cause	Solution
No display	System not plugged in and turned on	Check system is plugged in and turned on.
	Fuse needs replacing.	Replace fuse as described in Section 6.4.1 of this manual.
	If the cooling fans at the back is working. The firmware has corrupted.	Call Vapourtec Ltd for a service visit.
Temperature Display reads ' SSR ERR ' (flashing)	Obstruction in one of the fans.	Turn R-4 off at mains connection. Check all fans are free from obstruction.
	Fan broken or damaged	Call Vapourtec Ltd for a service visit.
	Electronic circuit failure	Call Vapourtec Ltd for a service visit.
Temperature Display reads ''	Temperature sensor not detected by the system.	Check temperature sensor is connected. Replace if damaged.
Slow heating	Low voltage supply	Check the voltage, step-up transformer required.
	Incorrect fitment of temperature sensor or reactor.	Check the reactor fitment and placement of the temperature sensor.

irtec

8 GENERAL SPECIFICATIONS

Independent Heating Zones	4
Temperature Range	-70 °C to 250 °C (depending on reactors fitted and channel used)
Locations for either column type or tube type reactors	4 (all channel)
Location for Photochemical reactor UV-150	1 (channel 3, with channel 2 used as reactor support)
Locations for cooled reactors	3 (channel 1, 3 & 4)
Locations for high temperature (250 °C) reactors	2 (channel 2 & 4)
Tube type reactor max working	10ml (standard PFA tube)
volume	20ml (PFA rapid mixing reactor)
	25ml (large bore stainless steel tube)
Column Type reactor dimensions	- 6.6mm ID x 150mm total length
	- 10mm ID x 150mm total length
	- 15mm ID x 150mm total length
Control interfaces: Switched I/O Inputs Switched I/O Outputs Serial	4 off 5V sensor inputs TTL level 4 off voltage free contacts, 0 – 48 V, 0.5 A max RS 232 communication standard
Environmental	Operational ambient temperature range: 15 to 25 C Operational humidity: 20 to 70% RH
Size & Weight	Width: 350 mm Height: 230 mm Depth: 280 mm Weight: 10 kg
Services	Power; 230V (+/- 10%), 50 Hz, 5A (see rating plate)
	Or, 110V (+/- 10%), 60 Hz, 10A (see rating plate)
Fuses	IEC socket (230 V external): 6.3 A, 20 mm,
	type I IEC socket (110 V external): 10 A, 20 mm, type T
	Low voltage fuse holder (12 V): 8 A, 20 mm, type T
Conformity	Conforms to all applicable EEC standards, CE marked.

vapourtec

Maximum Safe Operating Pressu	res for Reactors in V	apourtec Flow Chem	iistry Systems		
Tube reactors (all standard		Rea	actor temperature rar	ıge	
pressure systems)	-70°C to -20°C	-20°C to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
PFA tube reactors (and PFA supply tubing)	40 bar (580 psi)	40 bar (580 psi)	25 bar (362 psi)	15 bar (217 psi)	Do not use
UV-150 Photochemical reactors	10 bar (145 psi) Min Temp: -40°C	10 bar (145 psi)	10 bar (145 psi) Max Temp: 80°C	Do not use	Do not use
Stainless Steel Copper Hastelloy®	42 bar (609 psi)	42 bar (609 psi)	42 bar (609 psi)	42 bar (609 psi)	42 bar (609 psi)
Tube reactors (High		Rea	actor temperature rar	ıge	
Pressure, 200 bar systems)	-70°C to -20°C	-20°C to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
PFA tube reactors (and PFA supply tubing)	40 bar (580 psi)	40 bar (580 psi)	25 bar (362 psi)	15 bar (217 psi)	Do not use

REACTOR TEMPERATURE AND PRESSURE RANGE

200 bar (2900 psi)

Min Temp: -40°C

10 bar (145 psi)

UV-150 Photochemical reactors

Stainless Steel

Hastelloy®

Copper

Do not use

Do not use

10 bar (145 psi) Max Temp: 80°C

10 bar (145 psi)

42 bar (609 psi)

- (Cont.)
Systems
Chemistry
pourtec Flow
Reactors in Va
Pressures for
e Operating
Maximum Saf

Column reactors (Used on		Rea	actor temperature rar	ıge	
any Vapourtec system)	-40°C to -20°C	-20°C to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
6.6 mm Bore Columns	20 bar (290 psi), Silicone O-rings only	40 bar (580 psi)	30 bar (435 psi)	20 bar (290 psi)	Do not use
10 mm Bore Columns	20 bar (290 psi), Silicone O-rings only	30 bar (435 psi)	25 bar (362 psi)	15 bar (217 psi)	Do not use
15 mm Bore Columns	20 bar (290 psi), Silicone O-rings only	20 bar (290 psi)	15 bar (217 psi)	10 bar (145 psi)	Do not use
Chip Reactor (Used on any		Rea	actor temperature rar	ıge	
Vapourtec system)	-40°C to -20°C	-20°C to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
All Chip Reactors	20 bar (290 psi)	20 bar (290 psi)	20 bar (290 psi)	20 bar (290 psi)	Do not use
	-	Rea	actor temperature rar	ıge	
lon electrochemical reactor (Used on any Vapourtec	-40°C to -10°C	-10°C to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
system)	Do not use	5 bar (72.5 psi)	5 bar (72.5 psi)	Do not use	Do not use
l arge diameter tubular		Rea	actor temperature rar	lge	
reactor for ranid mixing	-70°C to Room Temp.	Room Temp. to 40°C	40°C to 99°C	100°C to 150°C	150°C to 250°C
(Used on any Vapourtec system)	10 bar (145 psi) Require External Chiller	10 bar (145 psi)	10 bar (145 psi)	10 bar (145 psi)	Do not use

10 VAPOURTEC WARRANTY

10.1 Standard limited warranty

The Vapourtec Ltd standard UK warranty follows. The warranty covers parts and labour for a period of 12 months, commencing the date of invoice, for any repairs deemed resultant of a defect in materials and/or workmanship by Vapourtec Ltd. This warranty excludes wear and tear of parts considered to be 'consumable', a list of these parts is given below. Replacement of consumable parts or repairs to equipment that is not covered by this warranty will be chargeable.

Any factory approved changes or extensions to this warranty should be received in writing from Vapourtec Ltd and filed with this warranty statement. If your equipment is eligible for coverage, please review this warranty thoroughly and contact Vapourtec Service Department with any questions you may have. If your equipment is not covered by our standard warranty, or you are seeking optional or additional coverage, see sections below for service plans offered.

Consumable parts and other items not covered by the standard warranty:

- Glass heat exchangers
- Temperature sensors

Items COVERED by the limited warranty

- Parts and labour for a period of one (1) year from date of delivery. Any part excluding those in the list above found to defective will be either repaired or replaced at the discretion of Vapourtec Ltd, free of charge by Vapourtec Ltd.
- On site labour if repairs require that Vapourtec Ltd personnel travel to the equipment.

Items NOT COVERED by the limited warranty

- Travel time, travel expenses and mileage expended by Vapourtec Ltd personnel if repairs require on-site labour.
- Transportation of equipment for repair.
- Vapourtec Ltd cannot be held responsible for incidental or consequential damages

The above statement is a final and complete statement of the agreement between the Customer and Vapourtec Ltd. Vapourtec Ltd makes no other warranties expressed or implied, of merchantability, fitness or otherwise, with respect to the goods supplied under this agreement, which extend beyond the description of this limited warranty.

Vapourtec Ltd will have the right to inspect the equipment and determine the repairs or replacements necessary. The customer will be notified within a reasonable time of any damages incurred that are not covered by this warranty prior to initiation of such repairs.

Any customer modification of this equipment or any repairs undertaken without prior written consent of Vapourtec Ltd will render the limited warranty void.

nurtec

10.2 Service contracts

Vapourtec recognises that by choosing Vapourtec R Series Flow Chemistry equipment you are investing both capital and resources. Our customer support program is designed to help you protect the value of your investment by prolonging the usable life of the equipment and maximising system performance & up-time in your laboratory.

For your convenience we present a range of support contracts to suit your budget and working requirements. Often our contracts provide more cost-effective cover than insurance, with faster response and no risk of refusal of cover in subsequent years.

Service and Breakdown Contract (SBC)

The SBC is a full service breakdown contract that includes preventive maintenance and all parts and labour. It is ideal for heavily trafficked or multi-user instruments in high throughput environments and guarantees peace of mind throughout the year. All service call outs and a scheduled preventive maintenance visit are included. If you do need to call in an engineer, there is no call out fee and all parts and labour are included. This is the best option for the busy laboratory where up time is at a premium.

Includes:

- One preventive maintenance visit per year
- All call-out, travelling time and labour charges *
- All parts needed for PM or Call out visits, inc. full Service Kit but excluding the items in the section below titled exclusions
- Average 48-hour response time
- · Attractive discounts for multiple systems on the same site
- Access to Vapourtec on-line technical support
- Free software downloads to keep your copy of Flow Commander updated

Exclusions:

- Glassware
- Reactor and reactor components
- Replacement back pressure regulators
- Replacement pump check valves
- Replacement of sealing components for the injection valves except during the annual PM when these parts are replaced. This applies to the R2PLUS only

* Please note, clearing of blockages caused by foreign objects or precipitation of compounds will be attended but travelling time and labour charges may be charged at Vapourtec discretion.

Preventative Maintenance Contract (PMC)

Our PMC is a popular option with budget-conscious laboratories that cannot afford the inconvenience of an unscheduled stoppage. It includes a planned maintenance visit and the cost of all parts needed during that visit and labour costs. Call-outs are charged at normal rates.

Includes:

- One preventive maintenance visit per year, including cost of full Service Kit of parts, travelling time and labour
- Average 48-hour response time
- Attractive discounts for multiple systems on the same site
- Access to Vapourtec on-line technical support
- Free software downloads to keep your copy of Flow Commander updated

Discount Schedule for SBC and PMC

If you have more than one Vapourtec system included in your Service Contract you will be eligible for attractive discounts that help stretch your service budget further. The systems are assessed for discount in installation date order, so your newest systems attract the highest level of discount.

1st (oldest) system = List Price

2nd system = 15% discount

3rd system = 20% discount

4th system = 25% discount

5th system = 30% discount

All subsequent systems = 30% discount

On-site Training of your own engineers.

For multiple installations of Vapourtec equipment it may be advisable to have your own engineers or technicians trained to provide first-line service diagnostics and repairs. Please contact Vapourtec if this is of interest.

Annual Training/User support Contacts

Training/User support contracts are offered on a 12 monthly basis, and cover on site and telephone support for users with application enquiries.

These contracts are based on 12 days per annum training/user support for Vapourtec system users. Please contact Vapourtec if this is of interest.

10.3 Sample copy of EC declaration of conformity EC declaration of conformity Product Vapourtec R-4 Flow Reactor Heater Serial no. Manufacturer Vapourtec Ltd Park Farm Business Centre Address Fornham St Genevieve Bury St. Edmunds Suffolk, IP28 6TS We hereby declare that the product above complies with the essential health and safety requirements of the following directives: MACHINERY Directive 2006/42/CE Implemented in the UK by Supply of Machinery (Safety) Regulations 2008 (SI 2008 no. 1597). Low Voltage Directive (LVD) Directive 2014/35/EU Implemented in the UK by The Electrical Equipment (Safety) Regulations 2016 (SI 2016 no. 1101). Electromagnetic Compatibility Directive 2014/30/EU (EMC) Implemented in the UK by The Electromagnetic Compatibility Regulations 2016 (SI 2016 no. 1091). **CE marking** Directive 93/68/EEC Implemented in the UK by The EMC (Amendment) Regulations (1994 no. 3080). The product has been designed and manufactured in accordance with European standards: EN 12100-1: 2004 Safety of Machinery: Basic concepts, general principles for design. Part 1: Basic terminology, methodology EN 12100-2: 2004 Safety of Machinery: Basic concepts, general principles for design. Part 2: Technical principles EN 13849-1: 2016 Safety of Machinery: Safety related parts of control system. Part 1: General principles of design EN 60204-1: 2006 Safety of Machinery: Electrical equipment of machines. Part 1 General requirements EN 61010-1: 2013 Safety requirements for electrical equipment for measurement, control and laboratory use Part 1: General requirements

A Technical Construction File is retained at the manufacturer's address.

Signed	
Name	
Position	
Date	

11 VAPOURTEC CONTACT DETAILS

Address: Vapourtec Ltd Park Farm Business Centre Fornham St Genevieve Bury St Edmunds Suffolk IP28 6TS U.K.

+44 (0) 1284 728659
+44 (0) 1284 728352
service@vapourtec.com
www.vapourtec.com