



## FlowLab™ User Manual



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## 1 Safety

The following symbols mean:



Caution: Read these operating instructions fully before use and pay particular attention to sections containing this symbol.



Caution: Surfaces will become hot during use.

### Always observe the following safety precautions



Do not check the temperature by touch!



Do not touch surfaces which become hot during high temperature operation.



Use only as specified by the operating instructions or the intrinsic protection may be impaired.



The user must wear safety glasses and suitable personal protective clothing.



It is the responsibility of the user to assess the risks associated with the substances to be used in the FlowLab and put in place the necessary safety precautions for the substances in question.



Connect only to a power supply with a voltage corresponding to that on the serial number label at the side of the unit.



Connect only to a power supply which provides a safety earth (ground) terminal.



Before moving, allow to cool and disconnect at the power supply socket.



If liquid is spilt inside the unit, not the drip tray, disconnect it from the power supply and have it checked by a competent person.



Before using any cleaning or decontamination method except those recommended in this manual, users should check with Uniqsis Ltd that the proposed method will not damage the equipment.



The unit must be placed on a level, non-flammable surface away from bulk flammable materials ensuring that all ventilation slots in the base are clear from obstructions.



The user should consider the *auto ignition point* of solvents when performing the risk assessment.

## 2 General Information

### 2.1 Introduction to FlowLab

#### Accessible Flow Chemistry

FlowLab™ is a starter flow chemistry system comprised of 2 or 3 high pressure pumps and up to 3 reactor modules for heating (HotCoil™) and or cooling (PolarBear Plus™) in the range -40°C to 260°C. It is supplied with the FlowLab software application that allows single chemical reactions to be programmed and controlled remotely by Wi-Fi. Experiment data is logged and saved in a .txt file. For convenience, previous experiments can be reloaded. In addition, data can be imported into eLNBS or spreadsheet software such as Excel and archived or reprocessed externally.

Depending upon the size of the pump heads fitted (10 ml/min or 50 ml/min), experiments are possible from mg to kg scale.

FlowLab has been designed as a cost-effective solution for flow chemistry. The system can be upgraded with an extra pump, additional reactor stations or with an in-line UV/VIS analytical detector with a high pressure flow cell.

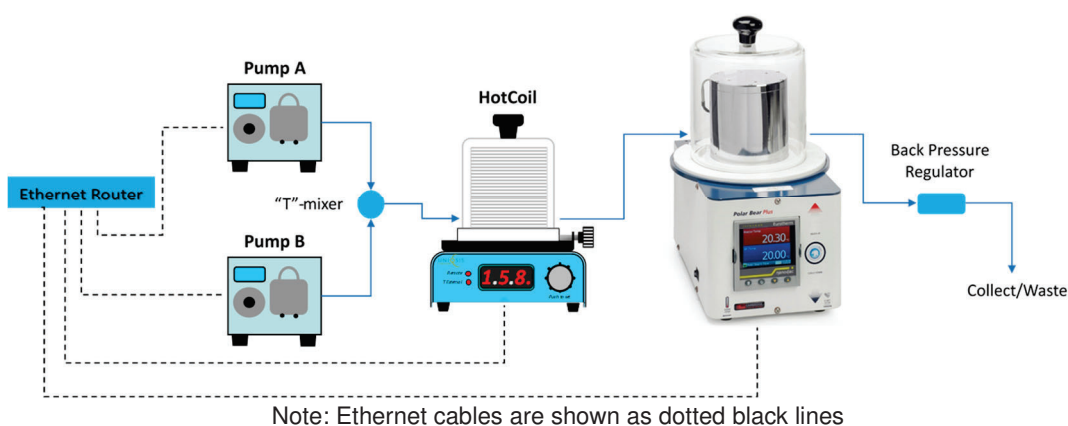
**Space Saving** — Compact footprint, fully integrated system.

**Safe** — Each high pressure pump has a built in pressure transducer and the FlowLab system control software allows safe operating pressure limits to be set. The HotCoil™ incorporates over temperature cut-outs.

**Efficient** — FlowLab allows either manual or automated control of the system and incorporates both priming and wash sequences.

**Straightforward** — The FlowLab control software has been designed to be as intuitive as possible and allows the system to be controlled remotely using Wi-Fi.

### 2.2 System Schematic of 2 channel FlowLab



## 2.3 Image of 3 channel FlowLab with dual reactor stations and Flow-UV



The FlowLab™ system comprises up to 3 high pressure pumps and 2 reactor modules selected from HotCoil™, HotChip™, Polar Bear GSM™ or Polar Bear Plus™ reactor stations under the control of FlowLab software application. The computer, pumps and HotCoil™ are connected over a LAN using an Ethernet hub. A combined hub/router is supplied, the system can be controlled remotely by Wi-Fi, allowing the control computer to be conveniently operated outside the fume hood. FlowLab Cold™ includes the Polar Bear Plus module which provides cooling down to -40°C without the need for liquid nitrogen or cardice.

The system can be further adapted using the HotColumn™ which can accommodate up to six column reactors for catalysts or scavenging applications, or in the case of FlowLab Cold™, fitted with a static reactor/mixer chip holder. In addition, there are options to add a third HPLC pump and a second reactor module such as a HotCoil™ or Polar Bear Plus™ which together provide an operating temperature range of -40°C to +300°C. An optional inline Flow-UV™ detector can also be included to monitor dispersion and help to determine the optimum point at which to begin and end product collection.


The FlowLab™ application provides control of the system via a step-through interface that is straightforward to use. Experiments can be planned and saved or uploaded from file. Reactions run automatically, including priming and washing. The program is monitored on a real time display

showing temperatures and pressures. FlowLab™ also automatically detects any modules that have been added to the system.

## 3 Getting Started

### 3.1 Unpacking

Remove packing materials carefully and retain them for future shipment or storage of the unit if required.

 **Caution:** Components can weigh up to 22kg - units should be lifted by placing hands underneath the casework to avoid damage.

### 3.2 Pack Contents

Pack contents vary according to the system purchased. A basic FlowLab system is comprised of the following components:

2 x High pressure pump units with pressure transducers, low pressure manual inlet selection valves and outlet BPR upgrades

1 x Reactor module (either a HotCoil, HotChip, Polar Bear *Plus* or Polar Bear GSM)

1 x 5ml HT PFA coil reactor

1 x 5ml 316L SS coil reactor

1 x Equipment stand

1 x Low pressure waste/collect valve

3 x Power cables

3 x Ethernet cables

1 x Wi-fi router/hub

1 x Router power supply

1 x Accessories kit including connection tubing and tools

1 x Back pressure holder (outlet) and 100psi BPR cartridge

5 x Bottles and caps

1 x Laptop computer with FlowLab v7.0 (or later) software application

1 x FlowLab User Manual

Optional items:

3<sup>rd</sup> Channel HPLC pump

Flow-UV inline detector

HotColumn column holder and columns

Set of 5 chemically resistant back pressure regulators

Other coil reactors

### 3.3 Assembly

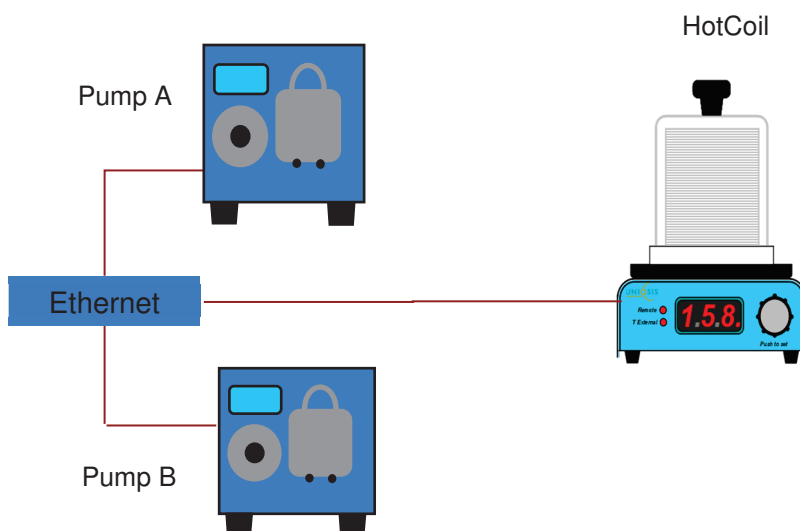
The FlowLab system **must** be placed inside a fume hood. A 5 cm gap should be left at the back to allow the cooling from the rear panel.

Plug each pump into its power supply and into a suitable main socket. Connect the HotCoil to a suitable IEC outlet. The mains adaptor socket is on the rear panel.

Turn the HotCoil on at the on/off switch on the rear of the unit.

The pumps have an on/off switch located on the top of the attached power supply.

### 3.4 Ethernet connection



**Wi-fi Router Password: Uniqsis#123**

The above diagram shows the connection for a 2 pump reactor system, for a 3 pump system and or 2 reactor system the connection are exactly the same just connecting more Ethernet cables to the HUB.

The computer needs to be connected to the Ethernet HUB to run FlowLab software program. This can be via an Ethernet cable or via Wi-fi using the router. The router is configured before shipment and it should not be necessary to change the settings.

The HotCoil, Polar Bear Plus and pumps have an Ethernet socket on the back panel. These should be connected to the Ethernet HUB using the Ethernet cables provided. The Ethernet HUB is then connected to its power supply. Once connected turn on the HotCoil and the Pumps and you should see the lights on the connected channel on the HUB flashing showing they are communicating.

When the HotCoil is being controlled remotely by the FlowLab program the red remote light on the front panel will be lit.

If a Polar Bear Plus is connected it has been factory configured for remote operation with the FlowLab software.

## 4 FlowLab Description of Parts

### 4.1 Bottle Tray

The bottle tray is manufactured from polypropylene and is designed to hold reagents and solvents contained in suitable containers. Bottles and caps are supplied with the system.

### 4.2 The Reagent Pumps

FlowLab has two high pressure pumps. These pumps have been modified internally to offer high chemical resistance. Permissible flow rates are 0.01 ml/min to 10 ml/min on each pump giving a total maximum flow rate of 20 ml/min. The high pressure pumps can be controlled manually from the pump control panel or using the FlowLab software. The pump shown has the optional 3-way reagent inlet valve. The latest pumps are also fitted with an independent BPR cartridge on the outlet.



To avoid compromising or damaging the pumps, **ensure** that reagent solutions are filtered and free of particulates.

The inlet filters provided should always be used to prevent micro particulate material entering the pump head. The pump can run up to 200 bar pressure.



### 4.3 Pump Priming and Pressure Transducers

High pressure pumps require priming with solvent before performing a reaction. This is easily achieved by attaching a syringe to the prime valves fitted to the left side of the pump pressure

transducer. Open the black valve and draw some liquid up into the syringe then close the valve. Set the pump flow rate at 1 ml/min on the front panel and check there is a stable pressure when pumping with an in-line back pressure regulator cartridge.

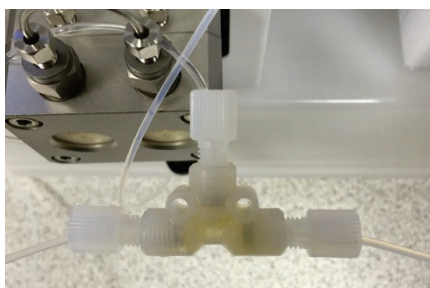


There is also a 'Prime' function in the FlowLab application. This will automatically run all the pumps at 1.0ml/min for up to 5 min and then stop.

#### 4.4 Mixing "T" and back pressure control

FlowLab comes complete with tubing and either a stainless steel or a PTFE mixer "T". Attach each of the pump outlets to one side of the mixer "T" with the tube supplied then the outlet of the mixer "T" to the coil inlet. Alternatively, connect the pump outlets directly to the glass static mixer (GSMs), if supplied.

Connect the coil outlet to the backpressure regulator supplied with the system noting the direction of the flow shown by the arrow. FlowLab is supplied with a 100psi pressure transducer. Other backpressure regulator cartridges of different pressure ratings can be obtained from Uniqsis (see section 8).



The system back pressure cartridge is held in a black PEEK or 316L SS housing. This can be clipped to the optional stand.

Back pressure can be varied by fitting different fixed back pressure regulator cartridges (40 – 100 bar cartridges are available). These are colour-coded as shown in the table below.

The backpressure regulator is a non-return valve that maintains a pressure in the system. The actual pressure achieved will depend on the flow rate and viscosity of the material, but should be close to the nominal set value of the cartridge used, and remain stable throughout an experiment.

*Note:* The maximum safe temperature and pressure rating for a column or coil reactor is determined by the material from which it is fabricated.

**PTFE/PFA maximum pressure 22 bar and maximum temperature 150°C**

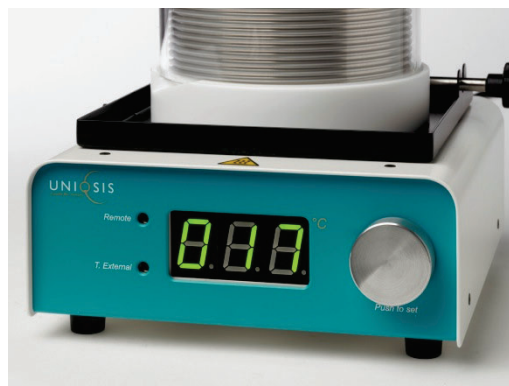
**Stainless steel and Hastelloy 200 bar and maximum temperature 260°C**

#### **4.4/1 Outlet Waste/Collect Valve (option)**

If the waste/ collect outlet valve option is included this is mounted on the stand as shown in the picture, this is useful to easily switch from the waste reservoir to the collect bottle as the product elutes from the reactor.



### 4.5.1 HotCoil™ Reactor station



The HotCoil is a compact heated reactor station compatible with all Asynt coil reactors from 2 mL to 60 mL. It can maintain temperatures from ambient temperature to 260 °C (300 °C optional upgrade is available).

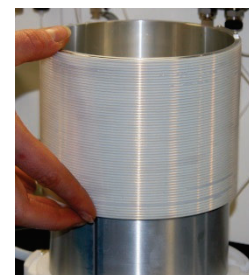
#### To manually set temperature

The temperature can be set on the front panel; a single press of the control knob (display turns red). Dial up the required temperature and press the knob again whereupon the display will turn green showing the actual temperature. Whilst the temperature is heating the green dots are visible moving from left to right. Once the target temperature has been reached within  $\pm 5^{\circ}\text{C}$  of the set point, all the dots flash simultaneously. Within  $\pm 2^{\circ}\text{C}$  of the set point just the temperature is displayed in green.

If a lower temperature is set the unit will cool down. In this instance the dots move from right to left until the target temperature is reached. The standard HotCoil has no active cooling so when running experiments, it is best to run the lowest temperature first and work upwards.

The coil reactor holder contains heating elements and a temperature sensor. It is used to heat the coil reactors. Coil reactors consist of tubing wound in a helical groove on an aluminium former (mandrel). Coil reactors are available in a range of pre-wound sizes and are easily fitted and removed from the heating module:

1. Ensure coil heater mechanism is closed by turning the knob anti-clockwise. This contracts the heater module so that coil reactors are released and can easily be removed or refitted.
2. Slide a coil reactor onto the heater ensuring the inlet and outlet tubing are located in the indent on the PTFE base and are not trapped (newer coil reactors have a coloured protective heat shrink tubing fitted over the PTFE tubing at this point to help prevent



damage to the PTFE tubing).

3. Turn the knob clockwise, this will expand the heater module to give good contact for heat transfer. (This is essential for good temperature control.)

The coil heater is calibrated (with the optional glass cover in place) so that the temperature in the heater is slightly higher than the set temperature.

This ensures the temperature of the reaction is as specified and compensates for radiative heat loss.



**To ensure the best heating performance, and safe operation of the unit, use the optional glass cover particularly at temperatures over 100 °C**



**Do not touch the coil reactor or glass cover when they are hot!**

#### 4.5.2 The Polar Bear Plus Reactor station

The Polar Bear Plus reactor station can be operated from -40 °C to 150 °C.

Fit the remote probe into the socket on the rear panel and place the temperature probe into the hole in the top plate. This will then use the remote probe to ensure the correct reaction temperature

The unit is pre-configured at the factory to work remotely with the FlowLab software. It has a fixed IP address set to 192.168.187.222. The Ethernet cable is plugged into the socket on the rear panel.

The Power on/off is located on the rear panel underneath the inlet socket. The unit will run a self-test when powered up, this may last up to a minute.

The switch on the lower left panel must be switched on for the cooling to function.

There is a large colour display showing the set temperature and the actual temperature.

The picture below shows the Polar Bear plus fitted with a 25ml coil reactor and an optional mixer/ reactor chip.

Please use the glass cover supplied to speed up cooling and heating. An inert gas purge can be employed to minimise condensation and help maintain visibility of the reactor(s). A gas inlet is provided in the optional adaptor ring.



There is a full operator manual on the USB memory stick supplied with the unit. The unit can be reconfigured for manual operation if so required.

## 4.6 Tubing

The system has been configured with the minimum dead volume. For information on tubing replacement – see Troubleshooting Section 7.

*Note:* If the 0.5 mm id perfluoropolymer tubing is fitted between the pump head outlets and the 'T'-mixer. This is able to withstand a pressure up to 60 bar and 160°C @ room temperature. PTFE and PFA coil reactors can be used up to 20 bar and 150°C.

Always use Stainless steel or Hastelloy for high temperature and high pressures up to 200 bar.

 **To ensure the best heating performance, and safe operation of the unit, use the glass cover particularly at temperatures over 100°C and below 5 °C**

 **Do not touch the coil reactor or glass cover when they are hot or cold as burns may occur!**

## 5 FlowLab™ Software

### 5.1 Overview

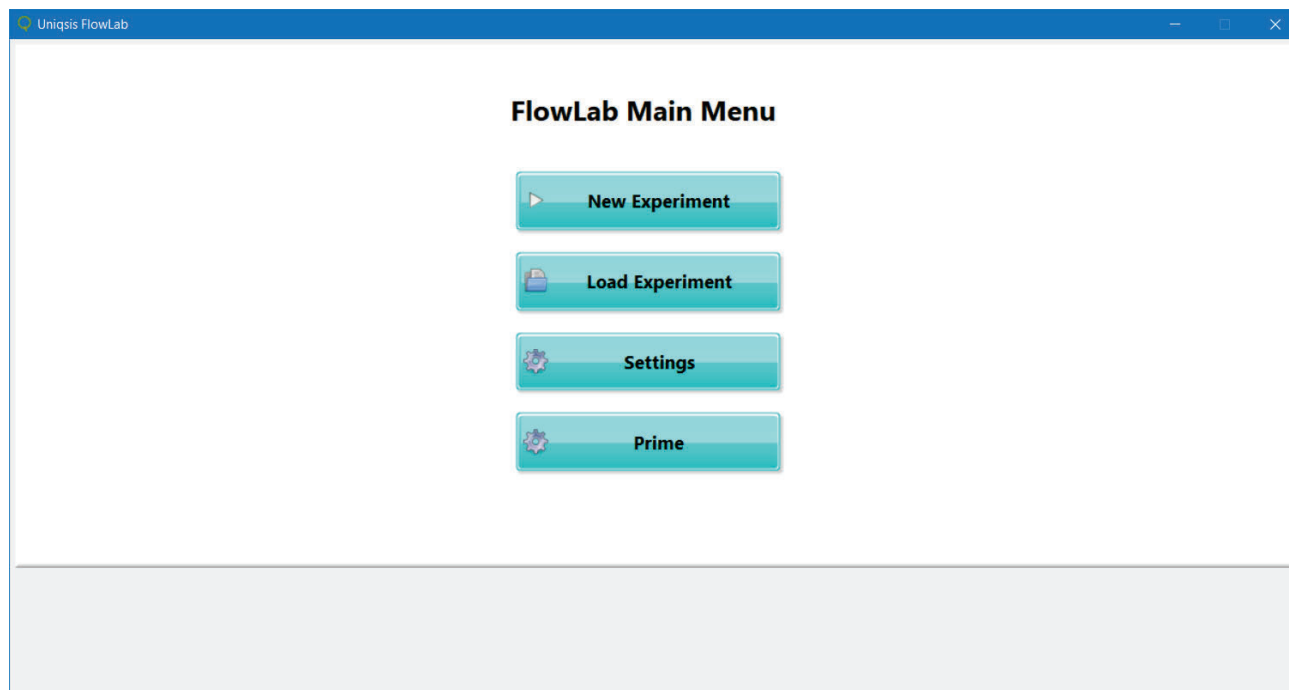
FlowLab is a system control software that is installed on the laptop supplied. Once connected via an Ethernet connector it can control the pumps and HotCoil. Experiment conditions can be saved including names of solvents and reagents. These can be loaded again later so an experiment can be repeated exactly.

The program benefits from an easy step through menu with prompts and safety information. The real time display shows the temperatures and pressures and the status of the experiment. The real time temperature and pressure data is saved with the experiment setup in a text file so that it can be viewed later using Excel.

**Please note:** All of the pumps and heater can be stopped immediately at any time by pressing the **Main Menu** button at the bottom LHS of any control screen.

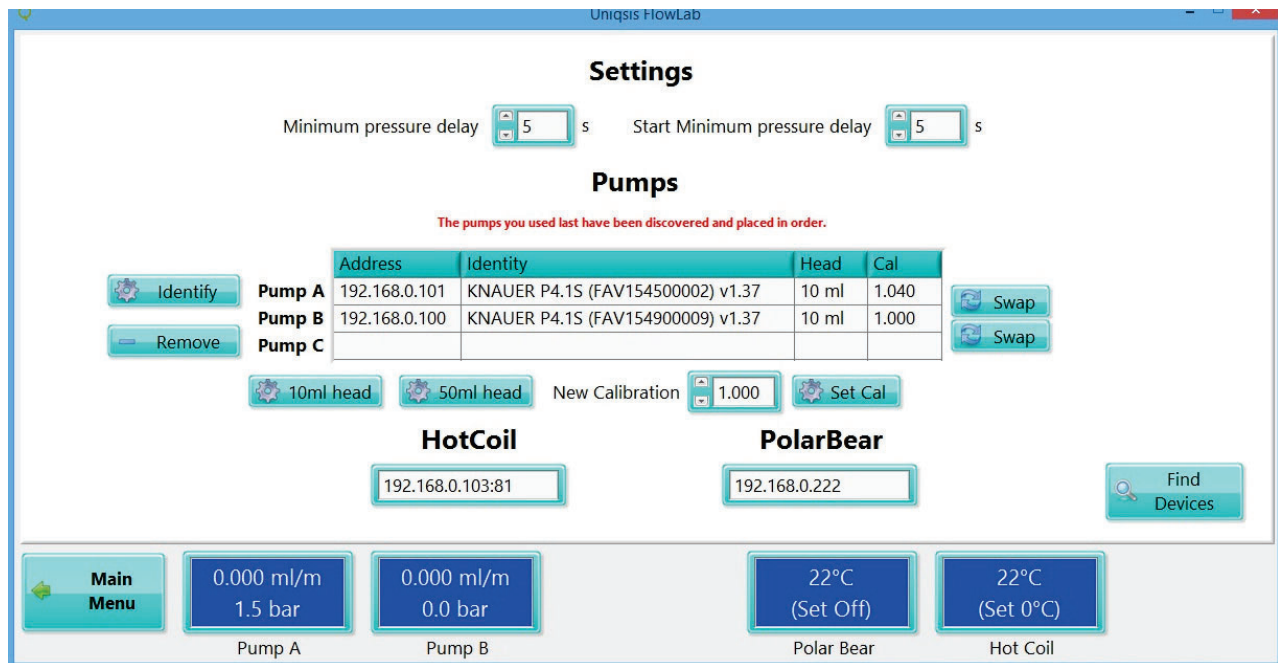
## 5.2 Setup

The icon to start FlowLab is located on the desktop. This will bring up a splash screen and the **Main Menu**.



Firstly, check that the program is communicating with the pumps and the HotCoil and or Polar Bear Plus. Go to the **Settings** page from the **Main Menu**.

If no units can be seen when using the Wi-Fi check to make sure the FlowLab is connected on the Network setting page on the computer.



The screen shot above shows a system with two pumps the HotCoil and the Polar Bear Plus.

The HotCoil IP address is setup on the unit before shipment so will not need changing.

Usually all connected devices are auto-detected and apart from changing the assignment as to which pump is A and which B (<Swap A/B>), no changes are typically necessary to the default system settings configuration. Use the “Find Device” to communicate with all items. Each box will turn blue to show it is connected and shows the current temperatures, flow rates and pressures. If the box turns red the unit is not connected on the network.

The IP address and serial numbers of the pumps are shown together with the HotCoil and Polar Bear Plus.

Press “find devices” to locate the items on the LAN connected to the Ethernet HUB

The pumps are normally configured for 10ml/min, where supplied. These can be changed to 50 ml/min pump heads by highlighting the required pump and pressing the 50ml head button.

The pumps can be swapped if required.

(If the FlowLab program can't communicate with any of the devices the box will turn red and it will report error 56 and then eventually the message “can't communicate”).

### 5.3 Safety

It is essential that the system is operated safely. When the program is running it continually monitors for leaks (low pressure) or blockages (high pressure).

Since small air bubbles may cause a transient pressure drop, from which the system will automatically recover, there is an option to set a **Minimum pressure delay** time i.e. the time whilst running an experiment after which the system will stop if a detected minimum pressure error is not corrected automatically.

Similarly, when the pumps are first started, particularly for large coil reactors, it can take a little while for the system pressure to build to the desired level. The pumps may interpret this as a low pressure error condition and stop. Therefore, there is the option to set a (Pump) **Start Minimum pressure delay** time, also.

Generally, these parameters should be set to 5 and 10 seconds respectively, and do not need to be routinely changed.

In contrast, high pressure monitoring is switched on all the time. When maximum pressure is reached the pump(s) will immediately shut down under any circumstances to avoid damage.

Minimum pressure delay  s      Start Minimum pressure delay  s

### 5.4 Pump calibration

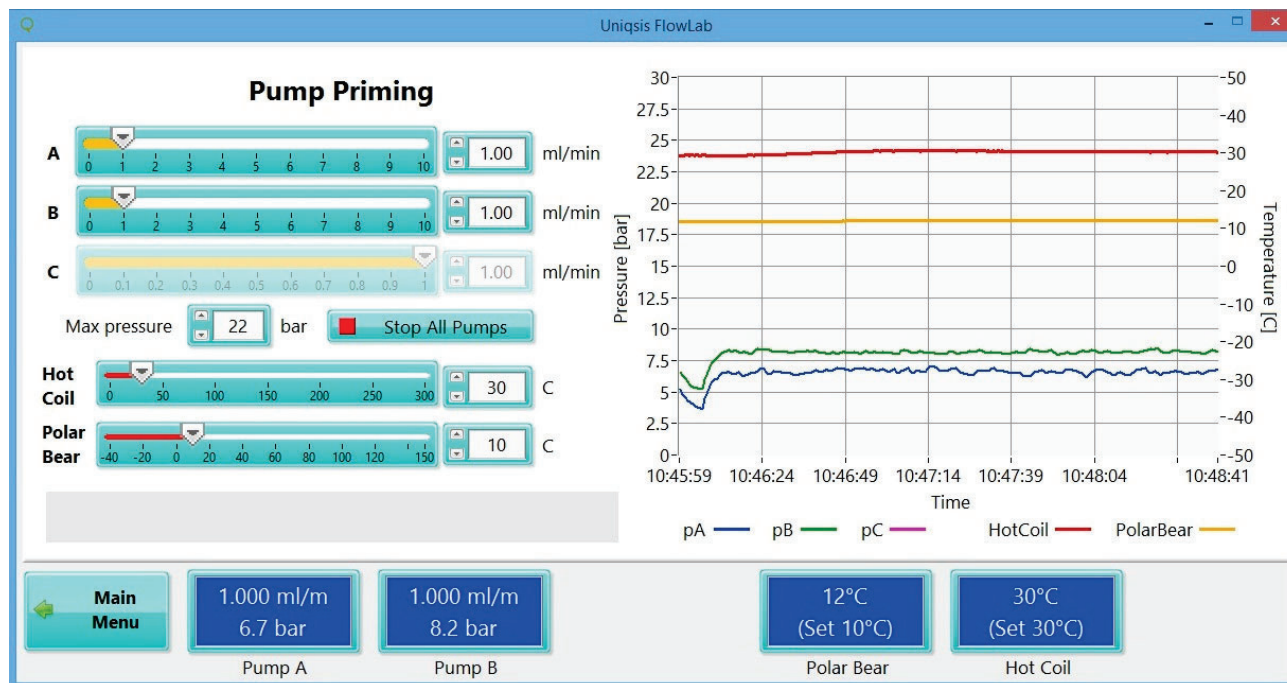
It is possible to calibrate the pumps for a given solvent and pressure to allow for the differences in viscosity, temperature and back pressure (compressibility).

To do this a calibration factor is entered for each pump.

Pump A Cal       Pump B Cal

**Tip:** set the flow rate at 1ml/min and collect for 10 minutes and measure the collected volume. Divide this by ten and enter this number into the Pump A or B Cal factor as appropriate.

## 5.5 Priming the pumps (equilibration)



Prime the pump heads as described in Section 4.3.

Set the flow rate of the pumps by using slider bar or by entering a value in the box. As soon as the flow rate is set the pump will start (an arrow will appear on the pump display).

Alternatively, press **Start Pumps**. Both pumps will start at a default flow rate of 1.0ml/min.

If a pump is not present it is greyed out (as shown above).

**Tip:** Set the flow rate at 1 ml/min and look at the real time graph pressure trace and check to make sure there is a stable pressure. If there are large oscillations, there may be air in the system. Stop and re-prime the pumps. During the priming and equilibration time the low pressure limit is switched off to allow time for air to be eliminated from the system.

The temperature of the HotCoil and the Polar Bear Plus can be set using the slider or by typing in the required temperature.

The real-time display auto scales. However, it is possible to manually change the Y-scale by clicking on the maximum value on the axis and setting the new desired value. (Applies to both the pressure and temperature axis). The X-axis uses the computer time real time clock. Values for the temperature can also be negative.

Set the temperature of the HotCoil and check it is correct and the same as indicated on the front panel display.

At any time both pumps can be started stopped simultaneously by pressing the **Stop Both Pumps** button. Once stopped, pumps can be restarted by pressing the **Start Both Pumps** button.



Check the display trace to make sure the pressures are stable. The oscillation should be no more than  $\pm 2$  bar, as shown in the screen shot.

The maximum pressure is also set for the prime sequence – this should be set to 22bar or less when using PTFE or PFA coil reactors. At least 100bar is safe for stainless steel coil reactors

During the prime process the maximum pressure is set to 22 bar (assuming PTFE/PFA coil reactors) this can be increased up to the maximum when using stainless steel or Hastelloy.

## 5.6 Experiment Setup

Press the **New Experiment** button from the **Main Menu** to continue and set up an experiment.

On the experiment screen it is possible to enter a description of the reagents used in the text fields.

**Experiment Setup**

Reaction name		Reactor Type	Reactor Size
TEST-001-1		COIL	25 ml
Reagent names		Flow rates	Pump delays
Reagent A	RGT A 0.8M	0.800	00:00:00
Reagent B	RGT B 1.0M	1.000	00:00:10
Reagent C	RGT C 1.2M	1.200	00:00:20
Temperature Control		Pressure Limits	
Hot Coil	Polar Bear	Equilibration	Total Expt Time
50 C	15 C	1.0 ml/m	00:05:00
		Max pressure	Min pressure
		45 bar	5 bar

Do not set maximum pressure greater than 22 bar for PTFE reactors

<b>Main Menu</b>	0.000 ml/m 21.6 bar	0.000 ml/m 21.0 bar	0.000 ml/m 23.0 bar	51°C (Set 0°C)	14°C (Set Off)	<b>Start Experiment</b>
	Pump A	Pump B	Pump C	Hot Coil	Polar Bear	

Reaction name (this is the file name used to save the experiment conditions)

Reagent A , B and C name description Reactor type and size

The HotCoil and or Polar Bear Plus temperatures and pump flow rates should be entered together with the total experiment time and any pump start delay time for each reagent channel; the (total) equilibration flow rate, minimum and maximum pressure for the experiment.

Set the flow rate to be used when the system is equilibrating together with total experiment time and maximum and minimum pressure.

**Tip** The total reaction time is the total experiment time (the total time the pumps are running) This must include the reaction time and any additional time to collect the products.

It may be necessary to set delay times on the pumps to ensure the reagents meet at the correct time, Uniqsis have 3 useful tools for this together with a back pressure and residence time calculation – see below.

## 5.7 Uniqsis tools for calculating flow rate, reaction times and temperature

There are some useful tools on the Uniqsis website for calculating the reaction time and pressure required for running a given solvent above its boiling point. [www.uniqsis.com](http://www.uniqsis.com)

**The Backpressure Calculator** estimates the system back pressure required to prevent a solvent boiling at any given temperature.

There is a pulldown list of most commonly used solvents.

**FlowSyn™ Back Pressure Calculator**    FlowSyn™ Reaction Time Calculator    FlowSyn™ Flow Rate Calculator    UNIQSIS

**Instructions:**

(i) Enter a known reaction temperature (Temperature 1) and time (Time 1)

**then either:**

(ii) Enter a new reaction temperature (Temperature 2) and click "Calculate" to give the corresponding new reaction time (Time 2)

**or**

(iii) Enter a new reaction time (Time 2) and click "Calculate" to give the required temperature (Temperature 2).

**Notes:**  
Calculations use the Arrhenius equation with a null frequency factor (A) and an Activation Energy ( $E_a$ ) of  $50\text{kJ mol}^{-1}$

**Known reaction temperature and time:**

Temperature 1: ? °C    Time 1: 00 : 00 : 00 : 00 (days) (hr) (min) (sec)

**Unknown reaction temperature or time:**

Temperature 2: ? °C    Time 2: 00 : 00 : 00 : 00 (days) (hr) (min) (sec)

Calculate    Clear

## Reaction Run Time Calculator

A second tool helps calculate the reaction time by converting a batch reaction time at a given temperature to a flow reaction at a different temperature.

FlowSyn™ Back Pressure Calculator    FlowSyn™ Reaction Time Calculator    FlowSyn™ Flow Rate Calculator    UNIQSIS

**Instructions:**  
This calculator that allows you to find the minimum pressure required to keep the solvent liquid when using our FlowSyn™ unit.  
Simply choose your solvent and enter your desired working temperature. If your solvent is not listed choose 'Other' and enter in the entropy of vaporization and known boiling point.

**Notes:**  
\* Exact entropy of vaporization unknown  
\*\* This is a suggested value based upon an estimated calculation of the entropy of vaporization using the boiling point of the solvent at atmospheric pressure (Trouton-Hildebrand-Everett rule) and is intended to be used as a guide only.

Solvent: Other    Entropy of vaporization (JK<sup>-1</sup>mol<sup>-1</sup>): 87

Boiling point at atmospheric pressure: ? °C

Desired working temperature: ? °C

Minimum BPR pressure: [ ]

Unit of pressure:  psi     bar

## Flow Rate Calculator

Another tool can be used for calculating the reaction time, flow rates and delay times to ensure reagent mixing is synchronised.

FlowSyn™ Back Pressure Calculator    FlowSyn™ Reaction Time Calculator    FlowSyn™ Flow Rate Calculator    UNIQSIS

**Configuration:**  
Reactor Vol. 1: 10 ml  
Reactor Vol. 2: ? ml  
Connection Vol. A: 0.1 ml  
Connection Vol. B: 0.1 ml  
Connection Vol. C: ? ml  
Connection Vol. D: ? ml

**Chemistry:**  
Molarity (M):    Stoichiometry:  
A: 1.0    1  
B: 0.67    1.15  
C: ?    1  
D: ?    1  
Res. Time 1: 00 : 18 : 00 (hr) (min) (sec)  
Res. Time 2: 00 : 00 : 00 (hr) (min) (sec)

A: 0.205 ml/min    B: 0.351 ml/min    C: 0 ml/min    D: 0 ml/min  
A: 1 ml    B: 1.716 ml    C: ? ml    D: ? ml

Delay A: 00 : 00 : 00    Delay B: 00 : 00 : 12  
Delay C: 00 : 00 : 00    Delay D: 00 : 00 : 00

For the FlowLab system enter the reactor volume, and put in a connection volume of 0.1 ml (standard configuration).

Values are conveniently calculated taking account of molarities and the desired reaction stoichiometry.

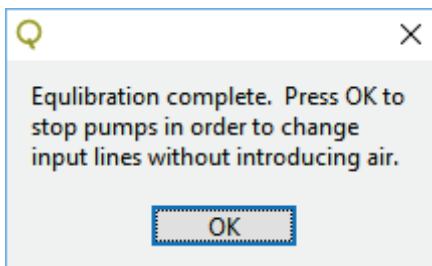
Enter these values including the flow rates and delay times into the FlowLab experiment setup page.

## 5.8 Running the experiment

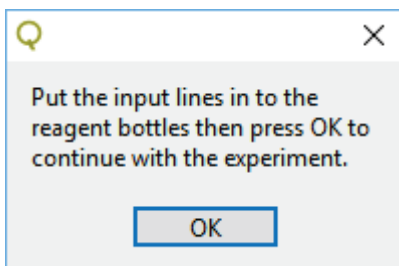
Once all settings have been entered, the experiment can be started. The first stage is the equilibration, when the temperature is within 2 degrees on the reactors the experiment is ready to star



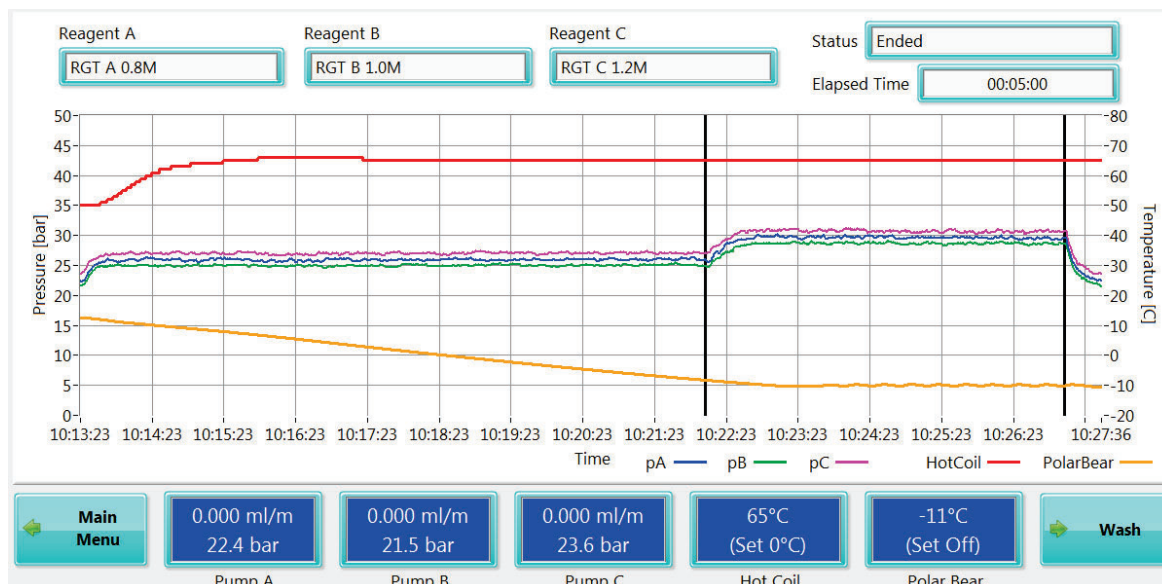
At the end of the equilibration period a message will pop up and the pumps will pause so the lines can be changed from the solvent bottle to the reagents without introducing air bubbles. The pumps are stopped during this process.



Once this is completed press OK for the experiment to start.



The experiment then starts and the experiment status page is shown. Vertical black lines indicate the beginning and end of an experiment.

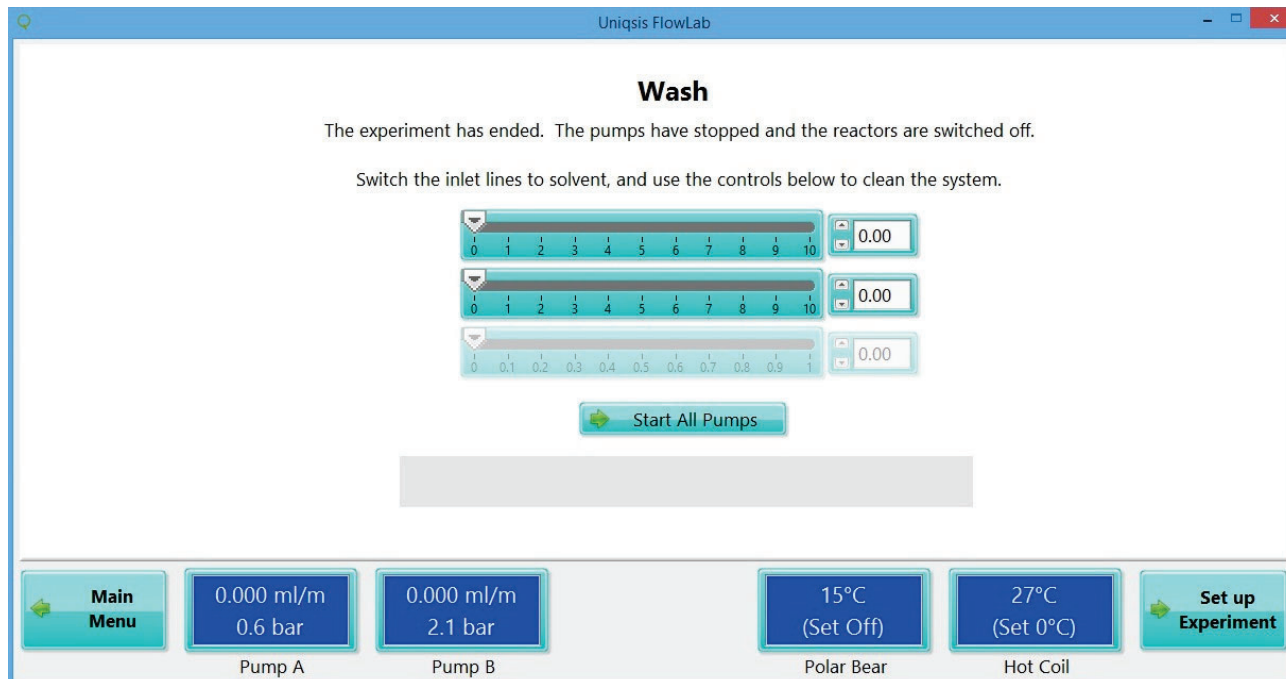


The experiment will then progress, the x axis shows the real time taken from the computer time clock.

The experiment can be aborted at any time by returning to the **Main Menu** or pressing the **Abort** button at the base of the display.

At the end of the experiment the pumps stop and the heaters/refrigeration switch off on the HotCoil/ Polar Bear Plus.

The system can then be washed by pressing the **Wash** button



The solvents should be switched back to the solvents and the pumps started to flush out the system. We would recommend at least 2 system volumes to completely clean the system.

## 6 Program save and load

### 6.1 Loading a previously stored experiment.

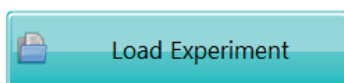
Experiment settings are automatically saved once an experiment is started.

The reaction name is used followed by the start date and time.

The experiment files are located on the Desktop file: 'FlowLab Expt Data'

An experiment can be reloaded using the **Load Experiment** button in the **Main Menu**.

Please note: pressure limits/settings are hardware sensitive and are archived in the experiment log file for completeness, these are reloaded during "Load Experiment".



## 6.2 Viewing the logged data stored

The logged data is stored as a Tab Separated file in the FlowLab Expt Data file on the desktop.

The data can be imported into Microsoft Excel or similar spreadsheet. The data has the experiment setup and the logged data can be manipulated and plotted with Excel or similar spreadsheet.

## 7 Troubleshooting and FAQs

### Using manual control the pump stops after a short time

Check the minimum and maximum pressure on the configuration page. Also, if the tubing reactor is empty – it will take some time to fill and begin to pressurise significantly.

### The pressure will not rise, there is no solvent coming out of the outlet

There could be an airlock in the pump. Use the pump priming valve to *slowly* pull 5ml of clean solvent through each pump.

If a leakage is observed from around a fitting it is possible that the thread has been damaged. In this case, a temporary repair can often be affected by wrapping the thread of the PEEK tubing nut with Teflon sealing tape and then refitting.

### A piece of tubing has popped out under pressure

Cut the end from the tubing as it will have deformed before reattaching. Typically, a new ferrule will have to be used because these deform slightly on use and may be difficult to refit. The stainless steel retaining ring can be re-used. It is important to 'pre-swage' super flangeless fittings onto the tubing (using another 1/4-28 fitting) before fitting to the FlowLab.

### The pressure is too high

Check for blockages. Blockages usually occur close to fittings i.e. typically at the inlet (or outlet) of a coil reactor or the heat exchanger tubing. Reversing the coil reactor and switching the pumps on will usually clear the blockage.

### When I try to pull solvent through the HPLC pump, nothing comes out?

Please ensure that the inlet tubes are below the liquid level in the stock bottles. Alternatively, there may be a blockage in the HPLC pump. Each pump head contains 2 check valves (non-return valves). Disconnect the inlet (bottom right of pump head) and take out the check valve.

Place it in a small vial with solvent (e.g. MeOH) and sonicate the vial for 5 minutes. Replace the check valve and try again. If this does not remedy the problem, there is another check valve in the

top right of the HPLC pump. Remove this check valve and sonicate in the same way. Uniqsis can supply a simple check valve cleaning tool which greatly facilitates the cleaning of CVs.

### **Under Manual Control, when I start the pumps the system runs for a while and then stops**

Check the minimum pressure setting. If this value is not exceeded after the pumps have been running for the ‘Delay’ period, the pumps will stop. This can happen if there is a significant amount of air in the reactors, for example when priming. Either increase the ‘Delay’, reduce the ‘Minimum Pressure’, or just persist in starting the pumps a few times until enough back pressure is present in the system to prevent the pumps from stopping.

### **The boxes are displayed in red and show Error 56 or “Can’t communicate”**

Check to make sure the units are switched on and the Ethernet cables are connected.  
 If using the Wi-Fi check the computer is seeing the FlowLab in the network setting on the computer  
 Reboot the router  
 Turn off the Firewall to see if this is preventing the communication on the network

## **8 Options and accessories**

<b>Pumps</b>	
UQ1028	10ml/min pump with pressure transducer
UQ9002	50ml/min pumps heads for 20P HPLC pumps
<b>Collect/waste valve</b>	
UQ1027-1	Outlet waste/collect switch valve (requires stand for mounting)
Inlet inerting adapters	
<b>Reactors</b>	
UQ-Inert	Inerting adapters for the luer bottle caps
	Over 40 reactors to choose from 1ml to 60 ml
UQ3005	HT coil reactor kit (2ml, 14ml Teflon HT and 2.5, 5ml, 20ml Stainless)
UQ3003	Stainless steel coil reactor set (2.5ml, 5ml, 10ml, 20ml)
UQ-1035-2	HotColumn with 2 column modules for 15mm OD columns (not included). (Shown below)
<b>Cover</b>	
UQ4001	Glass cover for HotCoil (150 x 135 mm)
<b>BPR's</b>	
U-469	Back pressure holder stainless
P-762U	Back Pressure Regulator Cartridge (5 bar blue)
P-763U	Back Pressure Regulator Cartridge (10 bar red)
P-766U	Back Pressure Regulator Cartridge (20 bar white)
P-765U	Back Pressure Regulator Cartridge (30 bar green)
P-795U	Back Pressure Regulator Cartridge (50 bar black)
<b>Mixer Reactor Chip</b>	
UQ5102	Glass Static Mixer/ Reactor Chip 2 ml volume
<b>In-Line UV-VIS</b>	
UQ1100	FlowWave UV/VIS spectrophotometer + Fibre optic cables

UQ1102	0.5 mm path length flow cell



HotColumn™ accessory

We also offer an inerting adapter that connects to the luer bottle caps together with a blanking plug as shown in the picture below.



## 9 Specifications

### 9.1 FlowLab Specification for a 2 pump single reactor system

Width x Depth x Height (mm) system	600 x 280 x 300
------------------------------------	-----------------

Weight (Kg)	32
HPLC pump minimum flow rate	50 µl/min
HPLC pump maximum flow rate	10 ml/min
Resolution	5 µl/min
HotCoil temperature range	Ambient to 260°C Optional upgrade 300°C
HotCoil Coil heat up time 25 to 100°C 25 to 200°C	12 min 17 min
Over temperature cut out	275°C
HotCoil Display resolution °C	1°C
Setting resolution °C	1°C
Minimum Setting Temperature °C	25°C
Polar Bear temperature range	-40°C to 150°C
Polar Bear cool down time (coil reactor) 25 to 0 0 to -20°C -20 to -40°C	10 mins 18 mins 20 mins
Fixed back pressure regulator	100psi supplied, up to 100 bar optional

## 9.2 Power Specification System Running 2 pumps one reactor

### Supply Specifications

	Voltage	Frequency	Power
Uniqsis	230V	50Hz	1000W
	120V	60Hz	1000W

## 9.3 Conditions of use

Temperature	5 to 40°C
Maximum relative humidity	80 % r.h. in room temperatures up to 31°C decreasing linearly to 50 % r.h. at 40°C
Altitude	Up to 6,500 feet (2,000m) above sea level

## 10 Warranty

The FlowLab is covered by a warranty for 12 months from the date of delivery.

### The warranty does not include:

- Damage caused by incompatible solvents or substrates
- Blockages caused by precipitation in the system (see user maintenance)
- Use outside of the parameters of the conditions of use (see conditions of use)

### Warranty includes:

- Protection against faulty materials or workmanship
- Labour for a qualified Uniqsis approved engineer to repair the system
- Shipment costs if unit is required to go back to base for repair

## 11 Service and support

If the fuses blow during operation, a competent person should check whether the unit has developed a fault needing rectification before the fuses are replaced. To replace the mains fuses, remove the mains lead from the unit, release the fuse drawer catches using a small screwdriver to extract the drawer. Replace the fuses with the correct type and rating: *Note:* if a main fuse blows, it is likely to be indicative of a problem with the unit that simply replacing the fuse will not resolve.

For service and support please contact Asynt via the details below.

