

VSD033

Knauer 50D UV detector installation on Vapourtec R-Series System



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1 INTRODUCTION

The Vapourtec R-Series software features the facility to graphically display axial dispersion that takes place within tube reactors, so that it is possible to automatically predict, (and correctly time the collection of) the steady state part of the reaction product stream when it emerges from the reactor. This means it is possible to collect the optimum amount of good product with the minimum waste of reagents, and it is possible to determine exactly how much good product can be made from a limited amount of substrate.

However, there are some circumstances (for example with heterogeneous columns) where either the dispersion is hard to accurately model (because the flow characteristics of the column contents simply cannot be known in advance), or else the working volume of the column reactor (the volume of the cavities between the substrate) is not accurately known. In these cases, it may not be possible to simply "predict" the location or shape of the peak, and so perfect collection timing cannot be assured. To overcome this, an in-line UV detector can be used to measure the UV absorbance of the fluid flow and provides a continuous signal that can be used to quantify the amount of chromophoric compounds emerging from the reactor. This UV signal can then be used to automatically trigger the collection of the (good) product according to the conditions set.

The triggering conditions might be based on either the level or the gradient or both. Criteria can be selected per reaction (i.e. a different trigger level can be set for each reaction in a sequence if need be). Collection will then proceed according to these criteria.

The Knauer 50D multiple wavelength UV detector is integrated to the Vapourtec R-Series via the touch panel USB port (any of the 3 available). The following sections elaborate the procedure on how to setup and control the 50D detector via Vapourtec R-Series system.

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.



Isolate equipment from mains

Caution. Hot surfaces.

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Note.

3 TOOLS

- USB/Serial converter (Vapourtec Part Number: 30-3596)
- Serial cable (supplied with detector)

4 INSTALATION / PROCEDURE



The 50D detector is usually installed next to the Vapourtec R-Series system. For those who has limited space in the fume hood, a special equipment frame can be purchased to elevate the R-series system and installed the 50D detector underneath the R4 module. See drawing below.





Connect the UV detector serial cable (supplied) to the Knauer UV detector at the rear of the device, in the port labelled "RS-232"





Using non-original serial cable might fail to connect the detector.



Connect the USB/serial converter to the RS232 cable, and then plug it to the touch panel USB port.



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60^ M Knauer Analytical A4042 Flow Cell is supplied and fitted as standard. A set of inlet/outlet tubing with PEEK adaptor is supplied. The inlet tubing is 50cm and the outlet is 75cm.

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The 50cm inlet tubing length is pre-set in the R-Series control software, user <u>do</u> <u>not need</u> to input this tube length in the software.

However, the outlet tube length of 75cm need to be input to the software for accurate flow control. See section below on software control for details.



4.1 R-Series Software Setting and Control



Power on the UV detector and R-Series system. In Flow Commander[™], browse to '**Setup**' -> '**System configuration**' -> '**UV/Vis Detector**', and select the detector type as "50D serial"

Flow Commander 1.12		
File Experiment Logs	Setup Detector Tools Help	Enter system
Charts Exporimont	System Configuration 🔸	configuration
Charts Experiment	Transfer Settings	
🔍 🔍 Reactor View	Install License	Point 🝷
0.1	View Installed Licenses	
0.09		
0.08		

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The 50D detector comes with 4 detection channels. The detection wavelenght for each channel can be set in the range of 190nm – 900nm.



Detection channel 1 (Wavelength 1) must <u>ALWAYS</u> be selected in order to activate the 50D detector.



The 50D detector shall be connected after the reactor and before the back pressure regulator (BPR). Refer to picture below for the tubing connection.



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User has to input the correct tubing length to the software to ensure the collection timing is accurate.

By default (see picutre below):

- The 'Tubing before Collection Valve' is set as 53 cm. This length consist of 50cm waste/collect tubing plus 3cm length of the BPR.
- The 'Tubing before UV/Vis Detector' is set as 50 cm. This is the length of the inlet tubing to the 50D detector.



Presumably a 75cm tubing is used on the detector outlet (standard length supplied), user can either:

- Increase the 'Tubing before collection valve' to 128cm OR
- Increase the 'Tubing before UV/Vis Detect' to 125cm



Users are free to choose what length of tubing to be used to connect the UV detector, provided the <u>total tubing length</u> is set correctly on the control software.

	System Configuration		×
Browse to 'Tubing'	System Configuration General Manifold Tubing Loading and Cleaning External Chiller UV / Vis Detector Mass Flow Controller Column Compression Ion Controller Fraction Collector Autosampler GSIOC Settings	Tubing before Collection Valve 53 cm length 1 mm bore When not using a UV/Vis detector, this is the length from the last reactor to the R2 Collection Valve. When using a UV/Vis detector, this is the length from the Detector to the R2 Collection Valve. Tubing before UV/Vis Detector Default setting = 53cm 50 cm length 1 mm bore When using a UV/Vis detector, this is the length from the last reactor to the detector. Default setting = 50cm	×
		Cancel OK	

60/

Once the configuration is completed, create the experiment and choose to use a UV/Vis detector. See picture below.

Edit Experiment		>
Reactor Setup		
Pressure Limit		
System Pressure Limit 10.0 🔹 bar		Cancel
Secondary Limit 10.0 🗘 bar		
Select UV/Vis	Detector	a a Datak
		<< Back
	Flow Control	
	 Specify Residence Time and Volumetric Ratios 	Next >>
UV/Vis Detection	 Specify Residence Time and Stoichiometric Ratios 	
O Not using detector	 Specify Flow Rates 	
Use UV/Vis Detector		
Collection	Solvent volume used for	



While the reaction conditions are being set, select UV triggered collection as in picture below and then select 'setup'.

Add Protocol				3
Add New Reaction Reagents	Name Reactor	Advanced Conditions	Cancel	ОК
A B C D Flow Rates 1.000 1 1.000 1 0.000 0 0.000 0 ml / min Residence in Reactors ⊇ 1 Residence Time 5m0s Loop A M Loop B M Loop C M Loop D M	Collectio Auto	n V Includ	30 ÷ C	ng edges Setup
Scale Delay ml Width x Reagent A 1.00 * ml 1.00 ml/min 0.0 * Reagent B 1.05 ml 1.000 ml/min 0.0 * 1.0 * Reagent B 0.00 ml 1.000 ml/min 0.0 * 1.0 *		/		
Reagent D 0.00 ml 0.000 ml/min Activ	vate 'U'	√ Trigge	red Colle	ection'
view: % Steady State · Curves: All A B	C D			
100%				
0% 5 6		ļ,		
				Time (mir



User can now define the trigger criteria by defining the starting trigger and ending trigger point.





When 'UV Triggered Collection' is activated, the R-Series control software will not trigger the collection valve or collector (fraction collector or autosampler) until the pre-set threshold has been achieved.



If any further assistance is required please contact Vapourtec support directly.

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Address: Vapourtec Ltd Park Farm Business Centre Fornham St Genevieve Bury St Edmunds Suffolk IP28 6TS U.K. Tel: +44 (0) 1284 728659

 Fax:
 +44 (0) 1284 728053

 e-mail:
 info@vapourtec.com

 Web:
 www.vapourtec.com