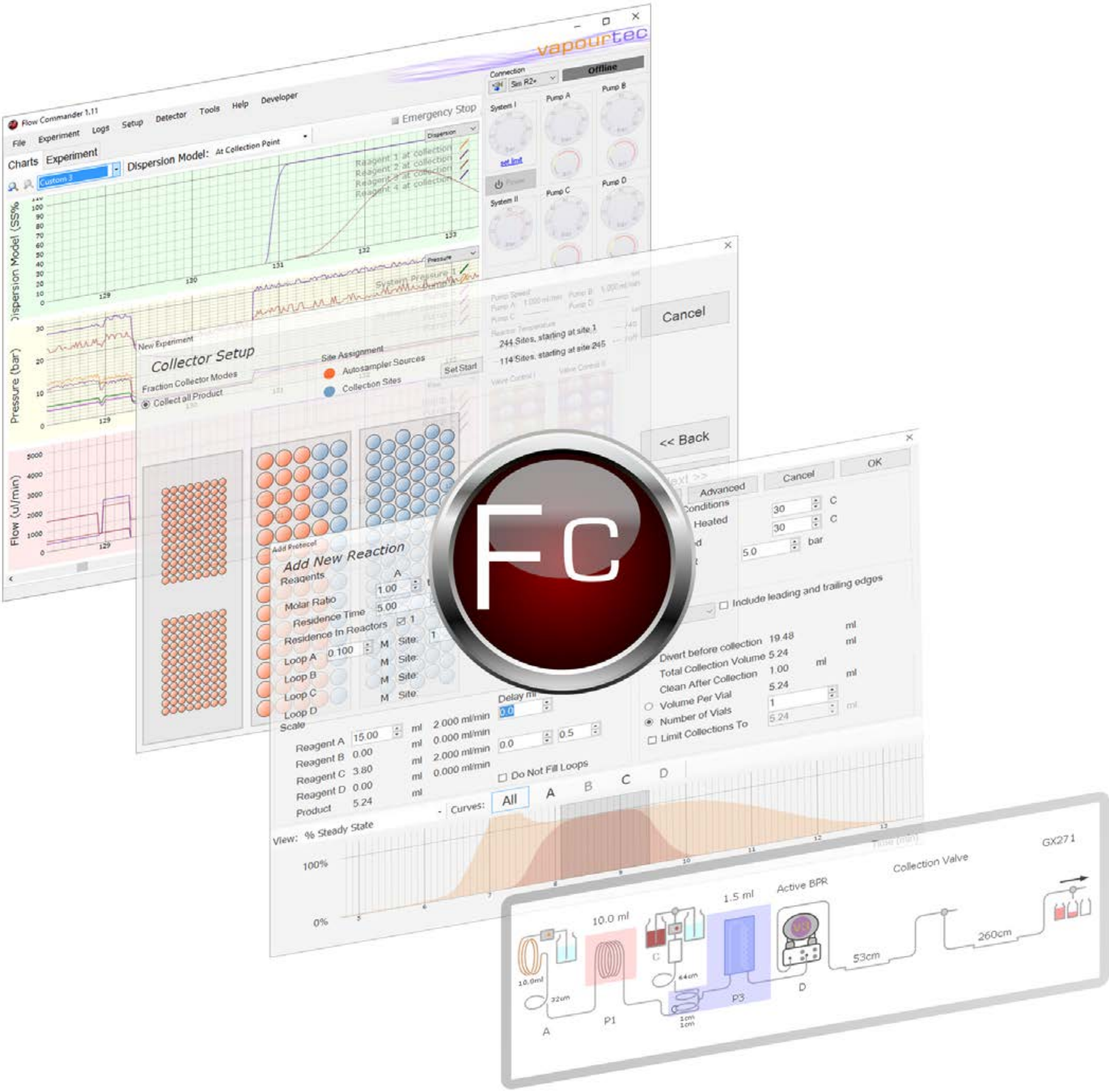

Flow Commander User Manual

Issue 2

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Scope of Document

This User Manual describes how to use the Vapourtec Flow Commander™ application software that runs on the Vapourtec Touch Screen and Microsoft® Windows® PC to control the Vapourtec R-Series flow chemistry system.

This User Manual does not cover the Flow Wizard™ application software.

Refer to the documentation provided with the R-Series system and also external components for information on equipment-specific usage and configuration.

Document History

Issue Number	Date	Description of Changes
1	Jan 2019	Initial publication for Flow Commander version 1.10.0.4
2	Apr 2019	Updated to reflect changes to version 1.11.1.3.

Safety information

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

Attention. Important notes.

Not permitted. Misuse may cause damage.

Note.

Copyright and trademark information

Flow Commander is a trademark of Vapourtec Ltd.

Gilson, **151**, **152**, **PrepFC**, **FC203B**, **FC204**, **GX271**, **402**, **401**, and **GSIOC** are trademarks of Gilson, Inc.

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1 Introduction

Flow Commander™ is powerful, feature-rich software that provides complete automation of the Vapourtec R-Series platform and associated external equipment.

By automating all the mundane set up and calculation steps, and by providing simplified steps to configure experiments and reactions, Flow Commander maximises productivity in the flow chemistry laboratory.

Flow Commander performs automated reactions in a reliable, and repeatable manner; reactor temperature is allowed to stabilise before beginning the reaction, the system is monitored for over-pressure conditions and leaks, and all tubing is cleaned after reactions are complete.

Flow Commander includes a unique and sophisticated reagent dispersion model that allows the user to identify when steady-state concentration will be reached during the reaction. For reactions using reactors in between pumps, the dispersion model ensures concentration peaks from each pump are matched in time, minimising usage of precious reagents.

1.1 Key Features

- Define reactor and tubing layouts with a simple graphical tool
- Automate low-level system calculations and routine tasks, reducing human error
- Quantify reactor behaviour with multiple data sets
- Dispersion modelling accurately predicts steady state conditions
- Save experiments enabling precise repetition and sharing
- Share reaction conditions across platforms and sites
- Run long duration optimisation series unattended
- Instantly scale up the throughput of proven reactions by simply changing to a larger reactor type
- Instantly scale up the volume of product by simply increasing the volume of reagent used
- Interface to third-party detection, autosamplers and fraction collection systems
- Log and store data for producing charts and reports
- “Walk up” open access interface for the less experienced user
- Access all features when offline (except Run Experiment)
- Control the system manually
- Control the system remotely using a mobile device or tablet
- Use external software to control the system using the high level Developer API (Application Programming Interface)

1.2 R-Series System

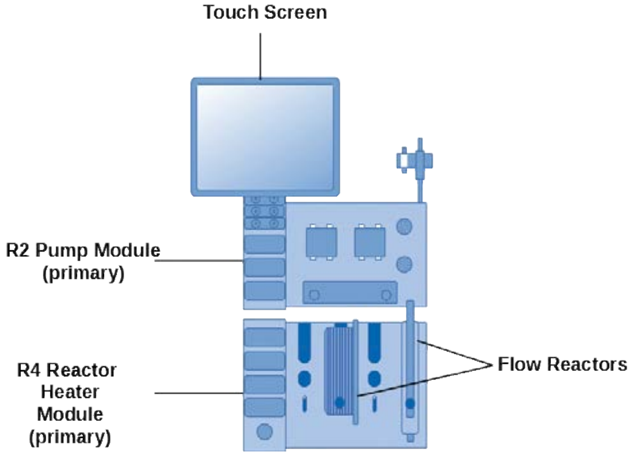


Figure 1.1: Example Basic Vapourtec R-Series Flow System

Support for an additional R2 pump module is available through the Flow Commander Professional Extension Licence.

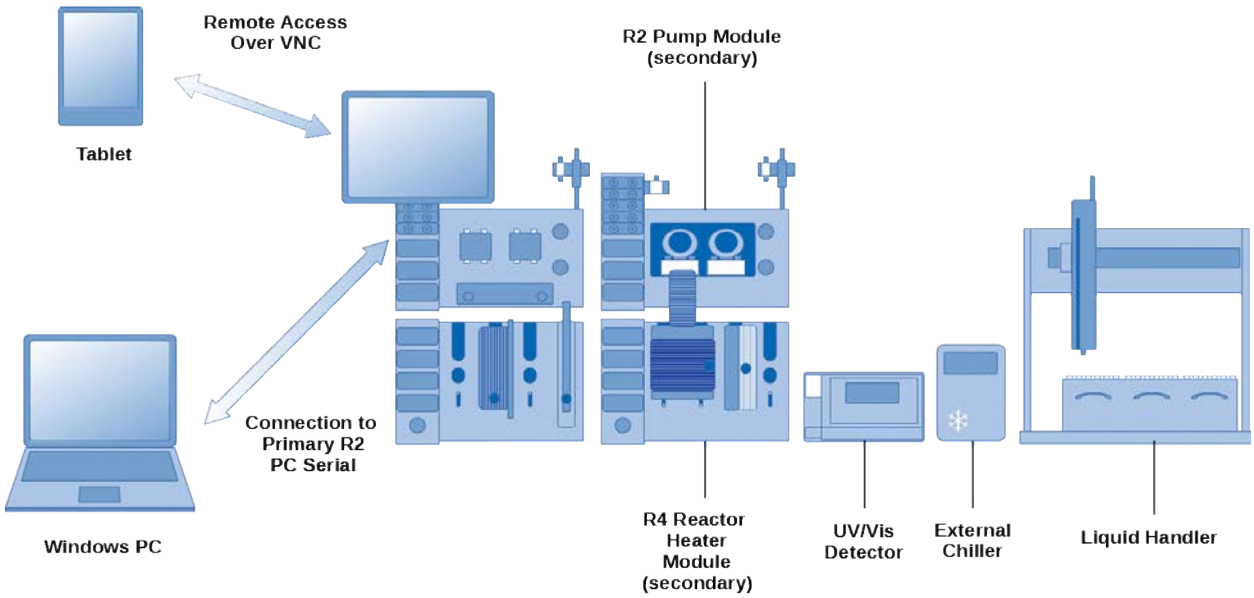


Figure 1.2: Example Advanced Vapourtec R-Series Flow System

1.3 Integrated features

Reaction automation

Experiments running Flow Commander can automate multiple reactions for each experiment, and wait for reactors to reach the correct temperature before commencing the reaction. A series of reactions with differing conditions can be set up in advance and allowed to run without user intervention.

As leaks and blockages occurring during experiments can be automatically detected by Flow Commander, the system can be left unattended.

Dispersion modelling

Axial dispersion of the reagent occurring in tubing and reactors is accurately modelled and displayed during reaction set-up. The user can verify that steady-state reactor concentration will be achieved, and an appropriate collection window can be selected that collects the product without undue waste or excessive dilution of the reagent.

For set-ups involving reactors in between pumps, the dispersion model allows the downstream pump to be started after a delay to synchronise reagent peaks.

Liquid handler integration

Flow Commander can communicate with a large number of flow chemistry devices, allowing reactions to be carried out with a range of reagents over differing stoichiometric, temperature, and residence time conditions.

- Autosampler can be used to load a sample loop prior to any automated reaction
- Fraction collectors can be used to collect any automated reaction

Flow Commander allows the user to specify how the reaction output is distributed across available vials in the fraction collector.

Triggering collection from an optical sensor

The output levels of a UV or IR sensor; level, gradient or both, can be used to control the collection valve downstream of a reactor.

Manual control

Flow Commander provides full control of the connected R-Series hardware including reactor temperature, flow rates, pressure trip limits, valve positions of reagent/solvent, sample load/inject and collection, all without needing to touch the physical buttons and control knob – ideal for experiments using hazardous chemicals.

Determine total consumables required

Flow Commander calculates the amounts of reagents and solvents required to perform the sequence of reactions for each configured experiment. The user can specify the reaction scale as well as which parameters are of greater importance in the calculation e.g. residence reactor time versus flow rate.

Logging and charting

Reactor temperatures, pressures, flow rates, valve positions, cooler power, and other conditions are charted in real-time, and the recorded data can be exported to a PDF or CSV file to facilitate analysis after the reaction is complete.

Flow Wizard™

Once the system set-up details has been configured using Flow Commander, a user with little or no knowledge of the system can use the Flow Wizard software running on the touch screen to run single reactions. Flow Wizard features a “Walk up” open access interface using step-by-step screens to capture experiment and reaction information, and inform the user of progress. See the *Flow Wizard User Manual* for more information.

PDF report generation

Flow Commander generates PDF reports which include charts and system configuration, for printing or storing in electronic notebooks.

Customised experiments

Flow Commander features a tubing layout editor with schematic display allowing reagent dispensers, reactors, pumps, detectors, samplers and collectors to be connected together to accurately reflect real laboratory set-ups. With a few simple clicks, a whole range of automated reactions can be created for optimisation or library synthesis.

Reagents may pass through heated reactors before being mixed with other reagents, and bottled reagents may be used in the same set-up with reagents in sample loops.

New reactions can be based on saved configurations and by simply changing a few parameters, the user can rapidly create a large set of unique experimental conditions to run.

Support for up to 4 pumps

With the Professional Licence Extension, Flow Commander models the dispersion and automates complex, customised set-ups involving 1 to 4 pumps, calculating pump and valve timing to synchronise the reagent peaks.

Flexible support for flow chemistry components

Flow Commander supports numerous flow chemistry components including reactors, heaters, coolers, fraction collectors, autosamplers, UV detectors, IR sensors, available from Vapourtec and third-party suppliers.

Remote desktop access

The touch screen can be viewable from a remote device such as wireless tablet, smart phone, PC or laptop, and if configured, can support adjustment of the remaining reaction schedule based on analytical data from previous reactions.

2 Licensing

A separate software licence is provided for Flow Commander when running on the Vapourtec Touch Screen or a PC running Windows® 10, and is checked whenever the software is launched.

Each standard licence enables the control of a 2-pump module system (primary pump module). For systems with an additional 2-pump module (secondary pump module) i.e. 4 pumps in total, the Flow Commander Professional Licence extension is required for the touch screen or PC.

2.1 Touch screen

The Flow Commander licence is tied to the unique serial number of the touch screen.

2.2 Windows PC

The Flow Commander licence is tied to the unique serial number of the primary R2 or R2+ pump module connected to the PC.

To enable a secondary pump module, the additional license can be added via the **Setup**>**Install License** menu.

Offline use

A separate licence can also be provided for use by any number of PCs in *offline* mode i.e. not connected to the pump module, and is useful for the purposes of setting up experiments or reviewing charts.

2.3 Developer API

The Flow Commander Developer API (Application Programming Interface) is available as a separate package that programmatically control Flow Commander. Contact Vapourtec for information on licensing and pricing.

3 Software Installation

3.1 Windows PC

Installer

The Flow Commander Setup Wizard installer is provided as a single setup.exe file, and when launched on the PC, guides the user through the installation steps.

1. Both Flow Commander and the license file should be installed from an Administrator user account.
2. Vapourtec recommends using the default settings in the Setup Wizard.

Directly connected (online)

A straight through RS232 serial communications cable connects the PC via its COM port to the RS232 port marked **PC** on the primary pump module. See the user manuals provided with the pump modules for further information on the connection.

If the port is unavailable or not fitted to the PC, Vapourtec supply a USB to Serial adapter that has been qualified for use with Flow Commander to plug into an available USB port on the PC.

Offline use

Flow Commander does not require a connection to the pump module.

Launching Flow Commander software

Once installed, Flow Commander can be launched from the **Windows Start** menu under the Vapourtec menu heading, see Figure 3.1.

Figure 3.1: Windows 10 Start Menu Entry

When launching the software for the first time, browse to the location of the licence file provided by Vapourtec and select it.

3.2 Touch screen

A combined power and serial communications cable provided by Vapourtec connects the touch screen to both ports marked **PC** on the primary pump module. The display draws power from the primary pump module via a circular 2 pin connector.

Launching Flow Commander software

Flow Commander can be launched from the main menu, see Figure 3.2.

Figure 3.2: Touch Screen Main Menu

4 Using Flow Commander

4.1 Screen Layout

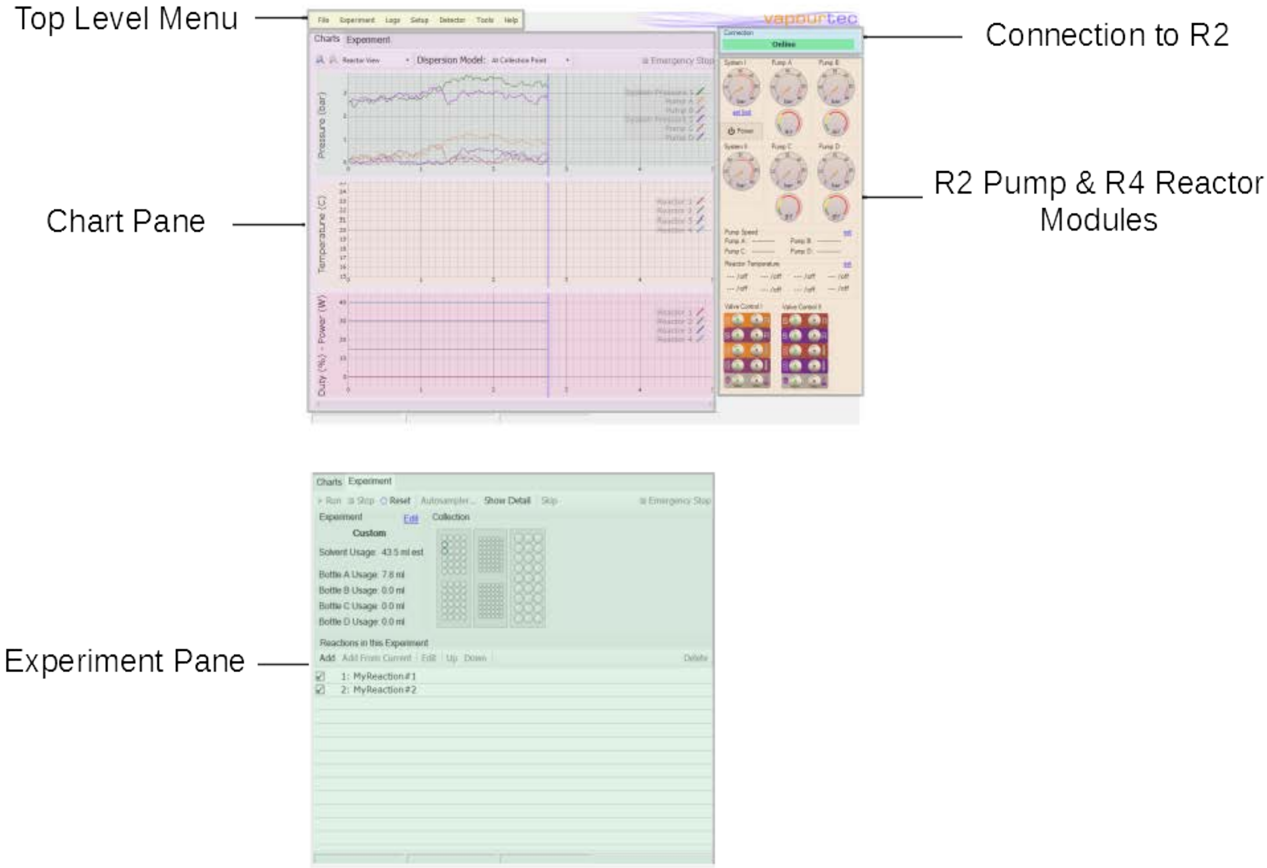


Figure 4.1: Flow Commander Screen Layout

- The top level menu provides access to:
  - Manage Experiments
  - Configure the flow chemistry system
  - Manage logged data Charts and Reports
  - Reset connected detectors
  - Manage licences
  - Access helper tools
  - Upgrade the R-series component firmware
- The connection settings provides a button to connect and disconnect the communication link to the primary R2 pump module.

- The pump module and reactor section allows full manual control of both primary and secondary R2 pump modules, and R4 reactor heater modules.
- The chart pane displays the live and recorded status of components and calculations graphically.
- The experiment pane allows the user to configure experiment set-ups and to view details of automated reactions.

4.2 Automatic control

The user flow to run the system automatically is shown in Figure 4.2:

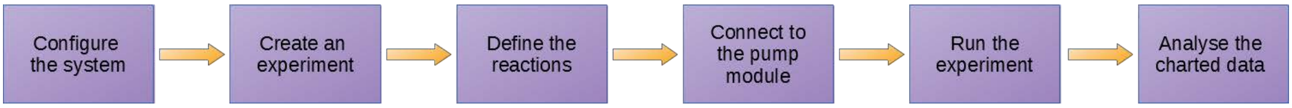


Figure 4.2: Automatic User Flow

To automate a reaction, Flow Commander must be configured accurately with all the parameters of the particular installation.

Parameters are stored in three levels, so that infrequently changing parameters do not clutter the user interface during regular use:

- The ***System Configuration*** stores the least frequently changed information.
- An ***Experiment*** describes a particular selection of pumps, reactors, tubing and other hardware components used for one or more reactions.
- A ***Reaction*** is a set of conditions to be run as part of an experiment. One or more reactions can be carried out in an experiment.

4.3 Manual control

The minimum user flow to run the system manually is shown in Figure 4.3:

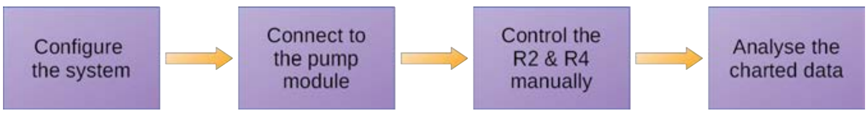


Figure 4.3: Manual User Flow

Manual control is ideal for cleaning the tubing, initially filling empty reactors with solvent etc.

5 Connecting to the primary pump module

5.1 Powering Up

Power up the primary R2 pump module.

If a secondary R2 pump module is fitted, power this up **before** powering up the primary pump module.

5.2 Windows PC

Select the appropriate communications (COM) port from the drop-down list in the connection settings and click the button displaying **Offline**.

The connection status changes from **Offline** to **Connected** if the licensed primary pump module is detected, see section 2.2.

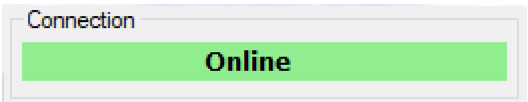


Figure 5.1: Connection Status/Button

Once connected, click the connection button to disconnect Flow Commander from the primary pump module and any communications required for data logging or displaying the hardware status, will cease.

1. If Flow Commander is unable to connect:
2. Check the correct COM port is selected. If USB to serial converter is used, the COM port can be identified by unplugging the USB connector and the associated COM port disappears from the drop-down list.
3. Ensure a straight through RS232 cable is being used between the PC and the primary pump module.
4. Ensure the pump modules are powered on.
5. Restart the USB to Serial converter by unplugging it from the USB port on the PC and then re-inserting it.

5.3 Touchscreen

The connection status displays **Connected** unless the licensed primary pump module is not detected.

6 Configure the system

System configuration is accessed from the top level menu and allows the user to configure parameters such as collection tubing lengths, communications with third-party equipment and fraction collector racks present in the system. System configuration does not affect the chemistry of a reaction and is only stored locally on the PC or display panel, and not saved along with an Experiment. This allows an Experiment created on one system to be run on another system with unique system configuration parameters, without modification. Parameters for optional equipment can still be set if Flow Commander is in *offline* mode or if the equipment is not present at the time of configuration.

Configuration settings are organised into logical groups which can be selected in the left hand pane.

6.1 General

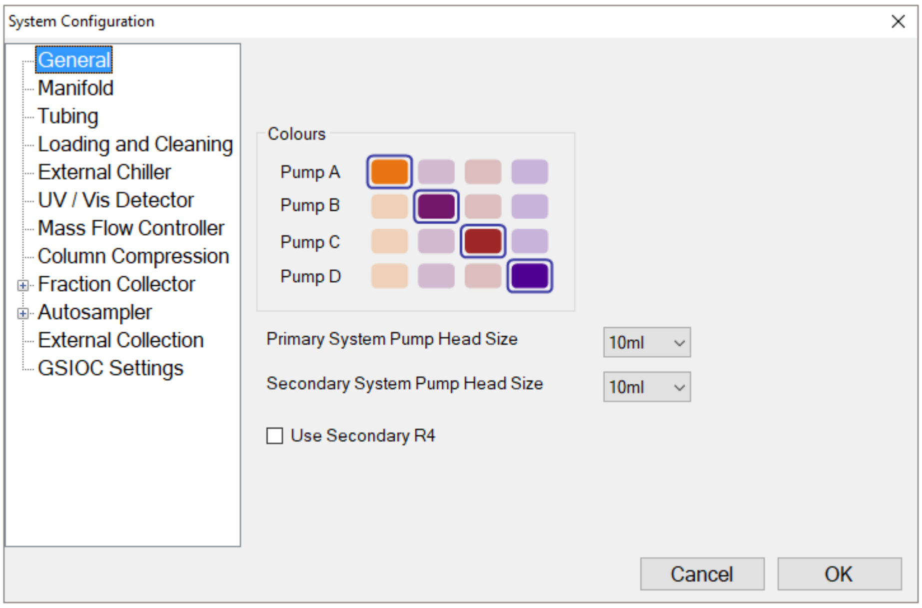


Figure 6.1: System Configuration - General

Pump module colours

Select one colour for each pump (up to 4 can be present in the system).

- Pumps A & B are located on the primary pump module.
- Pumps C & D are located on the secondary pump module.

The selected colours should match the colouring on the pump module hardware.

Pump head size

Select the head size for primary and secondary pump modules from the drop-down list.

Secondary R4 reactor heater module

Tick the check box to enable a secondary R4 reactor heater module to support up to 8 reactor channels.

6.2 Manifold

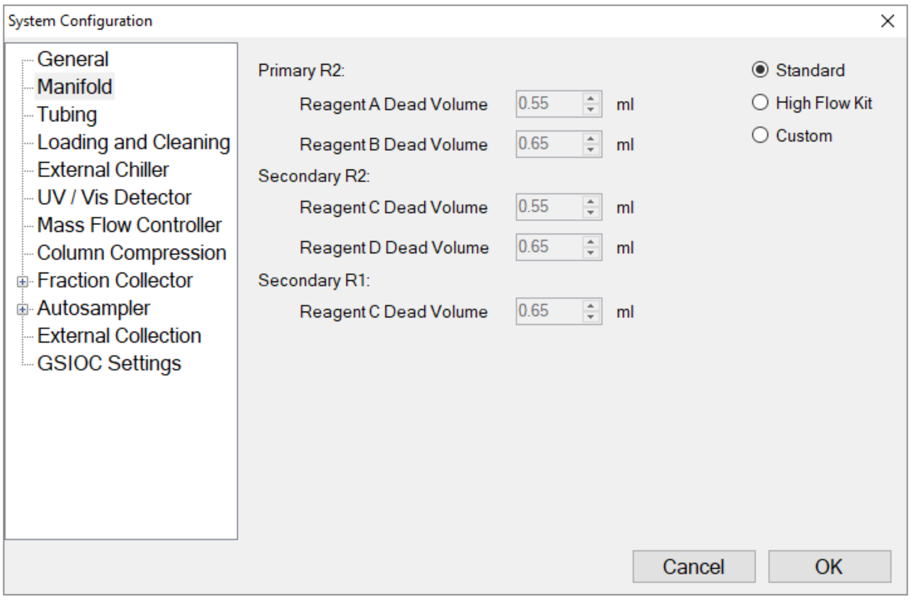


Figure 6.2: System Configuration - Manifold

The **Manifold** settings screen allows fine tuning of the dead volumes in the manifold if bottled reagents are used. The dead volume is defined as the volume from the reagent valve, through the pump, tubing and manifold, through to the mixer tee-piece. Select the option corresponding to the tubing and manifold used. Pre-set dead volumes are displayed for the standard and high flow kit supplied by Vapourtec. The **Custom** option allows the user to specify individual volumes.

If a secondary pump module is present, Flow Commander will use the same dead volume for the pump C tubing path as pump A, and for D will use the B dead volume.

6.3 Tubing

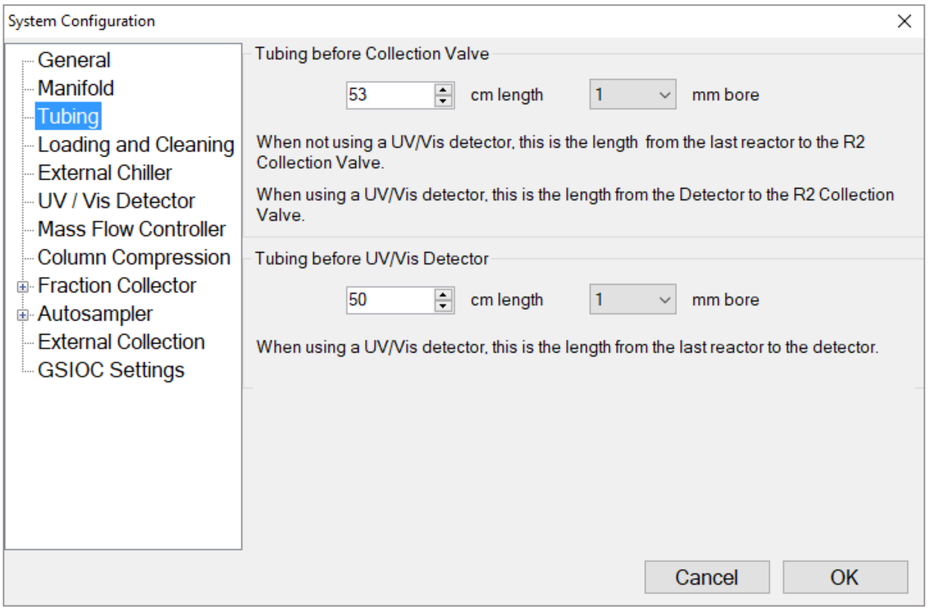


Figure 6.3: System Configuration - Tubing

The tubing installed between the last reactor in the flow path, and the pump module collection valve is configured in the **Tubing** settings screen.

If an optical detector (UV/Vis detector) is installed in the flow path, sizes for the tubing between the last reactor and detector, and between the detector and collection valve must be specified.

If a fraction collector is used, refer to the device’s settings to specify any additional collection tubing.

6.4 Loading and cleaning

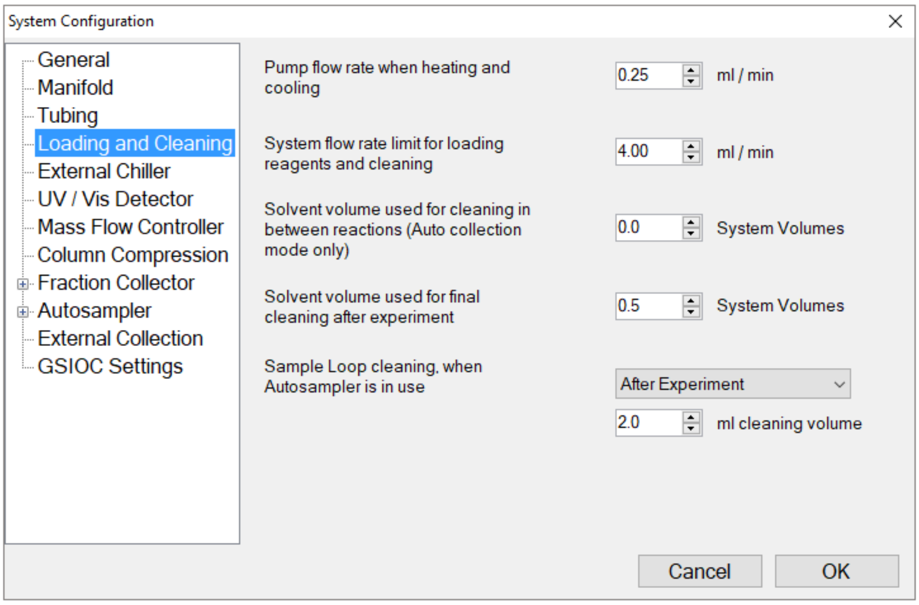


Figure 6.4: System Configuration - Loading and Cleaning

The **Loading** and **Cleaning** settings screen sets the flow rates and cleaning volumes of the various components in the flow path.

Heating and Cooling Flow Rate

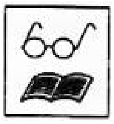
When the reactors are heating or cooling, all the pumps in the system will continuously pump a small amount of solvent to guarantee pressure is maintained in the system.

- Higher flow rates may waste solvent, or cause the pressure limit to be exceeded if there are restrictive components in the system such as packed column reactors.
- Lower flow rates may not maintain pressure if the back pressure regulator is contaminated or the system is cooling.
- Stainless steel tube reactors cause a pressure drop when cooling because there is no elasticity in the reactor tubing and may require a higher flow when cooling to avoid the false triggering of detected leaks.

Flow Rate Limit

This limit applies when loading reagents from reagent bottles and when cleaning the system using solvent.

- Bottled reagents are loaded into the manifold dead volume before the reaction starts, and governed by this flow rate.
- System solvent is pumped at this rate during system cleaning.



Higher flow rates will load and clean faster but may cause the system to halt due to overpressure if there are restrictive components in the system.

Cleaning Volumes

The system can be cleaned between individual reactions and also after the Experiment has been completed.

As reagents are driven through the system with solvent, successive reactions are separated with solvent and normally additional cleaning between reactions is unnecessary. A specified solvent volume to achieve an additional clean between reactions is only used in automatic Collection mode, see section 8.7, where Flow Commander automatically determines the reaction collection window.

The cleaning volumes are specified in System Volume units, where a unit is defined as the volume of all the components (reactors and tubing) present in the system. The actual amount of solvent used is easily scaled with reactor choice and tubing, without having to make any additional adjustments.

The solvent volume used to clean sample loops when an autosampler is used is also specified for cleaning after each reaction, or after the experiment has completed.

6.5 External chiller

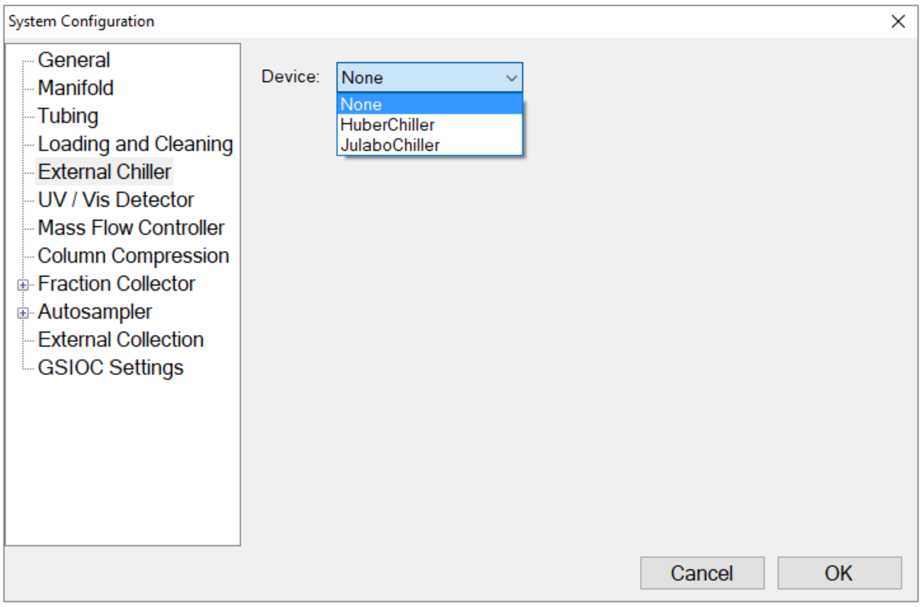


Figure 6.5: System Configuration - External Chiller

The **External Chiller** settings screen enables third-party refrigeration external chillers and recirculators from Julabo™ and Huber™ to be used with reactors. Flow Commander sends temperature set points using the RS232 serial communications link and the settings must match those configured on the chiller equipment.

A 3-wire crossover RS232 serial cable must be used with the male connector plugged into the PC/touch panel via a USB to Serial adapter, and female connector plugged directly into the chiller.

Tick **Leave Running** to maintain the chiller temperature after the Experiment has completed.

Select from the **Sensor** options to instruct the chiller to use either its internal built-in, or externally attached temperature probe.

6.6 UV/Vis Detector

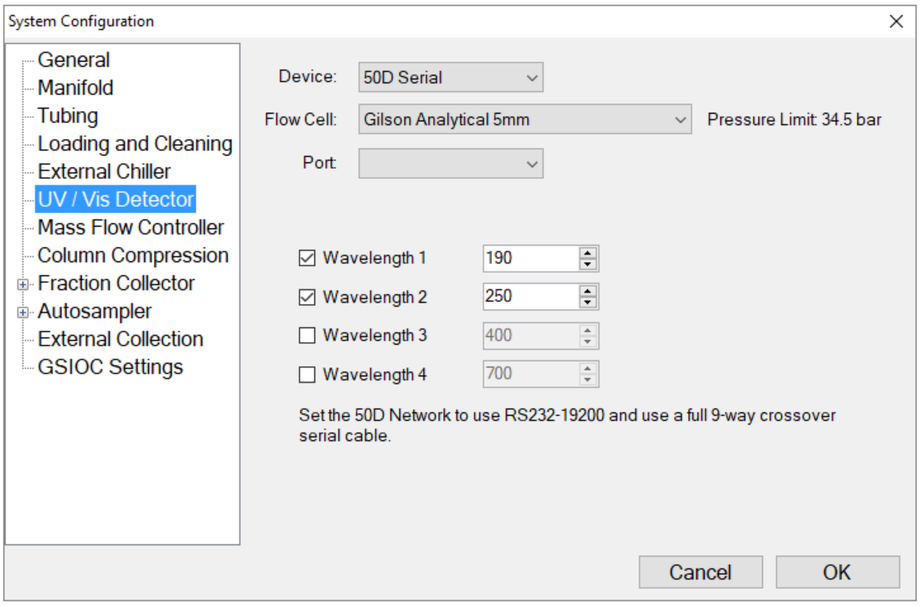


Figure 6.6: System Configuration - UV / Vis Detector

The **UV / Vis Detector** settings screen allows the third-party optical detectors to be selected and configured.

Select the appropriate detector device before selecting from the list of flow cells attached to the detector. The pressure limit for the chosen flow cell is displayed.

Gilson™ Detectors

The Gilson 151, 152 and 155 detector devices are controlled via the port marked **GSIOC** on the primary pump module. The GSIOC ID must be set on the GSIOC settings screen, see section 6.12.

Only the GSIOC cable supplied with the Gilson detector can be used to connect the detector to the primary pump module. Serial cables marked PC or RS232 **do not** support Gilson devices.

Knauer™ Detectors

- Select **SL200** if the detector device is connected via the RS232 port marked **Spare** on the primary pump module.
- Select **SL200 serial** or **50D serial** if the detector device is connected via a serial communications (COM) port on the display panel or PC.

Use the Knauer supplied RS232 cable to connect to the detector device.

A USB to serial converter can be used to provide a COM port.

Network Detectors

Select **Network** from the list of devices to enable detectors that are controlled using the Network tools supplied and installed by Vapourtec.

6.7 Mass Flow Controller

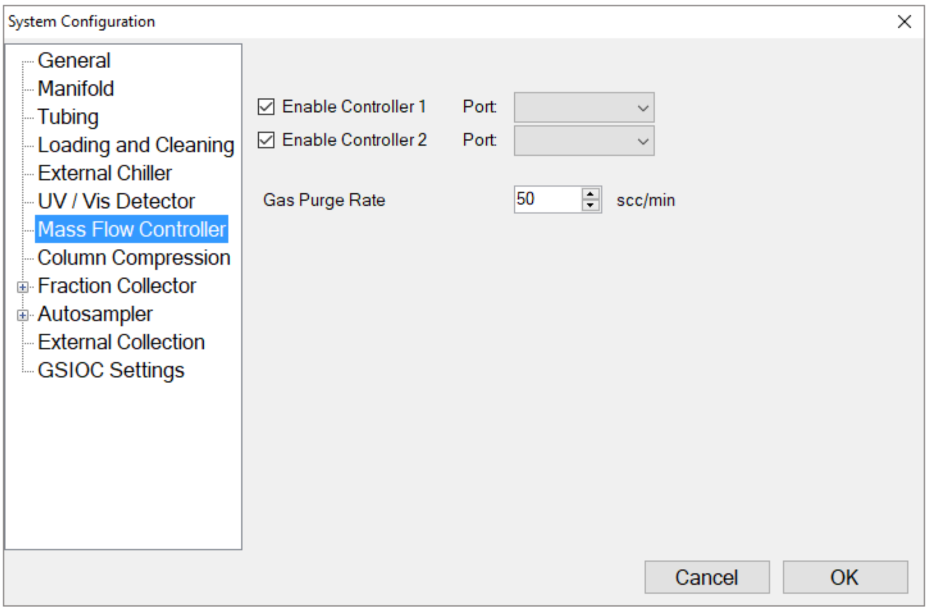


Figure 6.7: System Configuration - Mass Flow Controller

The **Mass Flow Controller** settings screen enables up to two Sierra™ mass flow controllers to supply a flow of pressurised gas for reactions.

Select the serial communications (COM) port used by the PC or display panel to connect to each controller.

Specify the **Gas Purge Rate** in standard cubic centimetres/minute.

Connect using the cable supplied by the MFC manufacturer. A USB to Serial adapter may be required.

6.8 Column Compression Controller

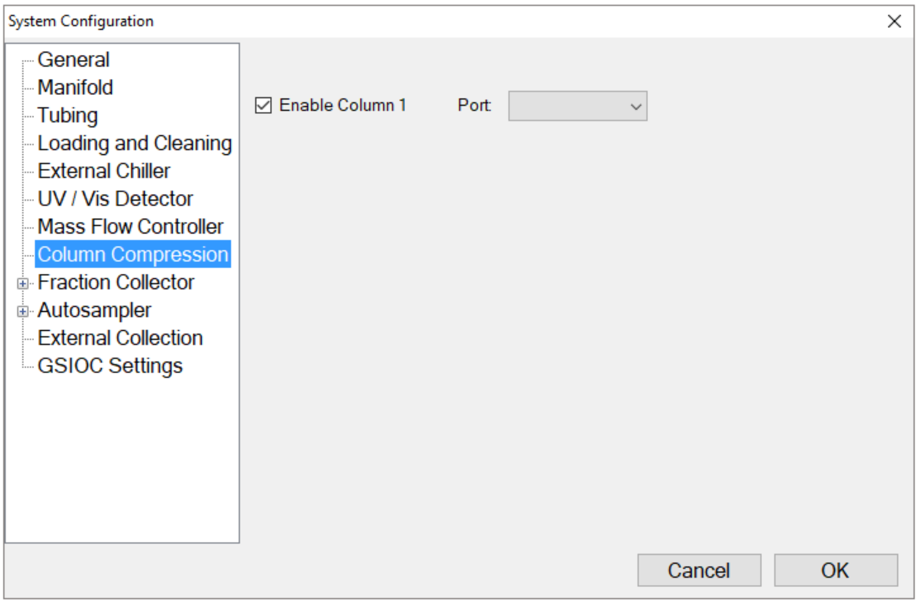


Figure 6.8: System Configuration - Column Compression

The **Column Compression** settings screen enables the resin within a column reactor to be compressed in order to ensure optimal and predictive reactions.

Select the serial communications (COM) port used by the PC or display panel to connect to each controller.

A USB to serial converter can be used to provide a COM port.

6.9 Fraction Collector

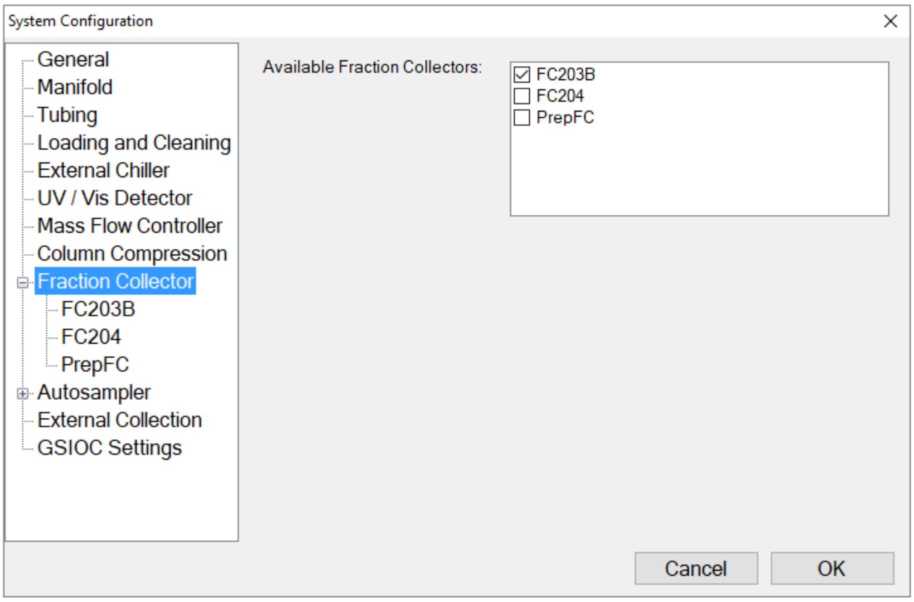


Figure 6.9: System Configuration - Fraction Collector

The **Fraction Collector** settings screen allows the filtering of available Gilson fraction collectors, tray inserts and racks. Tick the check box for each item to appear in the **Collector Setup** screen when creating a new experiment.

Specify the collection tubing size for each type of fraction collector, see Figure 6.10.

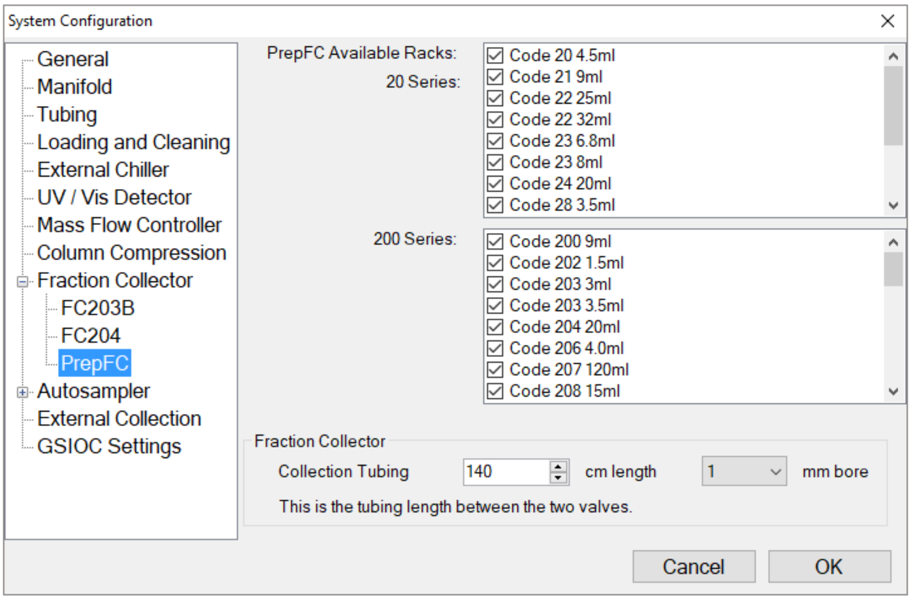


Figure 6.10: System Configuration - Fraction Collector - PrepFC

Only the GSIOC cable supplied with the Gilson fraction collectors can be used to connect the device to the primary pump module. Serial cables marked PC or RS232 **do not** support Gilson devices.

The Gilson GX271 and 215 liquid handlers can operate as an autosampler and/or fraction collector and are configured from the **Autosampler** settings, see section 6.10.

6.10 Autosampler

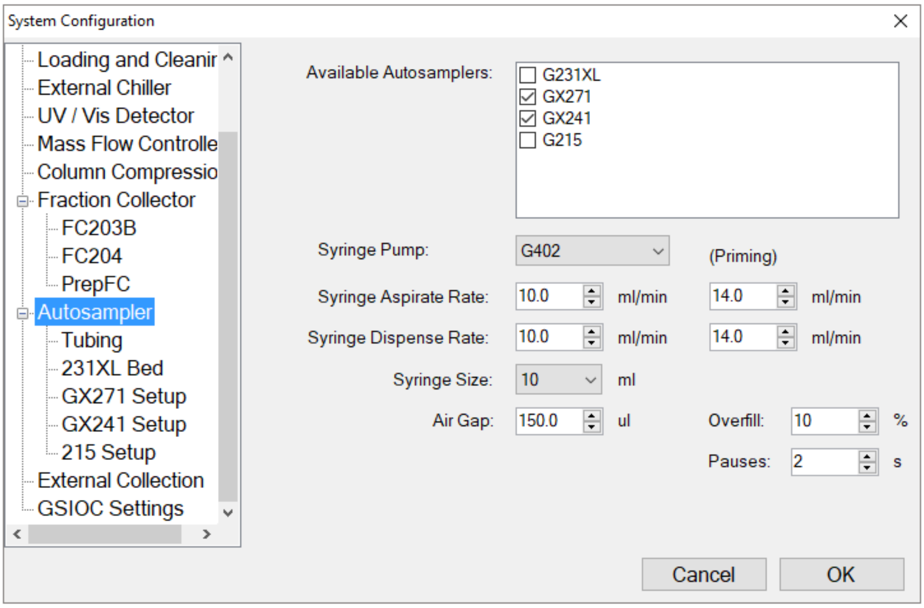


Figure 6.11: System Configuration - Autosampler

The **Autosampler** settings screen allows the filtering of supported Gilson autosamplers.

Only the GSIOC cable supplied with the Gilson autosamplers and syringe pump can be used to connect to the primary pump module. Serial cables marked PC or RS232 **do not** support Gilson devices.

Tick the check box for each item to appear in the **Collector Setup** screen when creating a new experiment.

Select the **Syringe Pump** type from the drop-down list.

The aspirate and dispense rates of the syringe pump can be adjusted as required.

Faster rates will speed up loading, but the pressure generated by the syringe pump is limited. Restrictive components such as long sample loops or partially blocked sample loop valves, for example due to crystals of reagent, may cause the syringe pump to bypass (leak) or trip.

The rates used to prime the autosampler loop may be higher than the rates used when filling a sample loop, as the sample loop will not be connected at this time and there is less flow restriction.

The **Syringe Size** ensures correct volumes are dispensed.

The **Air Gap** controls the air volume used to separate the sample from system solvent when loading, to prevent sample dilution. A larger air gap permits slightly higher loading pressures without failure of the gap.

The **Overfill** percentage causes a larger volume of reagent to be loaded than needed, to guarantee the sample loop is completely filled. Flow Commander positions the sample in the centre of the loop, leaving an equal amount of wastage at either side of the loop. For example, if a 2 ml sample loop is being filled, and the overfill is set to 10%, Flow Commander aspirates 2.2 ml and attempts to leave 0.1 ml at each end of the loop. When the lengths of the load tubing are set accurately, the sample loop valves are clear and not generating excessive back pressure. When the sample loops are accurately sized, and the pause time is sufficiently long, it is possible to use smaller overfill percentages to save reagent, especially if larger sample loops are being used.

The **Pauses** setting is used whenever liquid is in motion in the tubing and an air gap is introduced. Flow Commander will pause after each movement to allow the pressure to equilibrate. As there is some flow restriction, some back pressure is always generated which causes the air gap to compress. This would cause fluid positioning errors if the air gap is not given sufficient time to return to its original volume. This value should be increased if the sample is still in motion when the needle is withdrawn from the needle port after loading.

Tubing

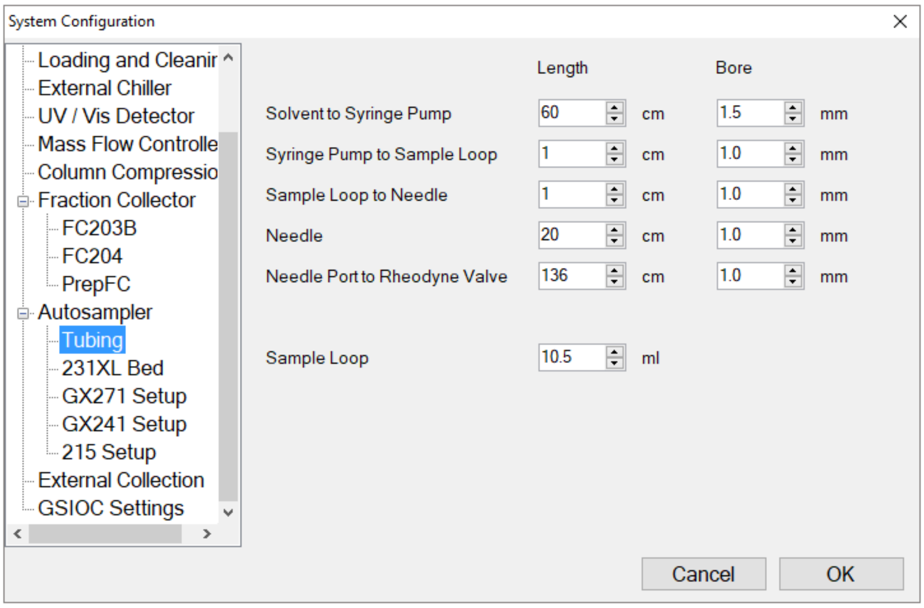


Figure 6.12: System Configuration - Autosampler - Tubing

The tubing sizes associated with the autosampler and syringe pump are specified in the **Tubing** settings screen.

Solvent to Syringe Pump is the length of tubing between the system solvent bottle and the syringe pump inlet. This volume is needed when priming the tubing.

Syringe Pump to Sample Loop is the length of tubing between the syringe pump outlet and the autosampler loop.

Sample Loop to Needle is the length of tubing between the autosampler loop and the top of the autosampler needle.

Needle is the bore and length of the autosampler needle.

Needle Port to Rheodyne Valve is the length of tubing between the needle ports and the sample loop valve load port. This value should be slightly higher than the actual tubing length to take into account the length and dead volume of the needle port.

Sample Loop refers to the autosampler loop volume and must exceed the volume of the largest experiment sample loop to be used.

The sample loop volumes are configured as part of the Experiment setup as they may change more frequently.

The tubing from the syringe pump outlet, the autosampler loop, and autosampler loop to needle tubing may be one continuous length of tubing, in which case the loop volume should just be the part of the tubing which is coiled.

The **Needle Port to Rheodyne Valve** tubing length can be adjusted to fine tune the positioning of the loaded sample relative to the sample loop. After loading, there should be an equal amount of wastage either side of the sample loop.

- Increase the value if the sample is not being loaded far enough.
- Decrease the value if the sample is being pushed too far.

If the sample is still in motion when the autosampler needle is withdrawn from the needle port, the **Pauses** value on the autosampler settings screen should be increased.

GX271 Setup

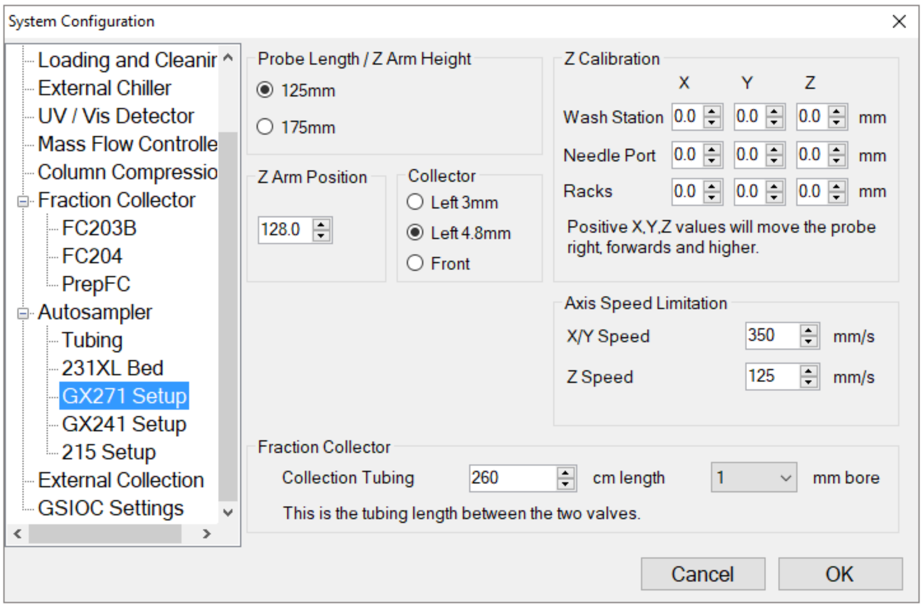


Figure 6.13: System Configuration - Autosampler – GX271 Setup

This settings screen allows the Gilson GX271 liquid handler (autosampler and fraction collector support) to be configured.

Select the Z arm height corresponding to the fitted sample probe, and the Z arm position.

Changing the probe length may require the Gilson calibration software to be run on the GX271 to program it for the new length.

Select the fraction collector foot hole used for collection corresponding to the tubing size used. Normally the smaller 3mm bore tubing is in use which uses the left hole. Flow Commander uses this information to align the appropriate hole with the collection vial centre.

Specify the X, Y, Z position offsets for the wash station, needle port and racks.

The Z calibration settings trim the needle height for the racks and the needle ports.

Reduce the axis speeds if required. This is useful for demonstration or safety purposes if the GX271 is in use outside a fume cabinet or in a public area.

Set the **Fraction Collector** tubing size (from the R2 collection valve to the GX271 collection valve).

GX241 Setup

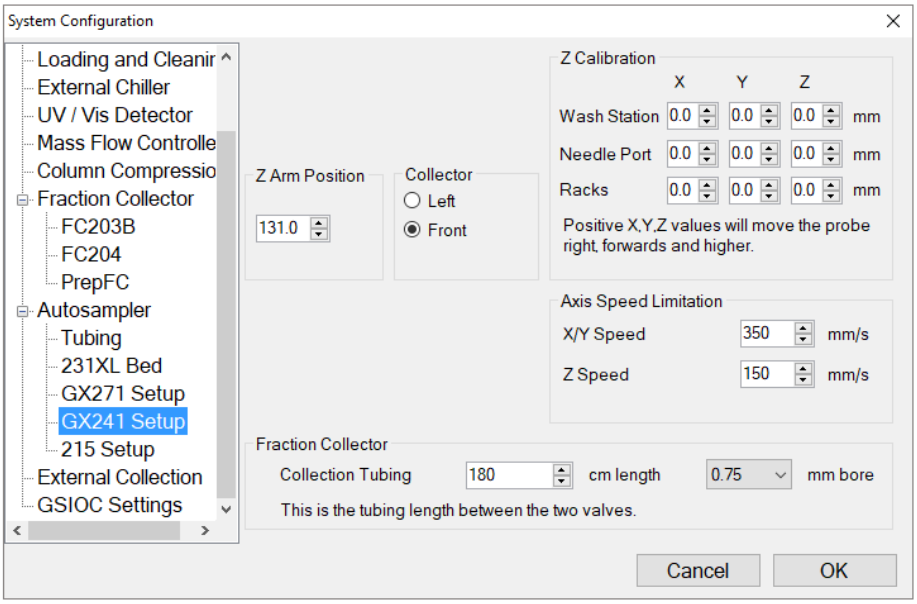


Figure 6.14: System Configuration - Autosampler - GX241 Setup

This settings screen allows the Gilson GX241 liquid handler (with autosampler and fraction collector support) to be configured.

Select the Z arm position.

Select the fraction collector foot hole used for collection corresponding to the tubing size used. Flow Commander uses this information to align the appropriate hole with the collection vial centre.

Specify the X, Y, Z position offsets for the wash station, needle port and racks.

The Z calibration settings trim the needle height for the racks and the needle ports.

Reduce the axis speeds if required. This is useful for demonstration or safety purposes if the GX241 is in use outside a fume cabinet or in a public area.

Set the **Fraction Collector** tubing size (from the R2 collection valve to the GX241 collection valve).

215 Setup

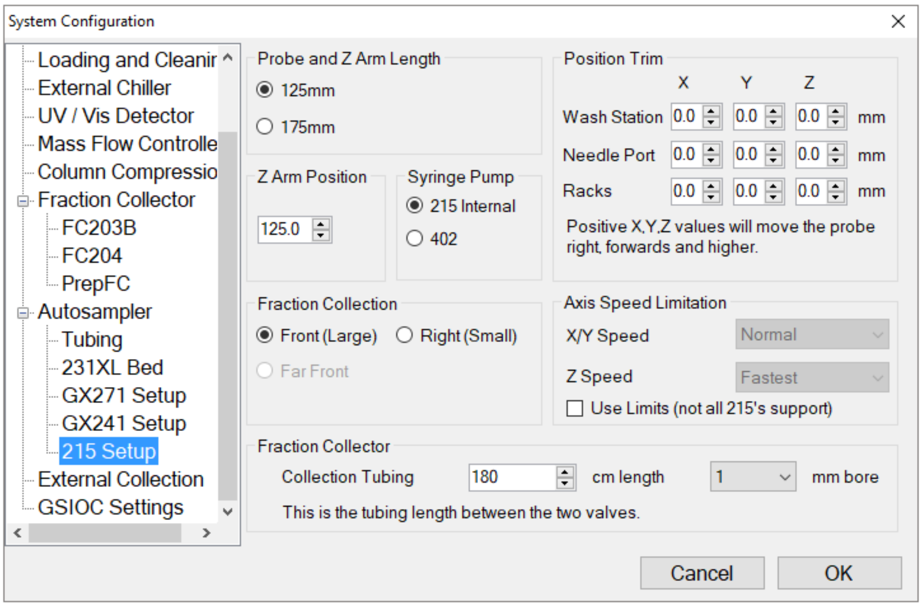


Figure 6.15: System Configuration - Autosampler - 215 Setup

This settings screen allows the Gilson 215 liquid handler (with autosampler and fraction collector support) to be configured.

Select the Z arm height corresponding to the fitted sample probe, and the Z arm position.

Changing the probe length may require the Gilson calibration software to be run on the 215 to program it for the new length.

Select the Syringe Pump to be used. Flow Commander can use the 215's internal syringe pump or a separate Gilson 402 syringe pump.

Select the fraction collector foot hole used for collection corresponding to the tubing size used. Flow Commander uses this information to align the appropriate hole with the collection vial centre.

Specify the X, Y, Z position offsets for the wash station, needle port and racks.

The Z calibration settings trim the needle height for the racks and the needle ports.

Enable the use of axis speed limits if required. This is useful for demonstration or safety purposes if the 215 is in use outside a fume cabinet or in a public area. Some older 215 devices do not recognise the speed commands, resulting in the 215 failing to move. Clear the **Use Limits** option to ensure compatibility with older 215 liquid handlers.

Set the **Fraction Collector** tubing size (from the R2 collection valve to the 215 collection valve).

6.11 External Collection

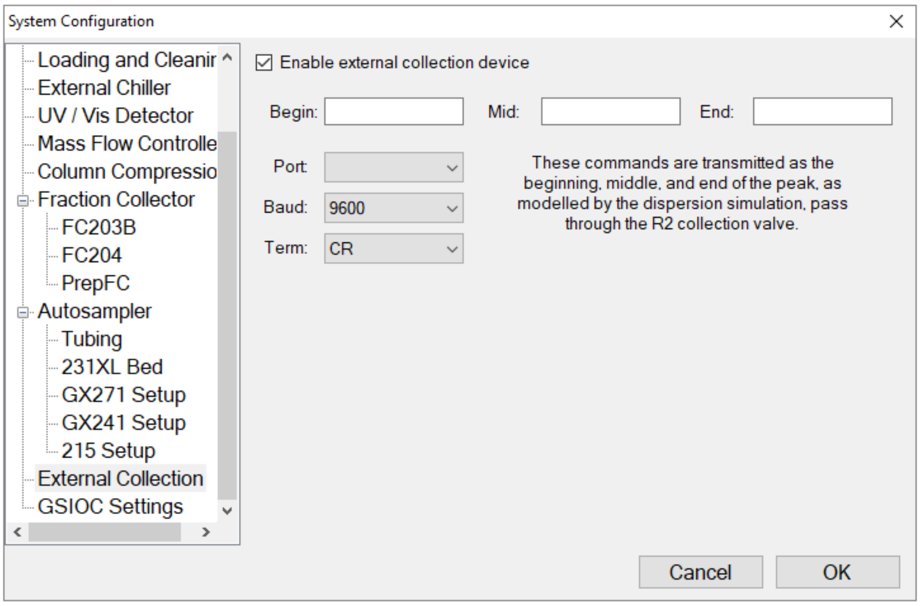


Figure 6.16: System Configuration - External Collection

The **External Collection** settings screen enables a third-party collection device to be controlled by Flow Commander.

Select the serial communications (COM) port used by the PC or display panel to connect to the collector.

A USB to serial converter can be used to provide a COM port.

Enter the commands to be sent to the device corresponding to the beginning, middle and end of the peak collection window, defined by the dispersion model.

Enter the baud rate settings for the communications link and select the termination characters used by the communication protocol.

6.12 GSIOC Settings

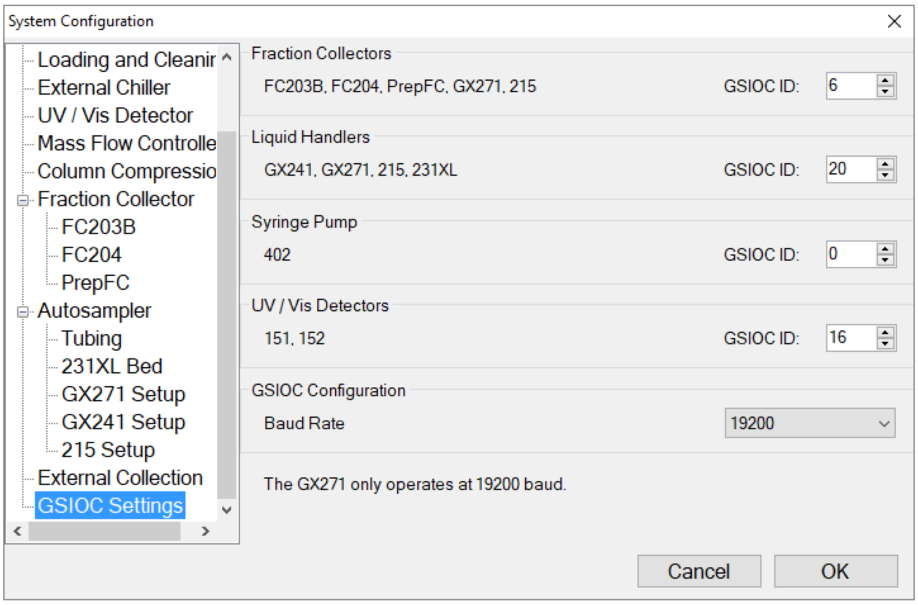


Figure 6.17: System Configuration - GSIOC Settings

The **GSIOC Settings** screen provides the communication parameters to be specified for Gilson devices. Each Gilson device in the system must have a unique GSIOC identifier (ID) which is used to direct control commands to the specific device.

Set the **GSIOC ID** corresponding to the equipment that is providing the functionality against each functional category; fraction collector, liquid handler, syringe pump or optical sensor. If a GX271 or 215 autosampler is to be used for both fraction collecting and autosampling, set its ID in both categories.

If two GX271/215 devices are being used; one as an autosampler and one as a fraction collector, set the corresponding ID against each category.

Select the communication **Baud Rate** for all Gilson devices.

The GX271 only operates at 19200 baud.

7 Create an Experiment

An Experiment defines the specific configuration of installed equipment with which to run one or more Reactions. Flow Commander uses this information in conjunction with the system configuration, see section 9, to automate Reactions.

Unlike the system configuration, the Experiment can be re-used on a different system as long as the components required by the Experiment are available.

7.1 Managing Experiments

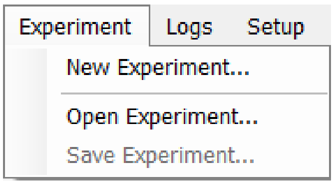


Figure 7.1: Experiment Menu

The top level menu, see Figure 7.1, provides options to:

- Create a new experiment
- Open an existing experiment
- Save an experiment

The Experiment configuration is always preserved when Flow Commander is exited and loaded when Flow Commander is launched.

A saved experiment contains the experiment configuration and reaction information.

Create a new experiment

Click **Experiment**>**New Experiment...** to create a new experiment. The previous Experiment configuration excluding any Reactions is loaded.

If an Experiment is not opened, clicking **Edit** on the Experiment pane will also create a new experiment.

A configuration wizard captures the Experiment configuration using a sequence of screens.

Open an existing experiment

Click **Experiment**>**Open Experiment...** to browse to the location before selecting the Experiment definition file (with .fcexp extension).

Save an experiment

Click **Experiment**>**Save Experiment…** to browse to the location before specifying the filename to save the Experiment definition to. A file with .fcexp extension is created.

7.2 Configuration wizard

Configuration screens

Simply navigate between the configuration screens, see Figure 7.2, using the **Next >>** and **<< Back** buttons.

Additional screens (*) are displayed to capture configuration details if sample loops or more complex collectors i.e. fraction collectors or fraction collector/autosamplers, are included in the flow path.

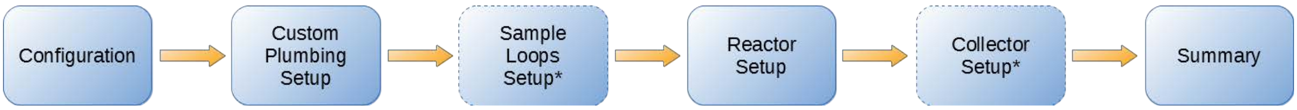


Figure 7.2: Configuration Wizard Screens

When editing an existing Experiment with Reactions already defined, some options which the Reactions depend upon cannot be modified and will appear greyed out. Create a new Experiment to change the option.

The **Finish** button on the Summary screen completes the wizard and displays the **Add Protocol** screen, see section 8.

7.3 Configuration

Specify the **Experiment Title**, **Chemist** details and **Notes**.

The information recorded will be visible in the generated PDF report.

7.4 Custom Plumbing Setup

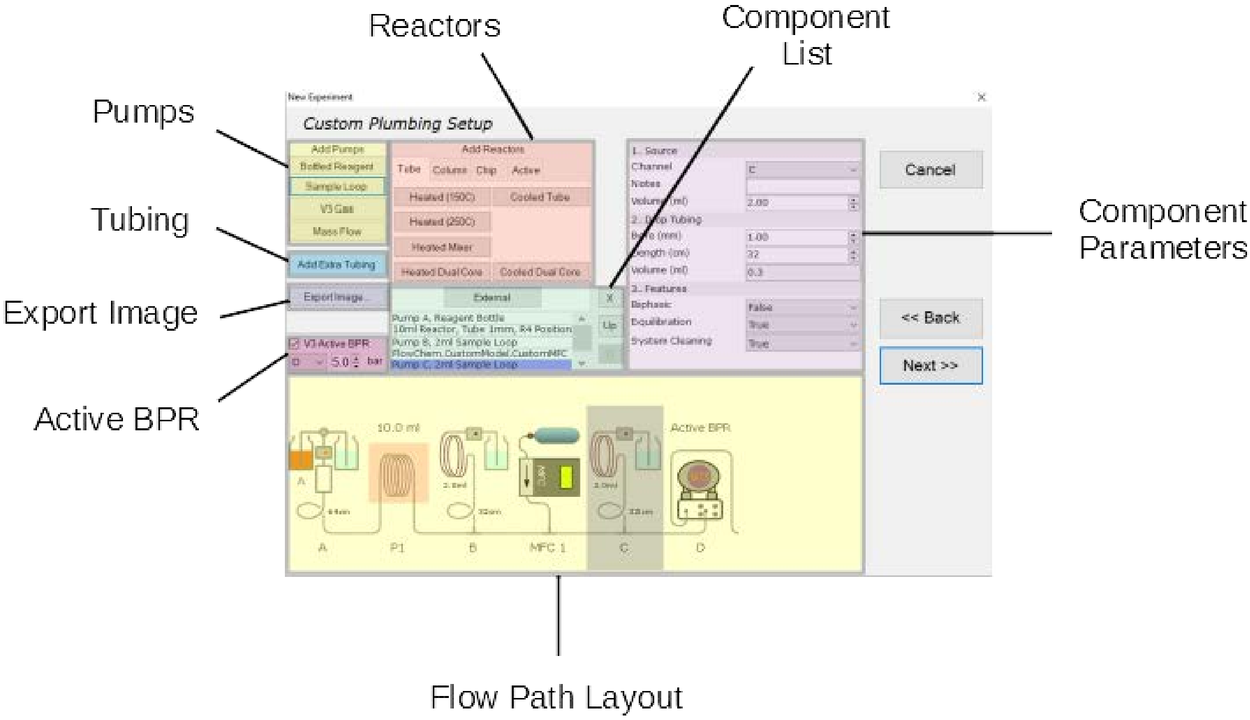


Figure 7.3: Custom Plumbing Setup

This screen captures detailed information about the flow path through to the back pressure regulator (BPR) and includes pumps, reactors, detectors, controllers, external devices and the tubing layout. The collection tubing is not shown but is added as defined by the system configuration.

Each component type can be added to the flow path by clicking on the appropriate button. Added components appear in the graphical flow path layout and also in the ordered component list. The position of the selected component within the flow path is changed by clicking **Up** or **D** (down). Click **X** to remove the component from the flow path.

It is possible to create complex systems with mixed pump types and to position pumps downstream of reactors, see Figure 7.4.

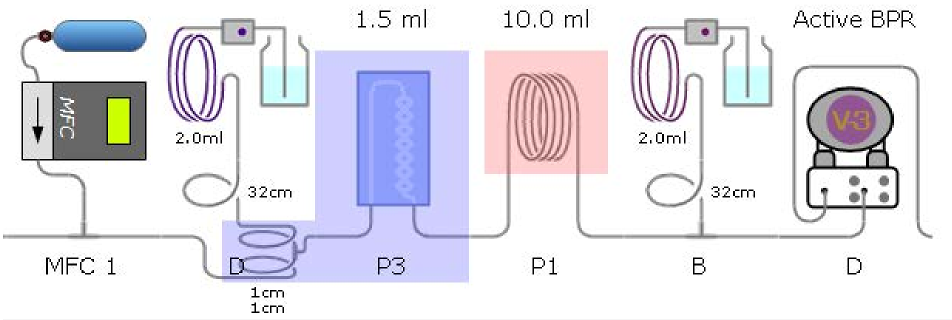


Figure 7.4: More Complex Plumbing Layout

Flow Commander determines when each pump is started in order to synchronise the reagent peaks with each other as they pass through the reactors.

The component buttons are disabled when the maximum number of that component in use has been reached.

The graphical layout can be scrolled horizontally to bring the component of interest into view. Components are highlighted and surrounded with a blue rectangle when selected from the component list or by clicking the component in the layout, resulting in the component's characteristics being displayed.

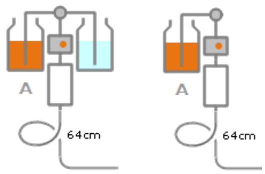
Once the user has accurately captured the flow path in the layout, Flow Commander highlights any invalid configurations to resolve e.g. pump A is being used both as a pump and an active BPR, before displaying the next wizard screen.

Add Pumps

The primary and secondary pump modules supports channels A & B, and C & D respectively.

To re-assign a pump from reagent/solvent bottle to sample loop or vice versa, remove the component first before adding it back to the flow path.

Bottled Reagent/Solvent



Click **Bottled Reagent** to add a pump and assign it to the reagent/solvent bottles.

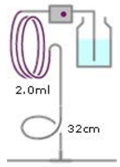
1. Configure the **Source**:
   - Select whether bottles of reagent and solvent are used or restricted to just a single bottle of reagent (e.g. when pumping a liquified gas).

   Selecting a single bottle of reagent means that the pump and associated tubing cannot be flushed with solvent during the cleaning regime.

   - Assign the pump channel.
   - Specify the molecular concentration.
   - Add notes if required.
2. Specify the drop tubing size; the volume is displayed.
3. Enable the features:
   - Enable **Biphasic** to model the mixing of reagent at the tee-piece downstream as non-dispersing.
   - Disable **Equilibration** to prevent solvent being pumped during the equilibration phase of a reaction, and reducing the duration of the experiment.

- Disable **System Cleaning** to prevent the pump being used during the system cleaning routine, and reducing the duration of the experiment.

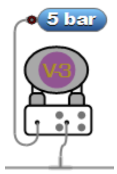
Sample Loop



Click **Sample Loop** to add a pump and assign it to the sample loop.

1. Configure the **Source**:
   - Assign the pump channel.
   - Add notes if required.
   - Specify the volume.
2. Specify the drop tubing size; the volume is displayed.
3. Enable the features:
   - Enable **Biphasic** to model the mixing of reagent at the tee-piece downstream as non-dispersing.
   - Disable **Equilibration** to prevent the solvent being pumped during the equilibration phase of a reaction, reducing the duration of the experiment.
   - Disable **System Cleaning** to prevent the pump being used during the system cleaning routine, reducing the duration of the experiment.

V3 Gas

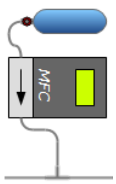


Click **V3 Gas** to add a V3 pump used to pump a gas.

The gas source can must be able to maintain 5 bar to ensure accurate flow rates.

1. Configure the Source:
   - Assign the pump channel.

Mass Flow Controller



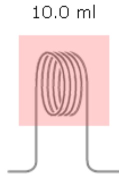
Click **Mass Flow** to add a mass flow controller (MFC).

1. Configure the Source:
   - Select from the channels available on the device.
   - Select the type of gas being measured.

Add Reactors

The R4 heater reactor module supports four reactor positions; P1, P2, P3 & P4. Enabling a secondary reactor module additionally supports the positions P5, P6, P7 & P8, see section 6.1.

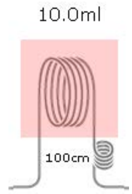
Heated Reactor (150°C)



Click **Heated (150C)** to add a heated reactor with maximum temperature of 150°C.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
   - Select the **Reactor Type** from the list.
   - Specify the **Volume**.
2. Configure the **Tube Reactor**:
   - Specify the **Bore**.

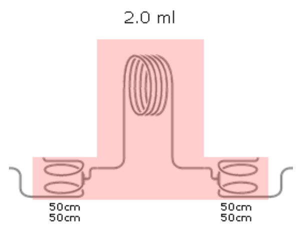
Heated Reactor (250°C)



Click **Heated (250C)** to add a heated reactor with maximum temperature of 250°C and with stainless steel or hastelloy tubing.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
   - Specify the **Volume**.
2. Configure the **Tube Reactor**:
   - Specify the **Bore**.
3. Configure the **Post-Reactor Cooler**:
   - Specify the **Bore** and **Length**.

Heated Mixer



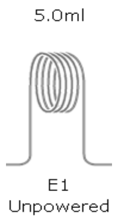
Click **Heated Mixer** to add a heated mixer reactor with PFA tubing. The **Mixer Bore** and **Length** is calculated from the **Heated Tubing** size.

1. Configure the **Reactor**:

- Add Notes if required.
- Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
- Select the reactor **Position**.
- Specify the **Volume**.

2. Configure the **Tube Reactor**:
   - Specify the **Bore**.
3. Configure the **Heating Tubing**:
   - Specify the **Bore** and **Length**.

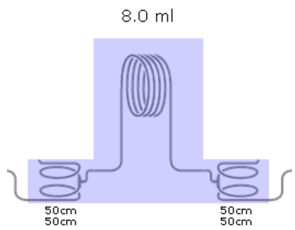
External Reactor



Click **External** to add an external reactor. As the reactor does not take up a slot marked P1, P2 etc in the reactor module, the position will be marked as E1, E2 etc.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the **Reactor Type** from the list.
   - Specify the **Volume**.
2. Configure the **Tube Reactor**:
   - Specify the **Bore**.
3. Configure the **External Control**:
   - Select the **Control Type**; unpowered or an external chiller, see section 6.5.

Cooled Tube

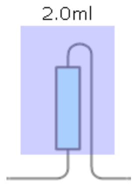


Click **Cooled Tube** to add a cooled tube reactor.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
   - Select the **Reactor Type** from the list.
   - Specify the **Volume**.

2. Configure the **Tube Reactor:**
    - Specify the **Bore**.
3. Configure the **Cooling Tubing**:
    - Specify the **Bore** and **Length**.

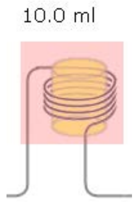
Cooled Column



Click **Cooled Column** to add a cooled column reactor.

1. Configure the **Reactor**:
    - Add **Notes** if required.
    - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
    - Select the reactor **Position**.
    - Select the **Reactor Type** from the list.
    - Specify the **Volume**.

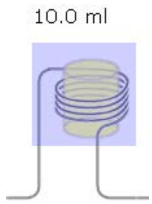
Heated UV-150 Photochemical Reactor



Click **Heated UV-150** to add a heated UV-150 photochemical reactor.

1. Configure the **Reactor**:
    - Add **Notes** if required.
    - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
    - Select the reactor **Position**.
    - Select the **Reactor Type** from the list.
    - Specify the **Volume**.
2. Configure the **Tube Reactor**:
    - Specify the **Bore**.

Cooled UV-150 Photochemical Reactor



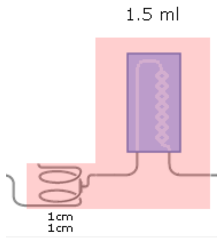
Click **Cooled UV-150** to add a cooled UV-150 photochemical reactor.

1. Configure the **Reactor**:
    - Add **Notes** if required.
    - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
    - Select the reactor **Position**.

- Select the **Reactor Type** from the list.
- Specify the **Volume**.

2. Configure the **Tube Reactor**:
   - Specify the **Bore**.

Heated Chip Micromixer Reactor



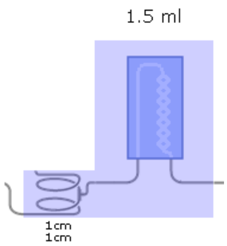
Click **Heated Chip** to add a heated glass chip micromixer reactor.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
2. Configure the **Glass Chip**:
   - Select the **Part No.** from the drop-down list, see Table 1. The **Preconditioning** and **Volume** characteristics of the micromixer are displayed.

Part Number	Description
50-1273	0.2ml Lab-MS Glass micromixer chip
50-1268	0.2ml Lab-MX Glass micromixer chip
50-1272	1.7ml Lab-V Glass residence time chip
50-1269	1.1ml Lab-VS Glass residence time chip
40-1773	1.5ml glass micromixer chip 2 input
40-1774	1.0ml glass micromixer chip 2 input preheating
40-1775	0.6ml glass micromixer chip 3 input preheating

Table 1: Glass Chip Reactors and Mixers

Cooled Chip Micromixer Reactor

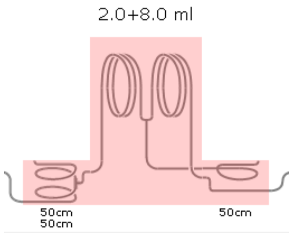


Click **Cooled Chip** to add a cooled glass chip micromixer reactor.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
2. Configure the Glass Chip:

- Select the **Part No.** from the drop-down list, see Table 1. The **Preconditioning** and **Volume** characteristics of the micromixer are displayed.

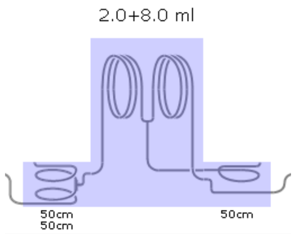
Heated Dual Core



Click **Heated Dual Core** to add a heated dual core reactor.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
   - Specify **Volume A** and **Volume B**.
2. Configure the **Tube Reactor**:
   - Specify the **Bore**.
3. Configure the **Preheat Tubing**:
   - Specify the **Bore** and **Length**.

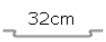
Cooled Dual Core



Click **Cooled Dual Core** to add a cooled dual core reactor.

1. Configure the **Reactor**:
   - Add **Notes** if required.
   - Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.
   - Select the reactor **Position**.
   - Specify **Volume A** and **Volume B**.
2. Configure the **Tube Reactor**:
   - Specify the **Bore**.
3. Configure the **Preheat Tubing**:
   - Specify the **Bore** and **Length**.

Add Extra Tubing

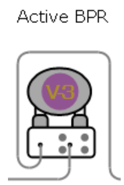


Click **Add Extra Tubing** to add extra tubing.

1. Configure the **Tubing**:
   - Specify the **Bore** and **Length**. The calculated **Volume** is displayed.

◦ Select **InlineWithPump** as the **Plumbing** option to move the component so it is only being fed from the preceding reagent or sample loop pump.

Add V3 Active BPR



The flow path is normally terminated with a passive BPR. Tick **V3 Active BPR** to replace the passive BPR with an active, controllable BPR.

1. Select the pump channel.
2. Specify the regulation pressure

Export Image

The displayed flow path can be exported to a Portable Network Graphics (PNG) file for viewing later. Browse to the export location and specify the filename.

7.5 Sample Loops Setup

The **Sample Loops Setup** screen appears if a sample loop has been added to the layout, see Figure 7.5.

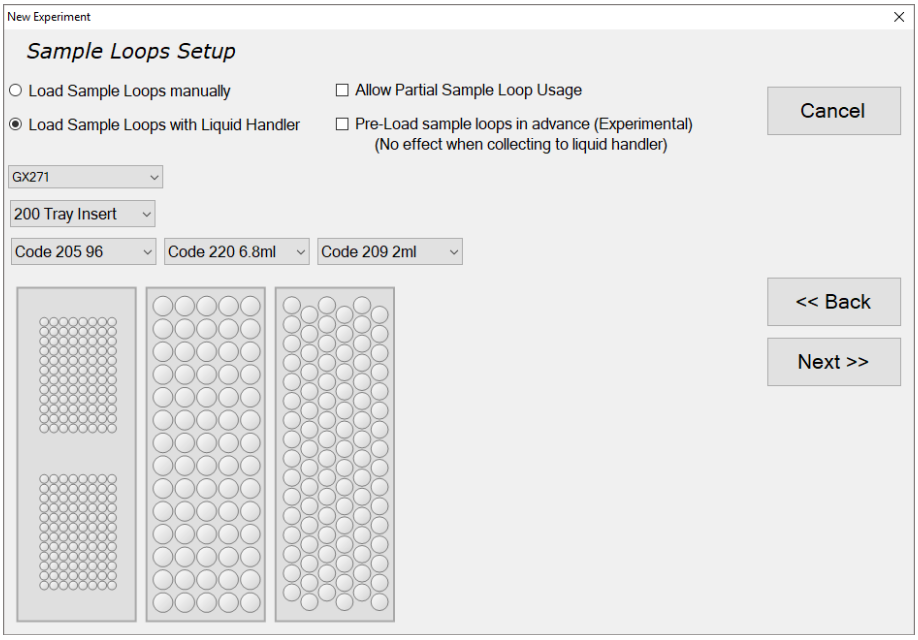


Figure 7.5: Sample Loops Setup

Loading Sample Loops

Select the option **Load Sample Loops manually** if using a syringe to manually load the reagents, or **Load Sample Loops with Liquid Handler** if using an autosampler.

A liquid handler device such as the 215, GX241 and GX271 can be configured to perform in:

1) a dedicated autosampling mode

2) a dedicated product collecting mode

3) an autosampling and product collecting mode

Tick the **Allow Partial Sample Loop Usage** option to allow a single sample loop fill to serve several reactions and hence reduce reagent waste.

Loading manually

Flow Commander prompts the user to load the reagents before each reaction is carried out.

Tick the **Allow Partial Sample Loop Usage** option to specify a limited volume of sample per reaction.

Loading with a Liquid Handler (Autosampler)

The reagents are loaded automatically before each reaction is carried out, allowing a large number of reactions to be carried out without intervention

Select the liquid handler, tray inserts and racks from the drop-down lists.

Tick the **Pre-Load sample loops in advance** option to allow the sample loop for a subsequent reaction to be loaded whilst the current reaction is ongoing, thus reducing the overall Experiment time.

If the same liquid handler device is also configured to collect product, this option is ignored as the probe will be in use collecting from the reaction when not loading a sample.

7.6 Reactor Setup

The **Reactor Setup** screen captures further configuration used by reactions, see Figure 7.6.

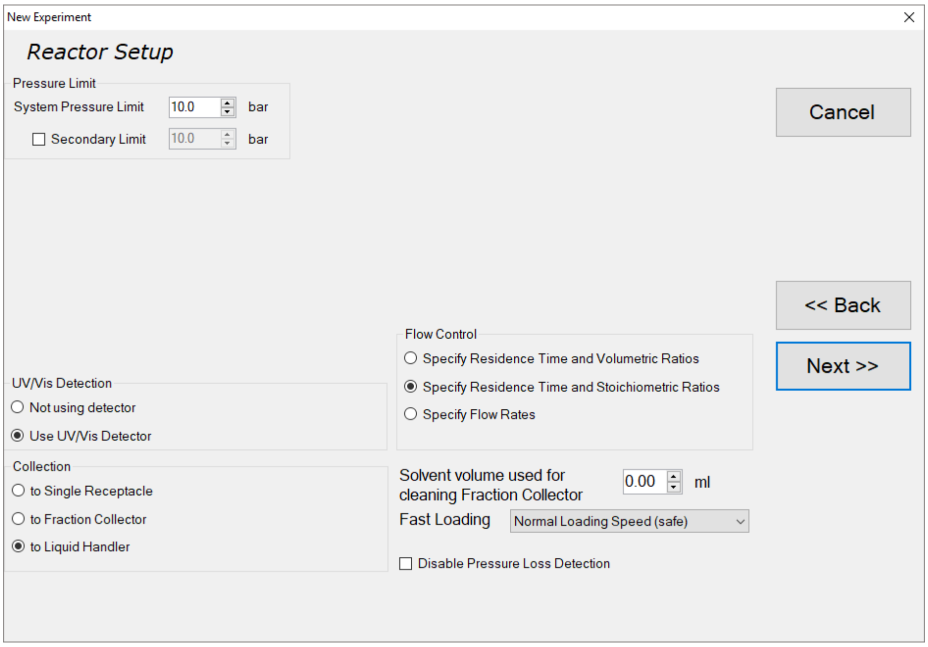


Figure 7.6: Reactor Setup

Pressure Limit

The system pressure limits are specified in the manual control pane, see section 10.1, but can be overridden by the Experiment.

Specify the pressure limits for primary and optional secondary pressure limit for the entire set of reactions. The pump module halts and Flow Commander reports an error if the system pressure exceeds this level.

Tick the **Secondary Limit** option to set a limit specifically for the secondary pump module.

Flow Control

Select how the flow rate is controlled (only editable when creating a new Experiment):

Residence Time and Volumetric Ratios

Specify reagents to be mixed by volume ratio, and by a residence time in the reactor. Flow Commander calculates the necessary flow rates.

Residence Time and Stoichiometric Ratios

Specify reagents to be mixed by molar ratio, and by a residence time in the reactor. Flow Commander calculates the necessary flow rates. The reagent concentrations must be set correctly.

Flow Rates

Specify the specific flow rates.

UV/Vis Detection

Select if an optical detector is used to trigger the collection of product from the reaction.

Collection

Select how the product is collected (only editable when creating a new Experiment):

Single Receptacle

A single collection is made for each reaction using a vial located directly under the collection valve on the pump module.

Fraction Collector

A fraction collector (or liquid handler in dedicated fraction collecting mode) collects and optionally fractionates each reaction's product. If a liquid handler is configured as the dedicated fraction collector, the entire bed can be used to collect the product, starting from site 1.

Liquid Handler

A liquid handler configured to collect and fractionate the product, in addition to autosampling.

A liquid handler device such as the 215, GX241 and GX271 can be configured to perform in:

1) a dedicated autosampling mode

2) a dedicated product collecting mode

3) an autosampling and product collecting mode

This option **should not** be selected if the liquid handler is not autosampling during the experiment.

Solvent volume for cleaning

Specify the volume of solvent used for cleaning the probe on the fraction collector or fraction collector/autosampler between reactions.

Fast Loading of reagents

The flow rate of the reagents can be increased up to a fixed location in the flow path in order to reduce the Experiment duration. Flow Commander calculates the pump speed to achieve the required flow rate increase for each reagent depending on the selected option:

Normal Loading Speed

Safest option; flow rates of reagents are not increased.

Accelerate to first Tee

Less safe; flow rates of reagents are calculated to the first tee-piece.

Accelerate to first Reactor

Risky; flow rates of reagents are calculated to the first reactor and may be unsuitable where the accelerated mixing of reagents could cause highly corrosive or exothermic reactions.

Disable Pressure Loss Detection

Tick **Disable Pressure Loss Detection** to prevent the Experiment halting when a sudden pressure loss is detected i.e. a drop from established system pressure down to atmospheric pressure.

7.7 Collector Setup

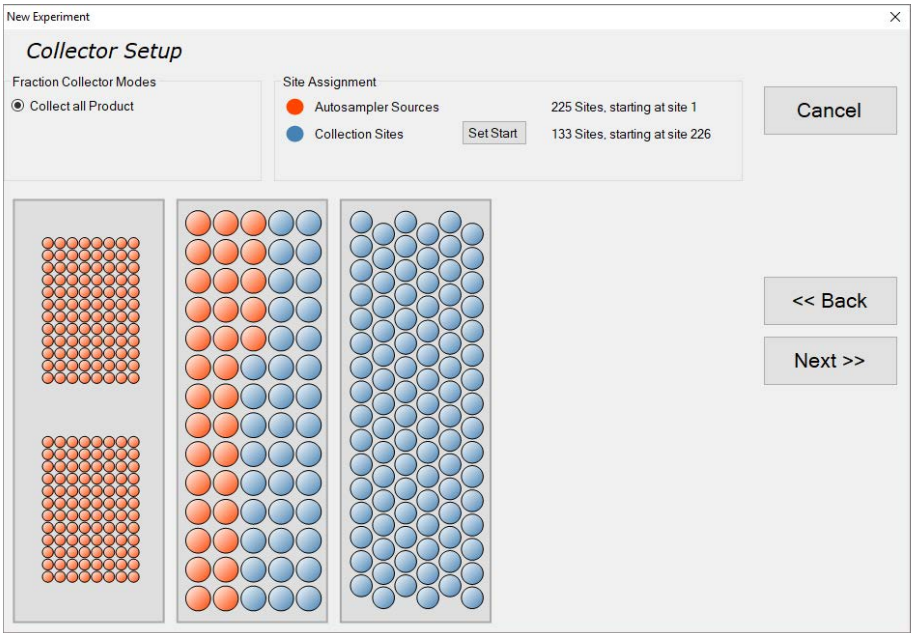


Figure 7.7: Collector Setup

The **Collector Setup** screen, see Figure 7.7, is displayed if a fraction collector or liquid handler is selected to collect the product, see section 7.6.

If a fraction collector is used, select the device, tray inserts and racks from the drop-down lists.

If a liquid handler is configured to collect the reaction product and is already configured to be an autosampler, the bed configuration will be fixed as per the autosampler configuration.

Site Assignment

When using the same device as both autosampler and fraction collector, the bed must be partitioned into separate sample sources and collection sites:

1. Click **Set Start** to display a crosshair.
2. Move the crosshair to a convenient collection site.
3. Click to assign the site as the start of the collection.

Autosampler sites are coloured red and reserved for reagents. Collection sites are coloured blue and can be used for loading into a sample loop in order to cycle reaction products back into the system to carry out additional reactions.

To change the bed configuration, click **Back** to return to the **Autosampler** screen.

7.8 Summary

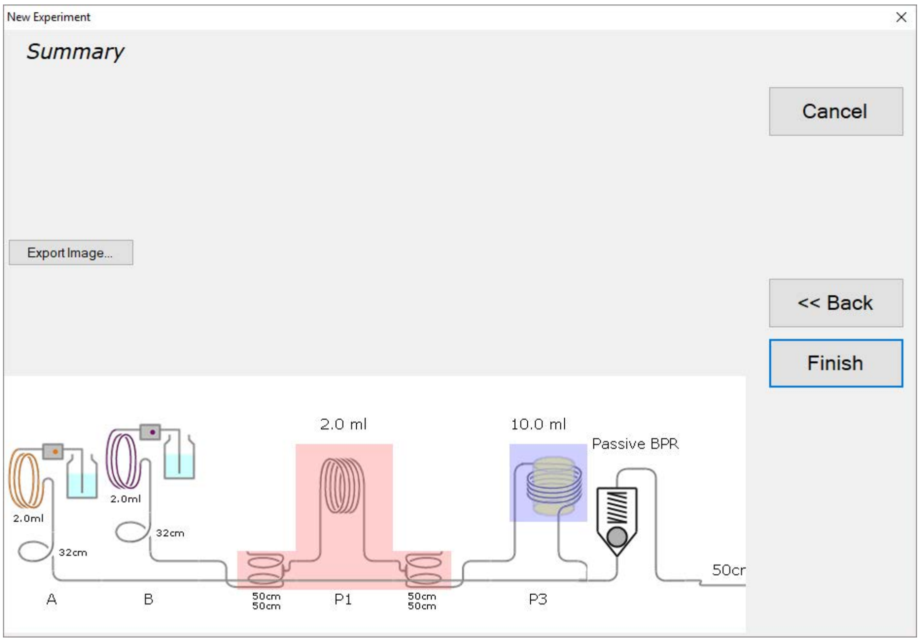


Figure 7.8: Experiment Summary

The **Summary** screen displays the flow path layout as displayed in the **Custom Plumbing Setup** screen, see section 7.4, and additionally includes the tubing between the BPR and the collection device.

Click **Export Image...** to export the flow path as a Portable Network Graphics (PNG) file for viewing later. Browse to the export location and specify the filename.

8 Adding Reactions

Experiments contain a number of reactions and are performed in a defined sequence.

After creating a new Experiment, Reactions are defined using the **Add Protocol** screen, see Figure 8.1. Alternatively, click **Add** from the Experiment pane.

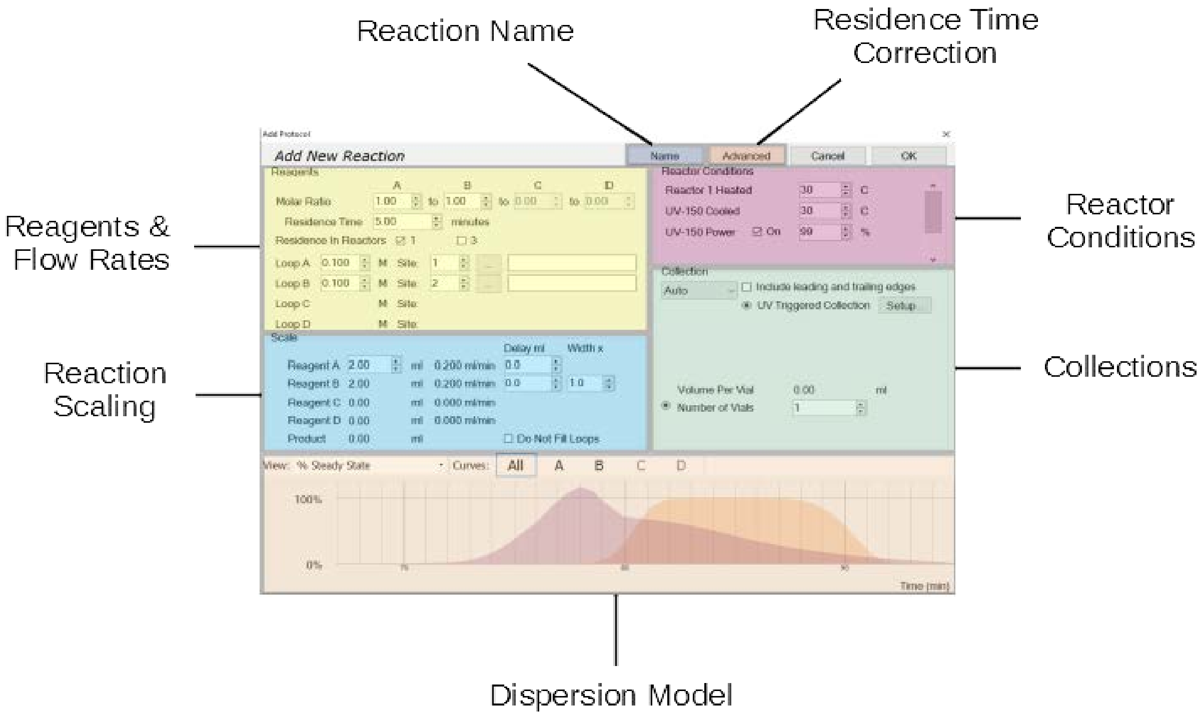


Figure 8.1: Add Protocol

8.1 Naming the reaction

Click **Name** to enter the name of the Reaction. Click the **?** button to display codes that can be inserted into the name as a template, see Figure 8.2.

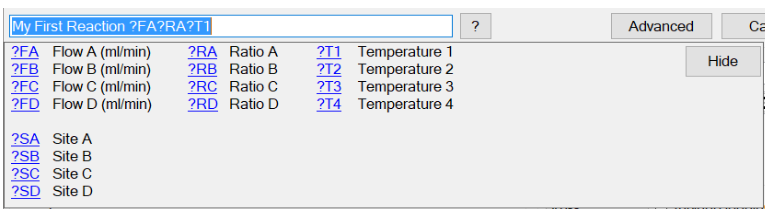


Figure 8.2: Reaction Name Codes

Click **Hide** to return to the **Add Protocol** screen.

Template Format

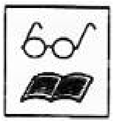
Click one or more key reaction parameters to form the Reaction name. e.g. “**My First Reaction ?FA ?FB ?RA ?RB ?SC**”

Each **?**xx symbol is replaced by the actual parameter value.

8.2 Specifying reagents and flow rates

The **Reagents** section controls the pump flow rates for each reagent. The method of specifying flow rates vary depending on the Experiment setting, see section 7.6.

Tick those reactors, including external reactors, across which the total **Residence Time** applies.



If a dual core reactor is selected for residence time calculations, only Volume A of the reactor is used, and all other reactors are excluded for the calculation. Hence, if a dual core reactor is ticked, all other reactors will be unticked.

The concentration of bottled reagents is fixed for an Experiment, and is present in the Experiment settings.

When using an autosampler, specify the molar concentration and site from which to load each sample loop. Alternatively click the **...** button and click on the individual site, see Figure 8.3.

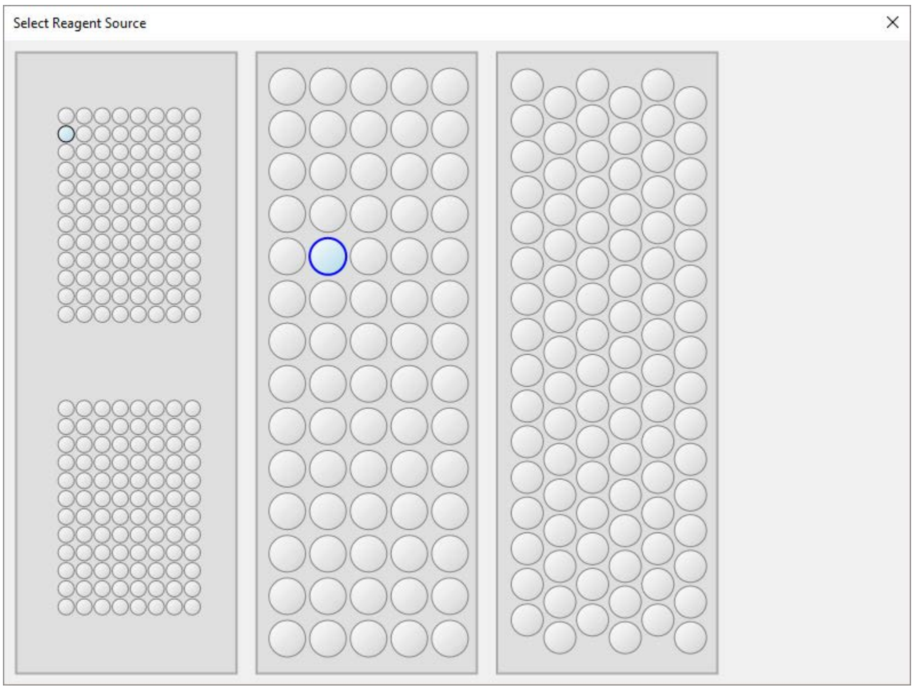


Figure 8.3: Reagent Source

Add a note if required.

Setting one of the ratio values or flow rate to zero will disable the associated pump for this reaction.

The calculated flow rates appear in the **Scale** section.

When a pump is positioned in the flow path between two reactors, each reactor has a different flow rate. Selecting both reactors to calculate the residence time calculation may generate erroneous values.

Specifying flow in ml/min

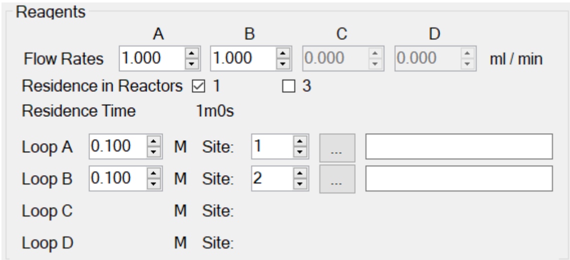


Figure 8.4: Specify Reagent Pump Flow Rates

The flow rate of each pump is entered directly, see Figure 8.4. Flow Commander calculates the required flow rates to obtain the desired residence time.

Specifying flow as a volumetric or stoichiometric ratio

The desired residence time is specified as well as the volumetric or stoichiometric relationship between the reagents, see Figure 8.5 and Figure 8.6 respectively.

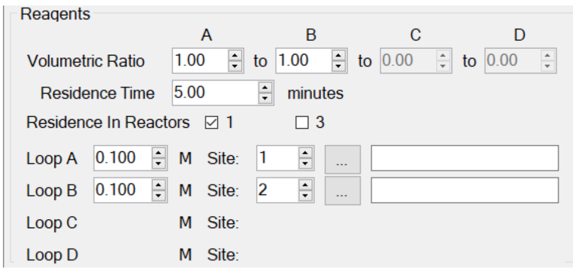


Figure 8.5: Specify Reagent Volumetric Ratios

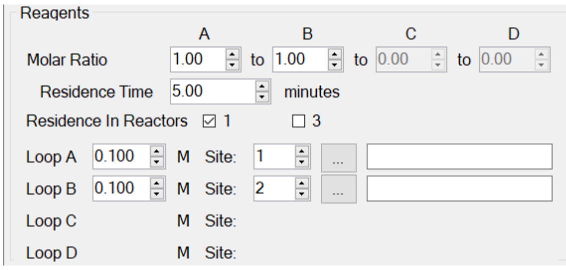


Figure 8.6: Specify Reagent Stoichiometric Ratios

Flow Commander calculates the required flow rates to achieve the desired residence time across the selected reactors. If more than one reactor is selected, the path within in the reactors increases resulting in a higher calculated flow rate.

The reaction cannot be added if the required flow exceeds the capability of the pumps.

8.3 Setting the Reactor conditions

Specify the operational parameters for each reactor in the flow path e.g. temperature, output power, gas flow rate etc.

8.4 Correcting the Residence Time (Advanced)

The solvent's density and hence solvent flow rate may be affected by the reactor pump material's composition or temperature. Click **Advanced** to specify the compensating **Correction Factors** for each reactor which are applied when calculating residence times. Click **Standard** to display the reactor conditions.

8.5 Reagent Dispersion View

The reagent dispersion **View** displays the concentration of each reagent at the collection valve across time.

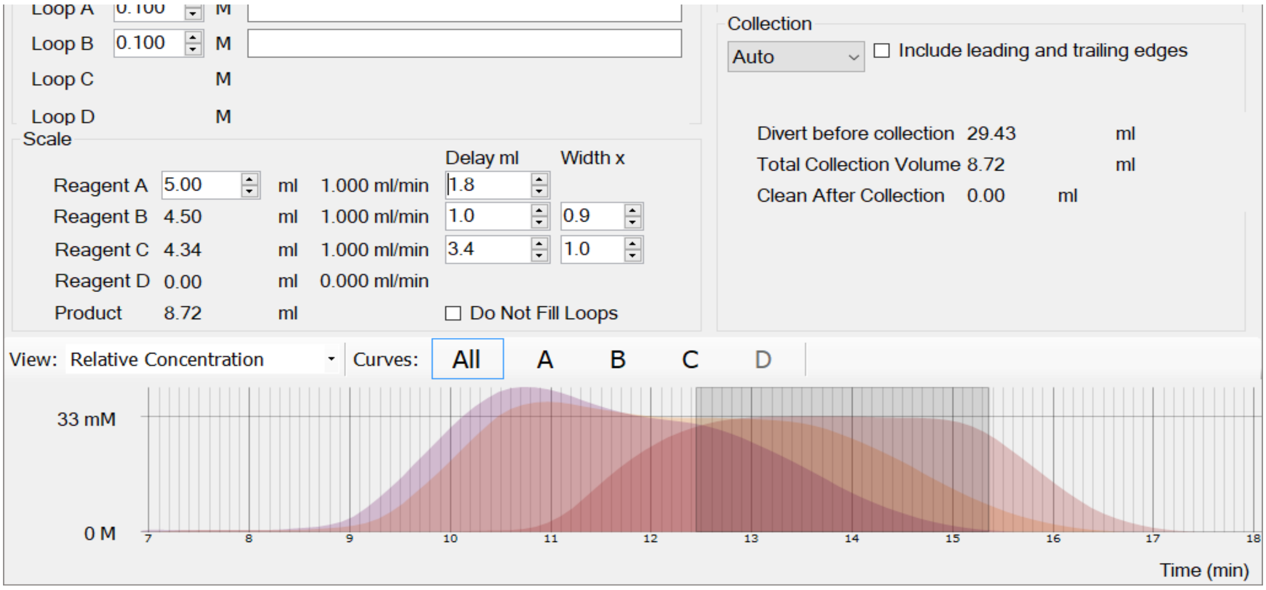
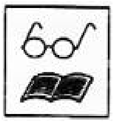


Figure 8.7: Dispersion View



The dispersion model assumes that the dispersion is not affected by the reaction of reagents.

Select between **% Steady State** and **Relative Concentration** views from the drop-down list.

Select the **Curves** to be displayed. By default, all reagent curves are displayed. Selecting individual reagents displays the other reagents as shadowed curves.

The collection window is displayed as a grey rectangle and reflects the period during which the collection valve is open and the product is being collected.

8.6 Scaling the Reaction

The **Scale** section allows the amount of reagent used in the reaction to be adjusted. This controls the scale but not the stoichiometry or duration of a reaction, and hence does not affect the chemistry. The scale may have an effect on the success of a reaction, for example, if the scale is too small for steady state conditions to be achieved.

The amount of collected product is calculated by the model and displayed.

For example, Figure 8.8 shows a reaction where 7.95ml of product is collected. Simply increasing Reagent A from 5ml to 10ml results in 15.30ml of product being collected. Reagent B is automatically increased to maintain the chemistry. The product collection window, highlighted in grey, increases commensurately, see Figure 8.9.

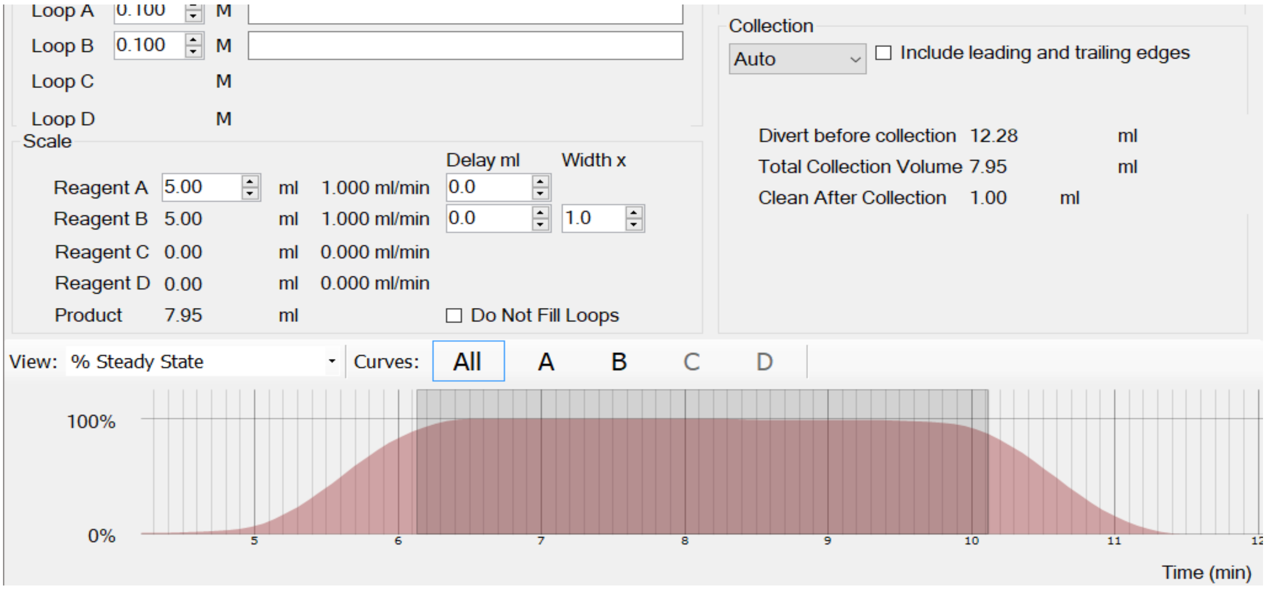


Figure 8.8: Reaction Before Scaling

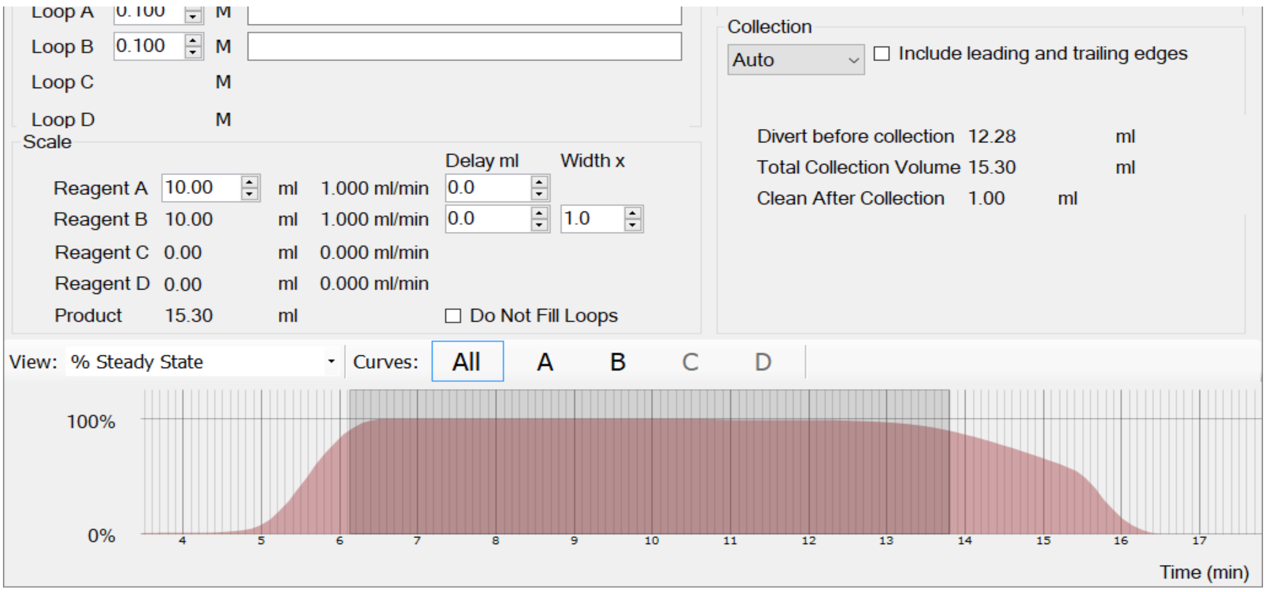


Figure 8.9: Reaction After Scaling

Scaling with Bottled Reagents

If a sample loop is not used, the reaction is scaled up by specifying the volume of bottled reagent to dispense.

Specify the **Delay** volume to delay the addition of the reagent, see Figure 8.10. A negative delay results in the reagent volume being added earlier.

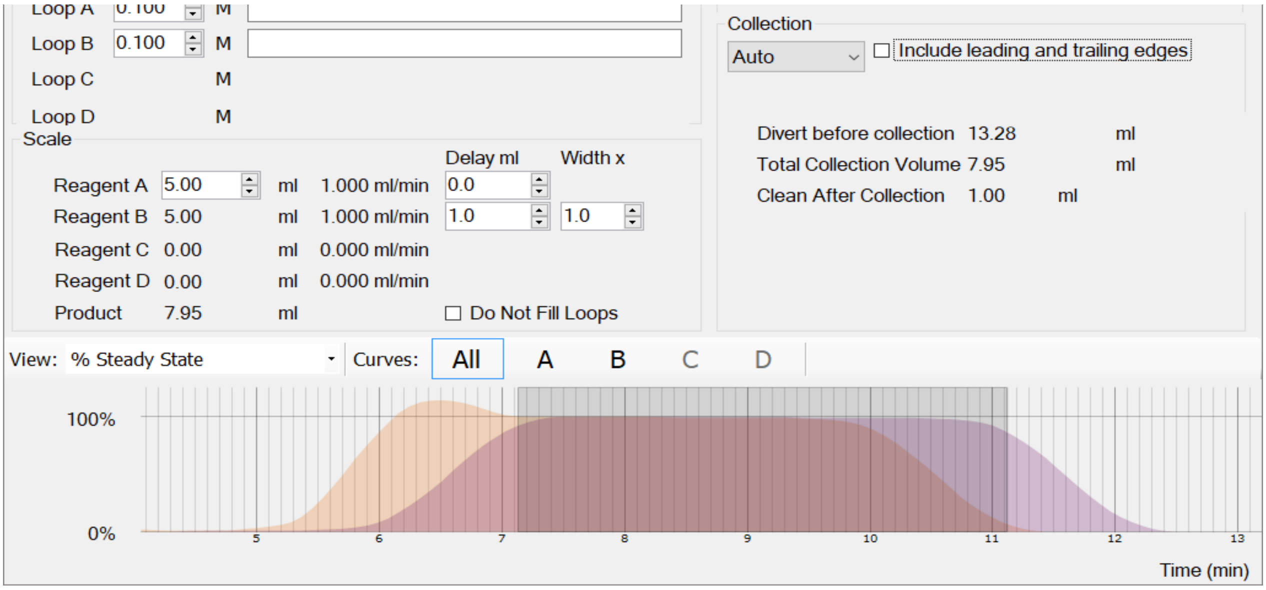


Figure 8.10: Adding a delay to Reagent B

Specify the **Width x** to apply a scaling factor to the volume of reagent used. See Figure 8.11, where the volume of Reagent B has doubled without increasing Reagent A, useful when Reagent A is an expensive or scarce resource.

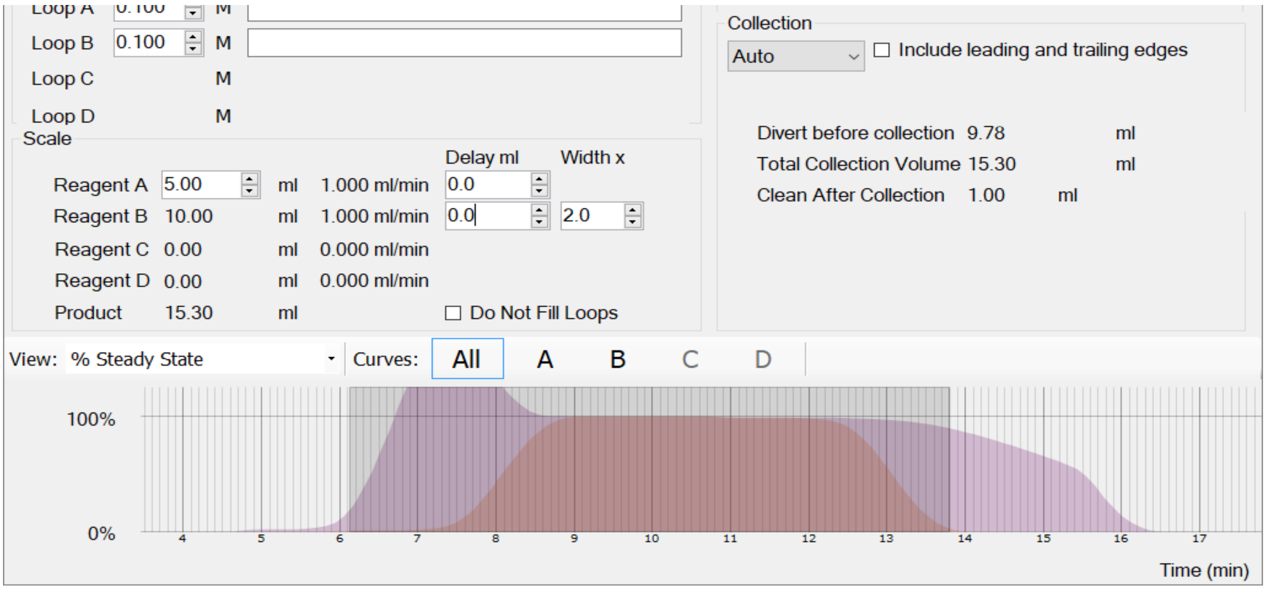


Figure 8.11: Increasing the Width Multiplier of Reagent B

Flow Commander matches the peak from subsequent bottled reagents to the first one in the flow path in order to maximise the resultant product.

Scaling with Sample Loops

Scaling is governed by the size of the sample loops, see section 6.3 for configuration. Flow Commander synchronises the timing of other pumps to match the peaks.

Specify the **Delay** volume to delay the addition of the reagent. A negative delay results in the reagent volume being added earlier.

The first reaction of an Experiment requires the **Do Not Fill Loops** option to be left unticked. This ensures the sample loop is filled with reagent at the start of the Experiment. Tick the option if the sample loop can service subsequent reactions without needing to be re-filled.

8.7 Specifying collections

The **Collection** section allows collection related parameters to be set.

Select the collection mode from the drop-down list. The selected mode determines whether recommended parameters are used based on the flow path model, and cleaning regime.

By default, values are shown based on Flow Commander calculating the collection window to achieve a minimum of 95% concentration of the last reagent in the flow path. Tick the **Include leading and trailing edges** option to achieve a minimum of 5% concentration.

The values calculated by Flow Commander are based on a model and hence provided as guidance; the values are not guaranteed.

Specify if the product is split across one or more vials and if the volume is limited per vial.

Auto

The volume of product to be collected, amount to be discarded falling below 95% (or 5% if selected), and solvent used for cleaning post-collection is fixed.

Specify the volume of product for each vial or how many vials to share the product across.

Manual

Specify the amount of solvent to discard (**Divert before collection**). Discarding more solvent before the collection value is opened means that a lower concentration of reagent would be dispensed to achieve the same overall volume of product.

Specify how much product to collect. A collection volume greater than that displayed by default (or in **Auto** mode) means that the concentration of the last reagent in the flow path is likely to be less than 95% according to the model.

Specify how much solvent to use once collection is complete.

None

The collection valve diverts all the product at the collection valve to waste.

Specify how much solvent to use once collection is complete.

This mode is useful when the product of real interest is retained in a component of the flow path e.g. a the surface of a column reactor, and is required by a subsequent reaction.

UV Triggered Collection

If UV/Vis detector is enabled in the **Reactor Setup** screen, click **Setup...** to configure the detection parameters, see Figure 8.12.

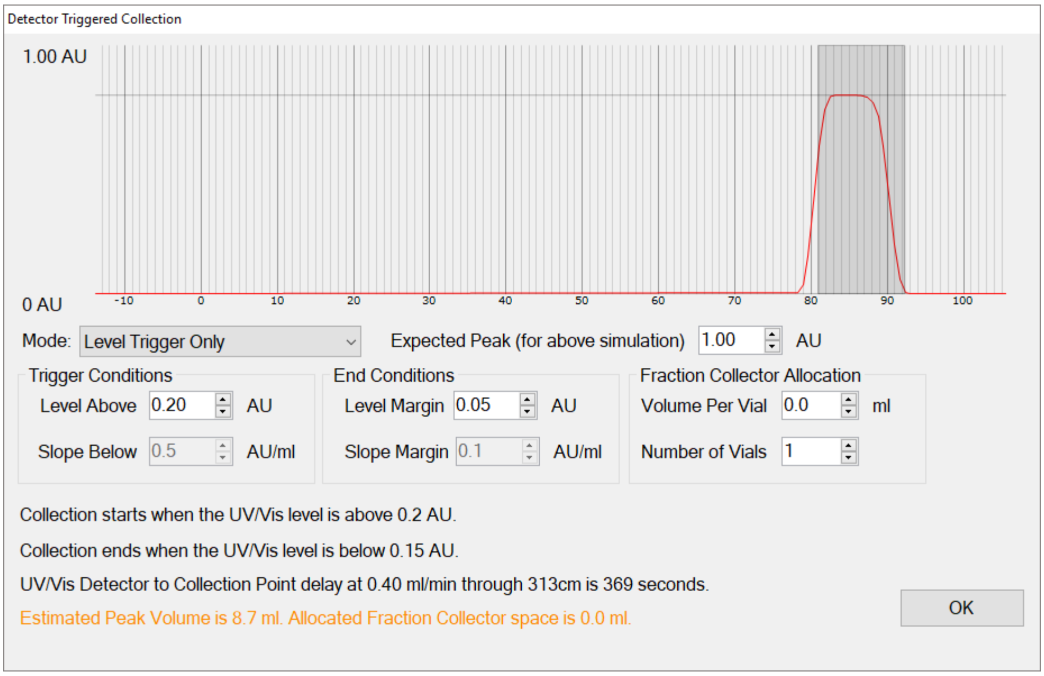


Figure 8.12: UV/Vis Detector Triggered Collection

Reset Detectors

UV/Vis detectors can be reset using the Detector menu.

- Click **Zero** to set the current detector reading as the new baseline; the value of zero is logged and displayed on the graph.
- Click **Restart** to reset the communications interface to the detector.

9 Running the Experiments

9.1 Experiments Pane

The **Experiments** tabbed pane displays information related to the Experiment including consumables, defined reactions consisting of individual actions, and product collection, see Figure 9.1.

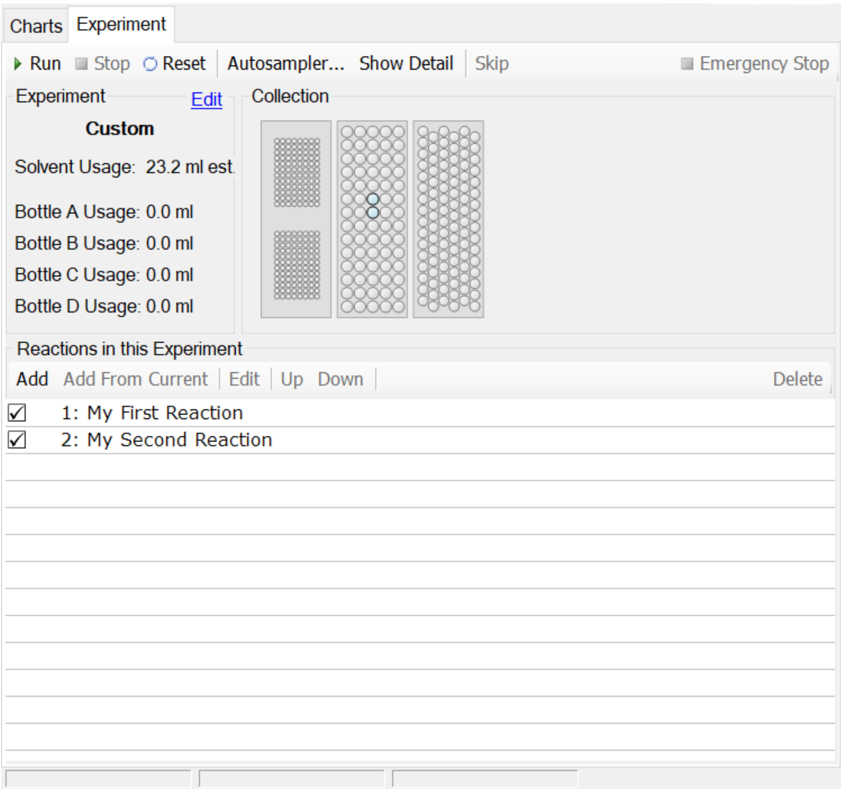


Figure 9.1: Experiment Pane

Experiment menu buttons

Buttons manage the experiment, reactions and actions:

- **Run**, Run, performs all the selected reactions listed in the Reactions in this Experiment section below.
- **Stop**, Stop, halts the current reaction. The experiment preamble is then run e.g. to run the cleaning regime.
- **Emergency Stop**, Emergency Stop, halts the current reaction. The experiment preamble is **not** run.
- **Reset**, Reset, clears the list of reactions already run. The entire experiment can then be run from a known state.
- **Skip** aborts only the current action, and only applies to specific **Wait** actions.

- **Show Detail** displays the detailed list of actions that are carried out in sequence.
- **Autosampler...** displays a number of buttons that are used to control the autosampler in preparation for the start of the experiment, see Figure 9.2.

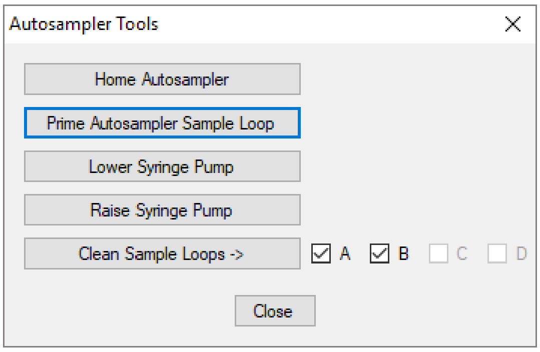


Figure 9.2: Autosampler Tools

Experiment

The Experiment section displays the volumes of consumables used. Ensure the quantities of solvents and reagents are available.

Click **Edit** to view or modify details of the Experiment.

Some configuration options and parameters may not be editable if reactions have been defined. A new experiment must be created to change these parameters.

Collection

The **Collection** section highlights those sites on the bed where the product will be collected. Sites are strongly highlighted when a single reaction is selected below.

Reactions in this Experiment

This section displays the full list of ordered reactions associated with the experiment and are managed using buttons:

- **Add** defines a new reaction using the experiment defaults.
- **Add From Current** defines a new reaction based on the selected reaction experiment.
- **Edit** modifies the selected reaction.
- **Up** moves the reaction to an earlier position in the sequence.
- **Down** moves the reaction to a later position in the sequence.
- **Delete** discards the reaction.

The reactions are numbered and listed by name (or reaction parameters if a name has not been provided). Tick a reaction to enable it for running at any time unless the experiment is underway and the reaction was scheduled to run in the past.

A reaction is coloured green when successful and red when failed e.g. if stopped by the user.

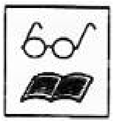
Click **Show Detail** to display every action of the experiment including:

- actions related to the experiment preamble e.g. initialisation of components
- current action being run, marked by a green arrowhead
- actions already run, marked with a green tick symbol if successful or red cross if unsuccessful e.g. stopped by the user
- remaining actions
- disabled actions, coloured grey
- actions related to the experiment postamble e.g. cleaning phase, resetting pumps and valves
- informational actions prefixed with the # symbol e.g. comments marking the start of a reaction, operation or phase such as experiment postamble.

All experiment and reaction data is logged and saved to the report.

10 Controlling the R2 pump modules and R4 reactor heater modules

To manually control the R2 pump and R4 reactor heater modules, Flow Commander requires a connection to the primary pump module. The pump module and reactor heater module region is populated depending on the connected hardware.



Flow Commander provides additional control functionality beyond that available when using the physical configuration interface consisting of buttons, dials, and segment display, located on the front panel of the R2 pump and R4 reactor heater modules.

10.1 System pressure

The system pressure gauges indicate current system pressure and upper pressure limit for both primary (**System I**) and secondary (**System II**) pump modules, measured inside the manifold, see Figure 10.1. For most systems, set the primary and secondary upper limits to the same value unless a back pressure regulator (BPR) is installed between primary and secondary tubing, causing pressure differences.

Figure 10.1: System Pressure

The entire system trips i.e. all reactor heaters and pumps switch off, if either primary or secondary system pressure measured inside the manifold, exceed the set pressure limit, and is indicated by the start of the red line on the dial.

Click **set limit** to adjust the pressure limits, see Figure 10.2.

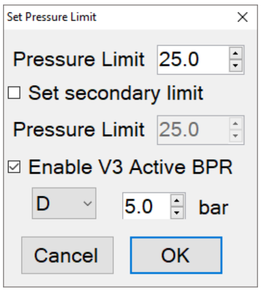
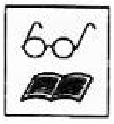


Figure 10.2: Set System Pressure Limit



If R2S pump modules are present in the system, one V3 pump can be assigned to actively regulate back pressure via this setting screen. Using the V3 pump in this way can only be achieved using Flow Commander and not from the hardware interface.

10.2 Pump pressure

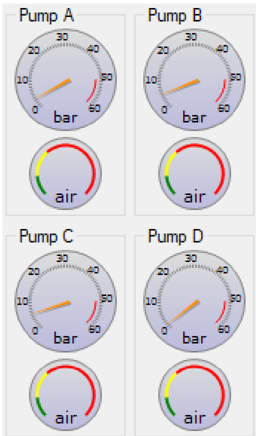


Figure 10.3: Pump Pressures and Performance

Gauges display the current pressure and upper pressure limit for each individual pump in the system, see Figure 10.3. The limit is fixed at 50 bar for standard pressure pumps.

10.3 Pump performance

Gauges accurately indicate the pump performance, defined as the amount of air in the pump, for each individual pump in the system, see Figure 10.3.

The gauge needles do not appear until performance information is available and may take several seconds after the pump has been started and dependent on the pump speed; very low speeds will take longer.

If the pump is free of air and performing to specification, the needle will be in the green area.

Air

Air in the pump will move the needle into the yellow or red areas, which indicates the pumps are not delivering the requested flow rate. The pump channel should be primed to remove the air.

The R2S pump module is fitted with self-priming V3 pumps.

Difficult Liquids

Some mixtures (often of immiscible solvents) cannot be pumped well with the Vapourtec positive displacement piston pump, causing a severe degradation of pump performance; the needle will move to the red area and **Air** will flash on the display.

10.4 Power

The **Power** button, ⏻ Power, starts the pumps and/or reactor heaters with the symbol colour indicating the status:

- Black – Not running
- Green – Pumps and/or Reactor Heaters are running
- Red – Not running, an error occurred

The pumps run at the speed specified in the pump speed region. The reactor heaters heat to the temperatures specified in the reactor temperature region, if the reactors have temperature sensors connected. Unused reactor positions with no temperature sensor detected are not heated.

1. Set the reactor temperature to 20°C to run the pumps without any heating.
2. Set the flow rate to zero to heat without running the pump.

10.5 Back Pressure Regulator (BPR) Purge

Click **BPR Purge** to purge the V3 pump safely when used as an active back pressure regulator, equalising the pressure with the surrounding atmosphere.

10.6 Pump Speed

The **Pump Speed** section, see Figure 10.4, shows the desired pump speeds required to maintain specified flow rates and are set by clicking on the section. The actual flow rate is affected by the pump performance as displayed on the air gauge.

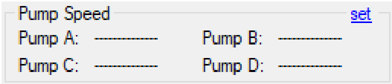


Figure 10.4: Pump Speed

10.7 Reactor Temperature

The **Reactor Temperature** section shows both the current and desired reactor temperatures and are set by clicking in the section. For example, **30/40** indicates the current temperature is 30°C and desired temperature is 40°C.

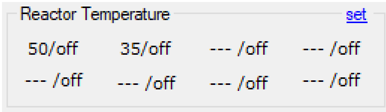


Figure 10.5: Reactor Temperature

It is only possible to set the temperature of a reactor heater if the channel has a temperature sensor connected, otherwise the channel is disabled.

10.8 Valve Control

The **Valve Control** section, see Figure 10.6, resembles the R2 physical indicator buttons to control the pump module valves (solvent/reagent, sample, and collection valves) and to display their status. The colour scheme can be set in the **System Configuration** menu to match the colours on the pump module hardware, see section 6.1.

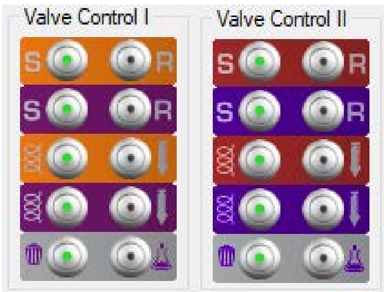


Figure 10.6: Valve Control

11 Analysing the chart data

The chart pane displays a graphical representation of sensor/model data and conditions over time, see Figure 11.1. Live data is charted whenever Flow Commander is connected to the primary pump module, see section 4.3.

Figure 11.1: Chart Pane

Flow Commander logs:

- Reactor temperature (°C)
- Pump pressure (bar)
- Flow (µl/min)
- Duty Power % for cooled reactor valve modulation & Watts (W) for reactor heaters
- Valves positions for reagent/solvent, sample load/inject and collection
- UV/Visual light absorption (AUFS)
- Dispersion model (steady state %)
- Column (mm) – for compression controller

Each graph displays the data unit along the Y axis versus time along the X axis and show multiple channels if applicable. Click on the magnifying glass icons to control scaling in time. The Y axis scales automatically depending on the range of the logged data.

11.1 Views

The sensor/model data and conditions are logically grouped into 'Views' and selected from the drop-down control, see Figure 11.2.

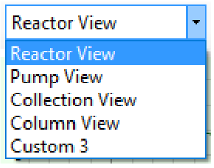


Figure 11.2: Chart View Selection

Table 2 lists the data included in each view. The Custom view allows the user to choose which three data values are displayed.

Data	View			
	Reactor	Pump	Collection	Custom
Reactor Temperature	✓			✓
Pump Pressure	✓	✓		✓
Flow		✓		✓
Duty Power (cooler)	✓			✓
Heater Power	✓			✓
Valve Positions		✓	✓	✓
UV/Visual absorption			✓	✓
Dispersion model			✓	✓

Table 2: Data Displayed Per View

11.2 Optimising the collection of produced material

The steady state dispersion model is calculated based on locations in the flow chemistry system. Select the dispersion model from the drop-down list, see Figure 11.3, to display a single graph of the model at the UV/visual light detector site, sample collection site or a combined graph.

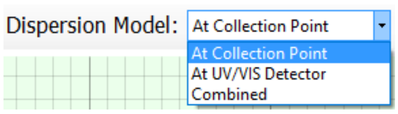


Figure 11.3: Dispersion Model Selection

The dispersion model graphs can be compared in time alongside the UV/visual light detector graph.

11.3 Emergency Stop

Emergency Stop, Emergency Stop, halts the current reaction. The experiment preamble is **not** run.

11.4 Managing logged chart data

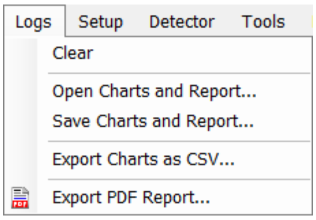


Figure 11.4: Logs Menu

Logged data is managed from the **Logs** menu, see Figure 11.4:

Clear

All logged data visible in the charts is discarded.

Save Charts and Report

Flow Commander disconnects from the primary pump module and saves the data to a Flow Commander Chart file.

Open Charts and Report

Flow Commander disconnects from the primary pump module before allowing the user to specify the Flow Commander Chart file containing data to display on the chart pane.

Export Charts as CSV

The logged data is saved to a file in Comma Separated Value (CSV) format to allow further processing using a spreadsheet software program such as Microsoft Excel®.

Export PDF Report

- A printer-friendly PDF report, see Figure 11.5, is generated and includes:
- Date & time corresponding to the start of the logged data
- System configuration details

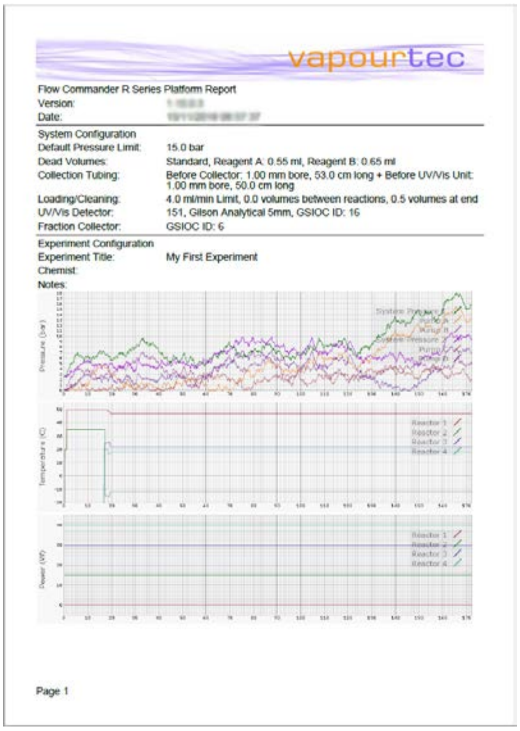


Figure 11.5: PDF Report

12 Using the helper tools

Flow Commander includes a number of useful tools to administer the flow chemistry system, see Figure 12.1:

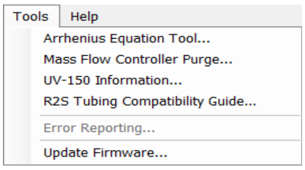


Figure 12.1: Tools Menu

12.1 Arrhenius Equation Tool

This tool calculates the increase in reactor residence time of a reagent for an increase in temperature.

1. Specify the reactor residence time for a particular temperature.
2. Specify the activation energy.
3. Specify the new temperature. The new reactor residence time is calculated.

12.2 Mass Flow Controller Purge

This tool purges a Mass Flow Controller when configured for use in an experiment.

12.3 UV-150 Information

This tool displays the status information retrieved from the Vapourtec UV-150 photochemical reactor.

12.4 R2S Tubing Compatibility Guide

This tool displays the reagent suitability of each tube type, red or blue, supplied by Vapourtec for use in the R2S pumping module.

12.5 Error Reporting

This tool allows error reports generated by Flow Commander to be saved to a USB memory stick for analysis by Vapourtec.

12.6 Update Firmware

This tool upgrades the firmware on the Vapourtec R-series components including R2 pump modules, R4 reactor modules, V3 pump modules and UV150 photochemical reactor.

Contact the Vapourtec service team (service@vapourtec.com) for information on firmware updates.

End of document.