Manual for the MicroSIA Instrument

Version 1.1

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Table of Contents

1 Introduction

1.1 General

The MicroSIA system is designed to be a compact, reliable instrument for automated assays as well as for R&D projects, based on the Sequential Injection Analysis (SIA) concept.

This manual will provide simple instructions on how to configure and run a MicroSIA unit. The manual will cover the basic operation of the MicroSIA system and usage of the SIAsoft control software. For a more detailed discussion regarding the science of SIA, please contact FIAlab. For additional information on the SIAsoft, please review the separate SIAsoft user's manual.

Note: On the computer that accompanied your instrument, the software and software drivers have already been installed prior to shipping. There is no need to install SIAsoft. All proper software settings have already been made. Installation instructions are provided in the SIAsoft manual for cases where software needs to be reinstalled (e.g. in connection with a computer update).

The computer regional settings should be set up such that numerical values use periods (".", as used in the USA) instead of commas for the decimal location. For example, 10 ½ should be written as 10.5, not 10,5. Make sure that this convention is utilized, and is also set up this way in the computer's Regional Settings.

1.2 Operation principle

The analyzer is based on the concept of Sequential Injection Analysis (SIA). A standard SIA instrument (see Figure 1) consists of a syringe pump and a stream selector valve, working in concert to draw in sample and reagent solutions and mixing them to carry out chemical derivatization of the sample. If necessary, additional modules (heaters, detectors, sorbent cartridges) can be included to provide further possibilities for processing the sample.

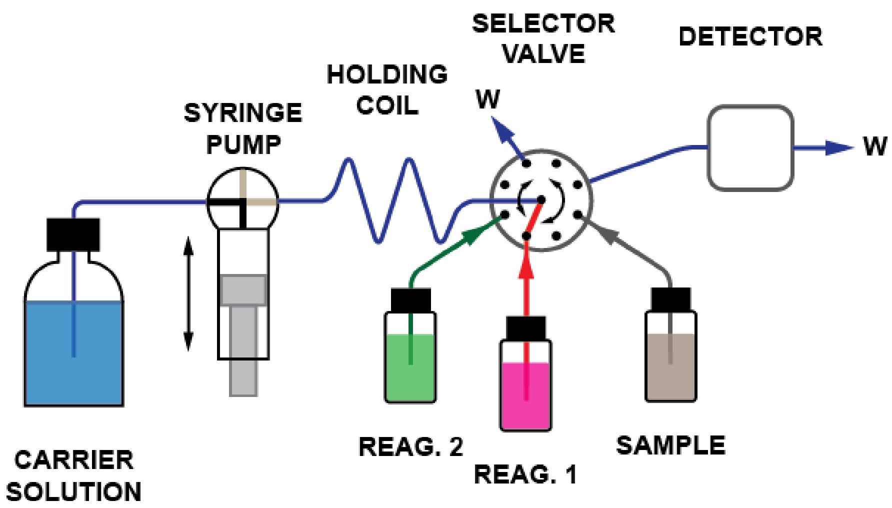


Figure 1 A schematic view of a Sequential Injection Analysis instrument.

2 Physical System Setup

This section of the manual shows the basic system setup.

The MicroSIA system is most commonly used for spectrophotometric assays. This section of the manual shows the basic system setup for this type of analysis.

Upon unpacking your FIAlab system configured for absorbance measurements you should find:

-(1) Basic MicroSIA unit
-(1) Desktop Computer pre-configured with SIAsoft
-(1) Autosampler (optional)
-(1) a UV/VIS or VIS/NIR Spectrometer (optional)
-(1) Tungsten or Deuterium-tungsten or LED light source (optional)
-(1) Flow cell (optional)
-(2) SMA terminated fiber optic cables (optional)
-(1) Flow-through heater-FT (optional)
-(1) Fluidic accessories kit

Installation requirements:
- bench space: 2-4 ft (0.6-1.2 meters), depending on accessories
- power outlets: 3 minimum (analyzer, computer, monitor)
- special power requirements: none

2.1 *Setting up the analyzer*

To set up the MicroSIA unit:

1) Make sure the switch on the back of the MicroSIA is in the OFF position.
2) Using the supplied USB cable, connect the MicroSIA's USB port to the computer's USB port.
3) Plug the 24 VDC power supply into the MicroSIA.

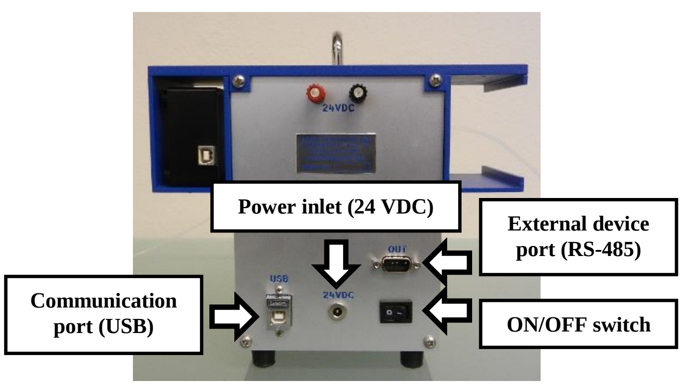


Figure 2 MicroSIA back panel.

Fluidic connections on the instrument are made using 1/16" (1.6 mm) or 1/8" (3.2 mm) OD FEP/PFA tubing, nuts and flangeless ferrules. To change tubing:

1) Remove the used or unwanted tubing by unscrewing the appropriate fittings on the instrument.

2) Slide a nut onto one end of the new tubing and follow with a flangeless ferrule.
3) Screw the tube into the appropriate port(s), pushing the tubing in as far as it will comfortably go. The nut should be turned so that it is snug finger-tight.
4) Gently tug on the tubing once screwed in to make sure it is secure.

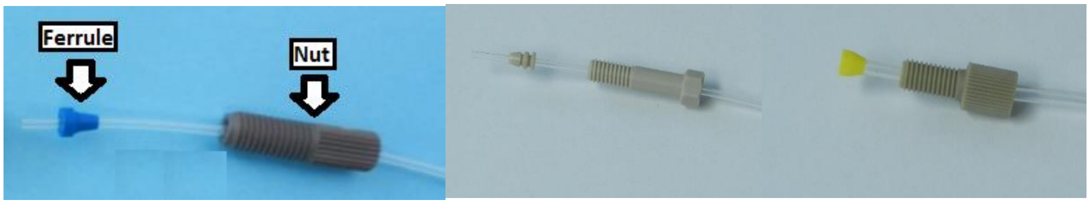


Figure 3 Connector assembly for FEP/PFA tubing. Left: 1/16" tubing for flat-bottom ports. Center: 1/16" tubing for conical ports. Right: 1/8" tubing for flat-bottom ports.

2.2 Instrument components

The MicroSIA has two main components: a syringe pump and a stream selector valve (see Figure 4). The pump and the valve are connected by the holding coil, which usually is 0.03 in (0.75 mm) i.d. tubing wound around a solid rod. The holding coil serves as a mixing vessel and also ensures that solutions drawn from the valve will not break through to the syringe.

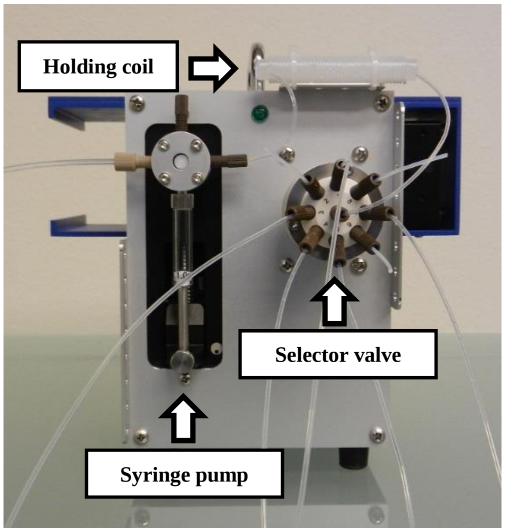


Figure 4 MicroSIA front panel.

2.2.1 Syringe pump

The syringe pump has three ports, any of which can be connected to the syringe by the action of the head valve. The left port is used as an inlet for carrier solution and should be connected to the appropriate buffer container. The connection is typically made with 1/8 in (3.2 mm) OD tubing as pump fill operations generally are carried out at a high flow rate. The right port is used as an outlet for carrier solution, and typically uses 1/16 in (1.6 mm) OD tubing. The top port may be used as an inlet, as an outlet, or left unused (plugged with solid tubing).

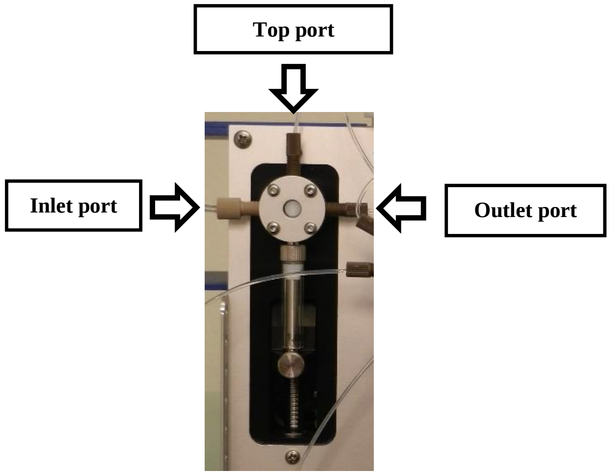


Figure 5 Syringe pump.

2.2.2 Stream selector valve

The selector valve operates by making a connection between the central port and one of the peripheral ports. The center port is always connected to the syringe pump via the holding coil, while the peripheral ports are connected to reagent containers, the sample source or to various sample processing modules (heater, detector etc.). The connections are shown in Figure 4.

2.2.3 Relay terminals

The relay terminals on the back panel can be commanded to put out 24 VDC. An internal resistor limits current through the terminals to max 4 mA. For that reason, the terminals can be used only for low-current applications such as driving an LED. The current is not enough to directly drive a solenoid valve or other high-current devices.

2.3 Accessories

2.3.1 Absorbance detector

For absorbance detection, a light source, a spectrometer, a flow cell and a pair of fiber optic cables are required.

To install an SMA-Z flow cell and fiber optic cables onto a MicroSIA analyzer:

1) Use a size #8 screw and nut to attach the flow cell to the side bracket of the MicroSIA.
2) Use one of the fiber optic cables to connect the light source to the flow cell.
3) Use the other fiber optic cable to connect the flow cell to the spectrometer.

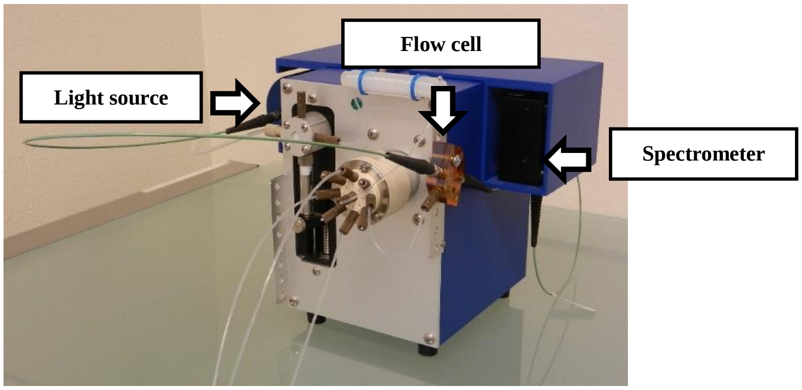


Figure 6 Connections between the light source, flow cell and spectrometer.

2.3.2 Heater

The most commonly used heater with the MicroSIA is the FT-Heater shown in Figure 7. It can be installed in place of the holding coil, or it can be placed between the detector flow cell and the stream selector valve.

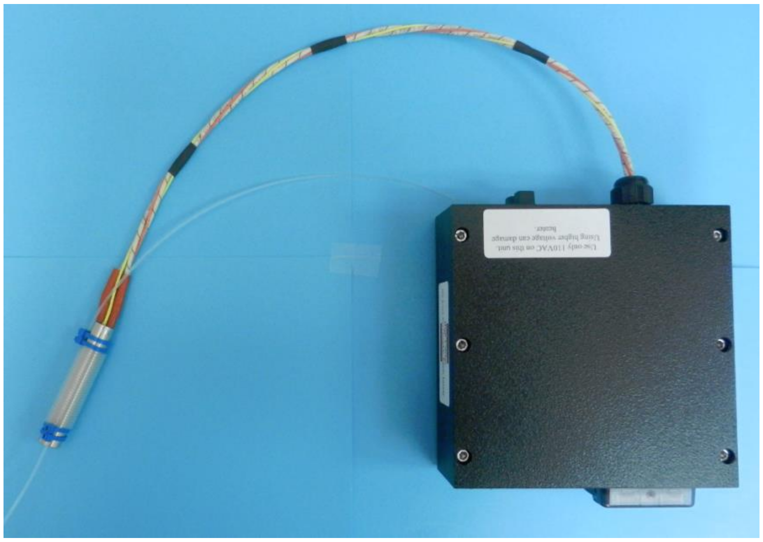


Figure 7 FIAlab Instruments FT-Heater.

To control the FT-Heater:

1) Turn on the heater via the switch in the back.
2) Set the heater to the appropriate temperature (°C) by using the arrow keys on the right side of the display. The green number represents the set-point temperature; the red number, the actual temperature of the heater's rod. Start the assay once the red number has stabilized; when both the red and green numbers are the same.

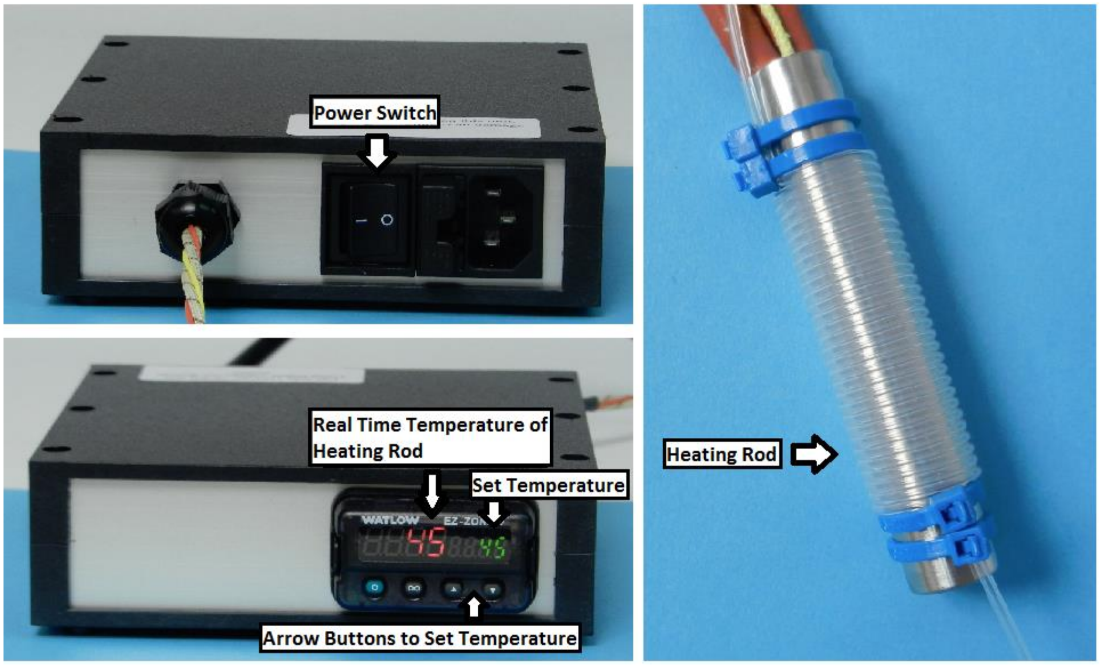


Figure 8 Controlling the FT-Heater.

2.3.3 ADU208 relay I/O module

The ADU208 relay I/O module can be used to control a set of relay ports (relay OUT) or to read the status of relay ports / TTL channels on external devices (digital IN). One ADU208 unit has a total of 8 relay OUT ports and a total of 8 digital IN ports.

The ADU208 is connected to a USB port on the control computer. Upon successful connection, the indicator LED on the ADU will light green. Please see the SIAsoft operation manual for detailed information about configuring and controlling the ADU208.

A relay I/O module can be used for a variety of different purposes. Some of the most frequent uses include:

- Sending and receiving PLC communication signals. Sending would be done via relay OUT ports, receiving via digital IN ports.
- Actuating solenoid valves. This is done via relay OUT ports, and requires a 12 or 24 V DC voltage source to be wired through that port.

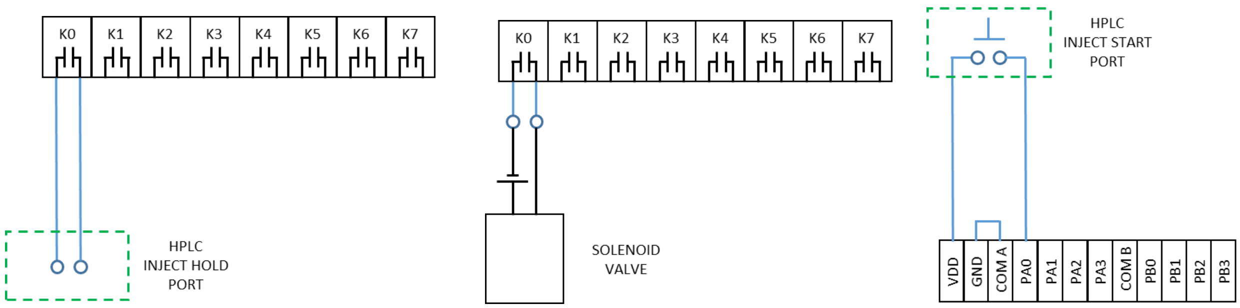


Figure 9 Examples of using ADU ports. Left: a relay OUT port connected to an Inject Hold port on an HPLC instrument. Middle: a relay OUT port connected to a power supply and a solenoid valve. Right: a relay IN port connected to an Inject Start port on an HPLC instrument.

2.3.4 External pumps

The MicroSIA setup can be expanded to include external syringe pumps. To use an external pump, it should be connected to the OUT port on the analyzer back panel by a cable (FIAlab P/N 43129). The cable provides both communication from the OUT port to the pump and power from an external 24 VDC power supply. The RS-485 address on the pump should be set to "B".

If multiple external pumps are desired, a special multi-drop power/communication cable is required (contact FIAlab for details). The RS-485 addresses should be set to "B", "C", "D" etc. The maximum number of external syringe pumps is three.

Software configuration of external syringe pumps is shown in section 3.1.1.

2.3.5 External valves

The MicroSIA setup can be expanded to include external selector or injection valves. To use an external valve, a connection should be made between the OUT port on the analyzer back panel and the "Serial A" port on the valve. The connection cable is FIAlab P/N 54111. In addition, power from an external 24 VDC power supply has to be connected to the back of the valve. The valve should be set to RS-485 mode, and the RS-485 address should be set to "4".

If multiple external valves are desired, a connector cable (FIAlab P/N 54112) is required to connect the "Serial B" port of the first valve to the "Serial A" port of the second valve (i.e. daisy chained). The RS-485 addresses should be set to "5", "6", "7" etc. The maximum number of external valves is three.

Software configuration of external valves is shown in section 3.1.2.

2.3.6 Combination of external pumps and valves

In cases where external devices include both pumps and valves, connections should be made as follows:

- The first valve is connected to the OUT port by cable P/N 54111.
- Any additional valves are connected to preceding valves by connector cables P/N 54112.
- Another cable P/N 54111 is used to connect the "Serial B" port on the last valve to the power/comm cable (P/N 43129) for the syringe pump. A DB-9 male-to-male gender changer is required to mate the two cables.

Figure 10 shows a schematic view of how the connections are made.

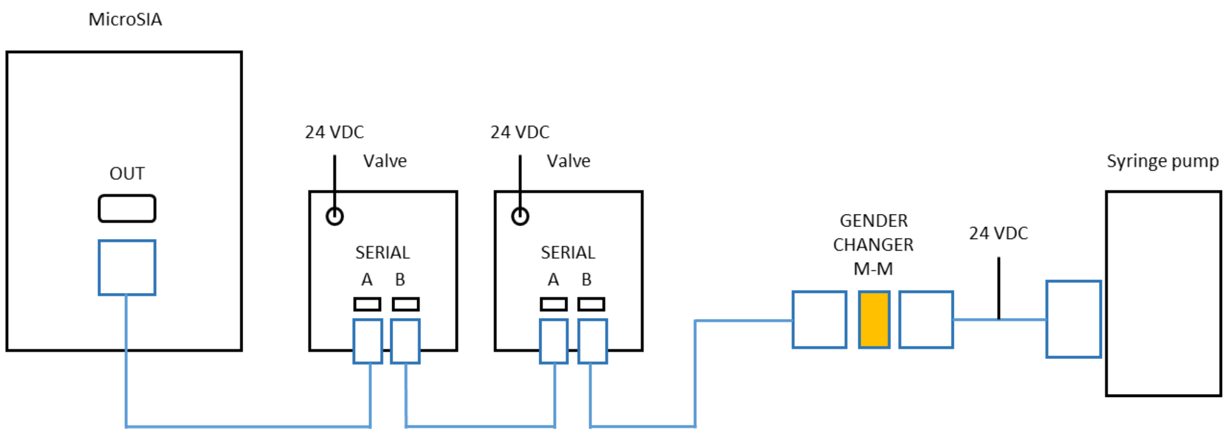


Figure 10 Connecting external pumps and valves to the RS-485 out port on a MicroSIA analyzer.

3 Control Software

The analyzer is controlled by the SIAsoft software. Refer to the SIAsoft manual for the software organization and features. The general layout of the software interface is shown in Figure 11.

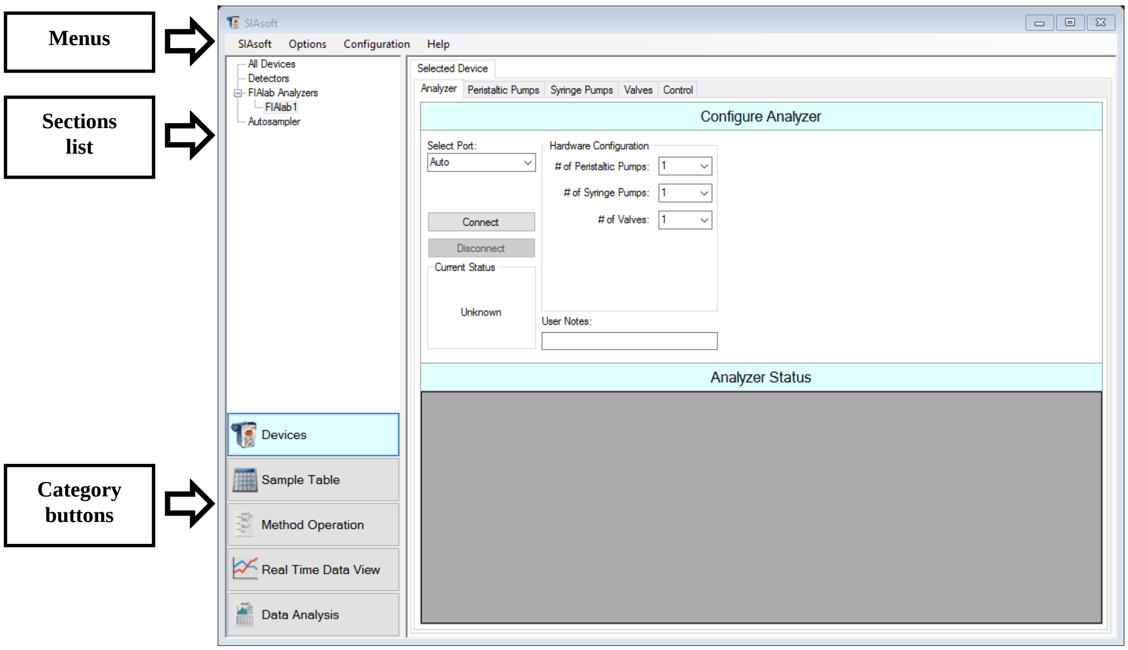


Figure 11 SIAsoft control interface.

3.1.1 Configuring external syringe pumps

When an external syringe pump is connected to a MicroSIA, the number of syringe pumps has to be increased from one to two. Furthermore, the external pump has to be set to use RS-485 address "B" (see Figure 12).

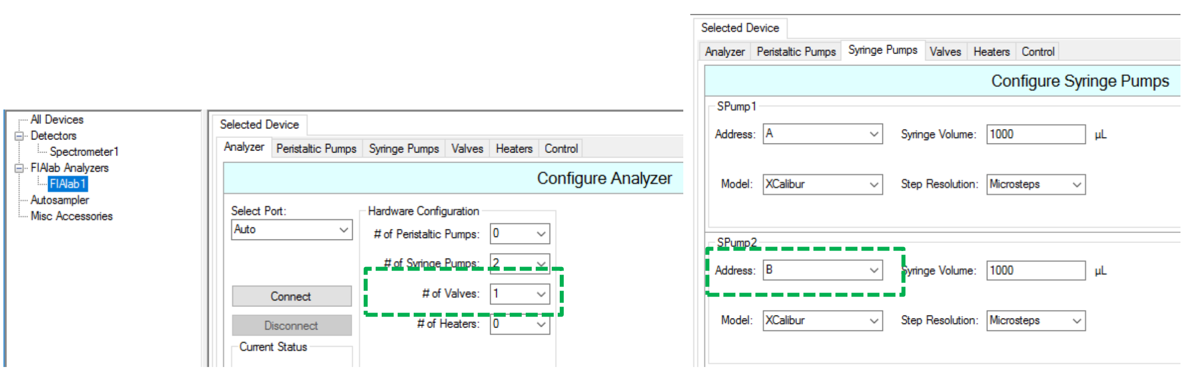


Figure 12 Configuring SIAsoft for an external pump: adjusting the number of pumps and setting the RS-485 address.

3.1.2 Configuring external valves

When an external valve is connected to a MicroSIA, the number of valves has to be increased from one to two. Furthermore, the external valve has to be set to use RS-485 address "4" (see Figure 13).

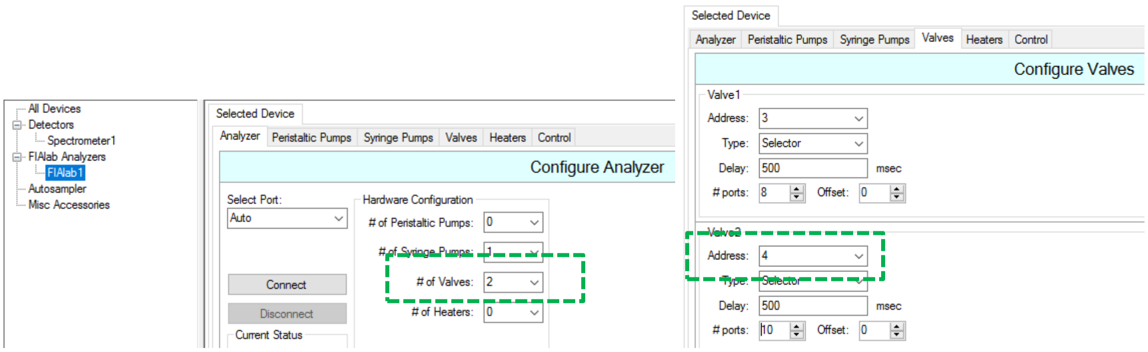


Figure 13 Configuring SIAsoft for an external valve: adjusting the number of valves and setting the RS-485 address.

4 Example Experiment

This section outlines an example experiment that can be used for becoming familiar with the MicroSIA. The experiment involves injection and detection of a sample solution containing nitrate. It requires a UV detector including

- a deuterium light source
- a pair of UV-VIS fiber optic cables
- a SMA-Z flow cell with 10 mm light path
- a UV-VIS spectrometer

The sample is an aqueous solution of 30 mg NO_3^-/L, most conveniently prepared from sodium nitrate. Water is used to mimic reagent 1.

Figure 14 shows a schematic view of the experiment setup.

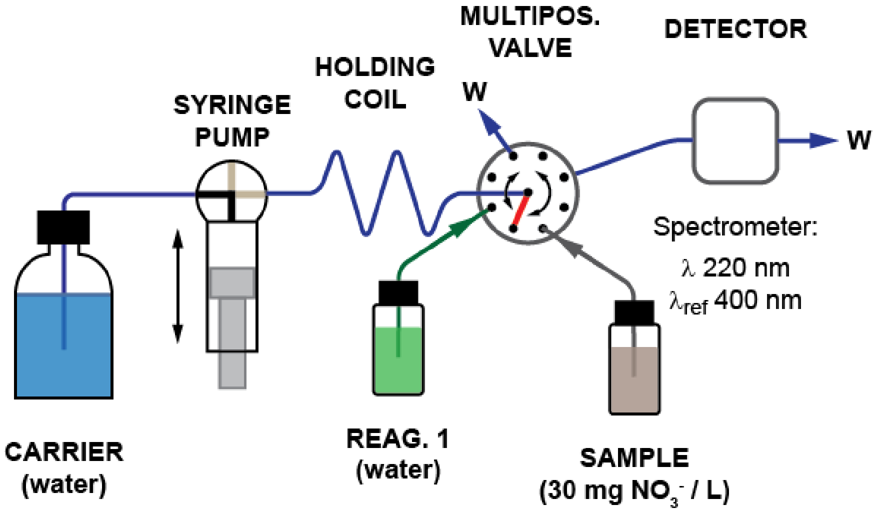
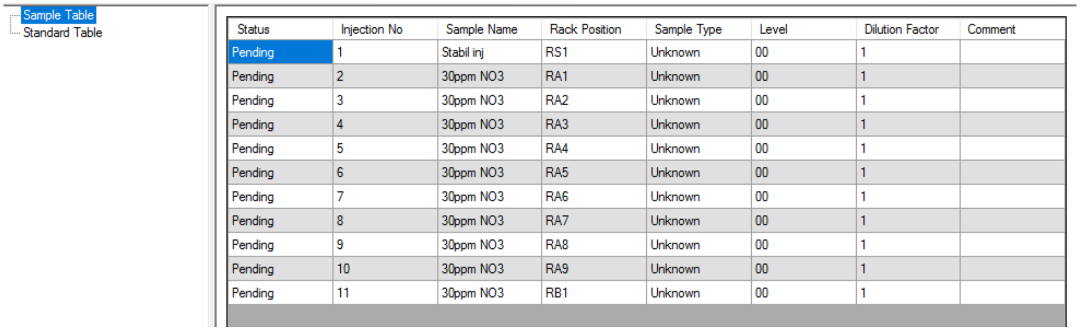


Figure 14 Schematic setup for example experiment. A vial of water is used as reagent 1 and connected to port 3 on the selector valve. A vial of 30 mg NO_3^- / L is used as sample and connected to port 5 on the selector valve.

After connecting the carrier, reagent and sample solutions, load the appropriate instrument configuration. You should find one on your computer's desktop. If not, contact FIAlab for assistance. The configuration sets the primary detection wavelength to 220 nm and the reference wavelength to 400 nm.

With all the solutions connected, the sample and reagent lines should be primed and the flow cell should be flushed with carrier, making sure that any air bubbles are expelled. The spectrometer intensity counts should be checked and integration time adjusted if necessary.

The next step is to create a Sample Table with multiple entries for 30 mg/L nitrate injections. An example is shown in Figure 15.



Sample Table
Standard Table

Status	Injection No	Sample Name	Rack Position	Sample Type	Level	Dilution Factor	Comment
Pending	1	Stabil inj	RS1	Unknown	00	1	
Pending	2	30ppm NO3	RA1	Unknown	00	1	
Pending	3	30ppm NO3	RA2	Unknown	00	1	
Pending	4	30ppm NO3	RA3	Unknown	00	1	
Pending	5	30ppm NO3	RA4	Unknown	00	1	
Pending	6	30ppm NO3	RA5	Unknown	00	1	
Pending	7	30ppm NO3	RA6	Unknown	00	1	
Pending	8	30ppm NO3	RA7	Unknown	00	1	
Pending	9	30ppm NO3	RA8	Unknown	00	1	
Pending	10	30ppm NO3	RA9	Unknown	00	1	
Pending	11	30ppm NO3	RB1	Unknown	00	1	

Figure 15 A Sample Table for repeated injections of 30 mg / L nitrate.

Next, an instrument method is loaded. Just like with the configuration, you should have one on your computer's desktop. The method draws in 20 µL of sample (nitrate solution), followed by 250 µL of reagent 1 (water). The sample and reagent are subsequently delivered to the detector for absorbance measurement. An example method (using the Standard Method scripting) is shown in Figure 16.

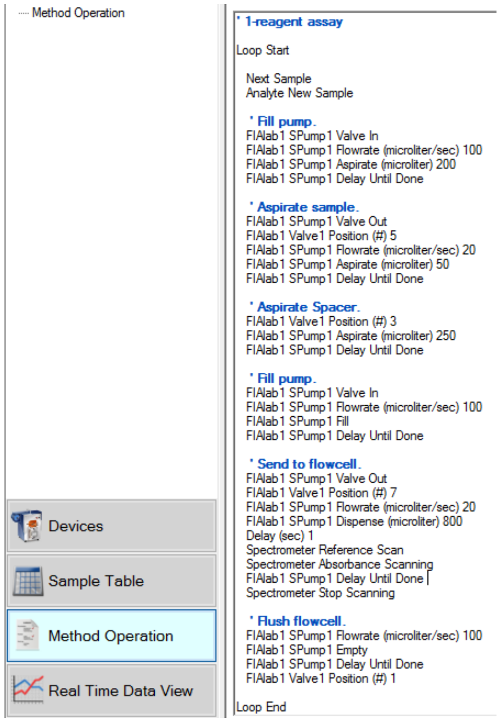


Figure 16 An instrument method (Standard Method script type) for repeated injections of 30 mg / L nitrate.

Figure 17 shows example data of 11 nitrate injections measured at 220 nm.

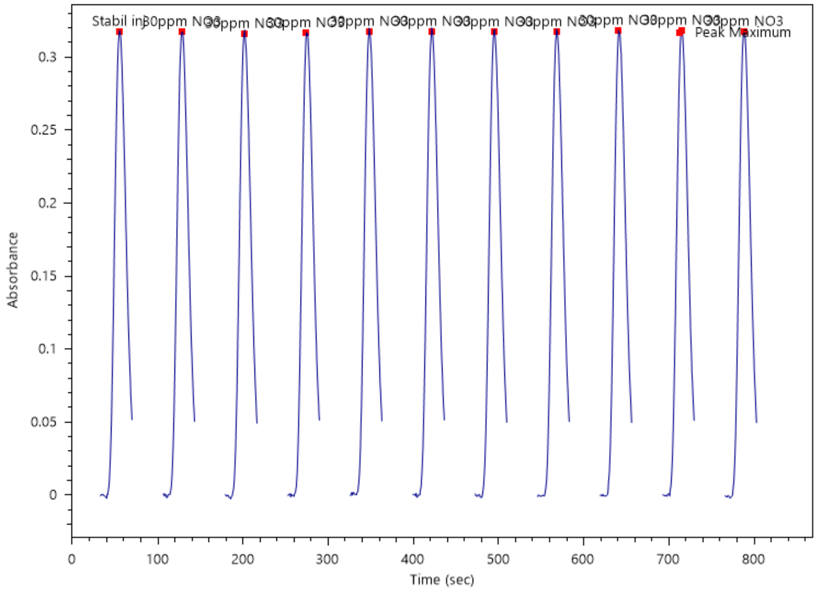


Figure 17 Example data.

5 Maintenance

5.1.1 Syringe pump

5.1.1.1 Syringe cleaning

It is recommended that the syringe is cleaned daily after use. Cleaning is best done in place without removing the syringe.

The cleaning process should always start by flushing the syringe with three strokes of deionized water. The syringe is then flushed with three strokes of cleaning solution (1:10 dilution of household bleach). Finally, the syringe should be rinsed with 10 strokes of deionized water.

In some cases the syringe may become soiled with substances that are not easily removed by bleach. Alternative cleaning solutions include 1% solution of laboratory detergent (e.g. Liquinox), 0.1 mol/L hydrochloric acid and 0.1 mol/L sodium hydroxide.

5.1.1.2 Syringe replacement

The pump syringe is replaced as follows.

1. Make sure that the syringe has been cleaned. The last solution pumped with the syringe should be water.
2. Place inlet tube in air. Pump all fluid out of the syringe.
3. Command the syringe to the full position.
4. Turn the plunger lock screw counterclockwise until it is loose.
5. Turn the syringe counterclockwise and remove it from the pump head valve.
6. Take a new syringe. Pull the plunger down about half-way. Screw the threaded tip of the syringe clockwise into the valve.
7. Push the plunger down manually until with the bottom end seats in the plunger holder on the pump drive.
8. Tighten the lock screw.
9. Initialize the analyzer to reset the pump.

5.1.2 Selector valve

5.1.2.1 Stator cleaning

1. Remove all connectors from the valve.
2. Undo the three mounting screws holding the stator and pull it off.
3. Soak the stator in 1% detergent solution and clean mechanically with non-abrasive tissue (e.g. Kimwipe). If heavily soiled, sonicate in detergent solution.
4. Rinse with water and dry.
5. Put the stator back on the valve.
6. Insert mounting screws and tighten in an alternating fashion. First tighten to snug finger-tight, then an additional 1/10 turn to make a good seal.

7. Re-attach fluidic connectors.

5.1.2.2 Rotor replacement

1. Remove the stator as described in section 5.1.2.1.
2. Pry off the plastic rotor from its steel cup.
3. Place a new rotor in the cup.
4. Wipe down the backside of the stator with detergent solution and water to remove any deposits.
5. Dry off the stator and put it back on as described in section 5.1.2.1.

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6 Specifications

Analyzer	
Type	Sequential Injection Analyzer
Enclosure material	Aluminum (powder-coated / anodized)
Syringe Pump	
Type	High-precision syringe pump
Wetted materials	Glass, Teflon®, PCTFE
Syringe sizes	50 µL, 100 µL, 250 µL, 500 µL, 1.0 mL, 2.5 mL, and 5.0 mL
Resolution	24,000 steps per full stroke
Speed	1.2 sec – 20 min per full stroke
Port type	Flat-bottom ¼-28
Valve	
Type	8-port multiposition selector valve. 6-port and 10-port also available. Optional Lab on Valve® manifold for 6- and 8-port valves.
Wetted materials	PPS, Valcon E2 (proprietary reinforced PTFE composite). In the case of Lab on Valve® manifold also Ultem® or Plexiglas.
Port type	Flat-bottom ¼-28
Relay output	
Voltage	24 V
Current	Max 4 mA
Dimensions	
Height	7 in (18 cm)
Width	5.25 in (13 cm)
Depth	6 in (18 cm)
Weight	8 lb (3.5 kg)
Communication	
Type	USB
Output	1 Serial RS-485 port
Control options	SIAsoft software
Power requirements	
Voltage	24 VDC
Current	max 2.5 A

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