FIBER OPTIC SMA Z-FLOW CELL MANUAL

Version 3

The line of fiber optic ***SMA Z-Cells*** is the top choice for many absorbance measurements. SMA-Z-Cell path lengths are available between 2.5mm - 100mm and can be made in PEEK, Plexiglas, Stainless Steel, Teflon, and Ultem. The cells contain fused silica windows and are compatible with standard SMA-905 terminated fiber optic cables.

The SMA Z-cell is designed to accommodate common optical measurements. It is designed to minimize bubble entrapment in flow injection and sequential injection and is readily disassembled for cleaning.

Please note that flow cells should not be exposed to aggressive materials for long periods of time and doing so may cause significant damage. If prolonged exposure is necessary, please consult the specification sheet in this manual to select a flow cell material most suitable to your application.

1 DESIGN

SMA-Z-Cells are designed to accommodate common optical measurements. The Z-shaped fluidic path allows continuous flow up through the flow cell, minimizing bubble entrapment.

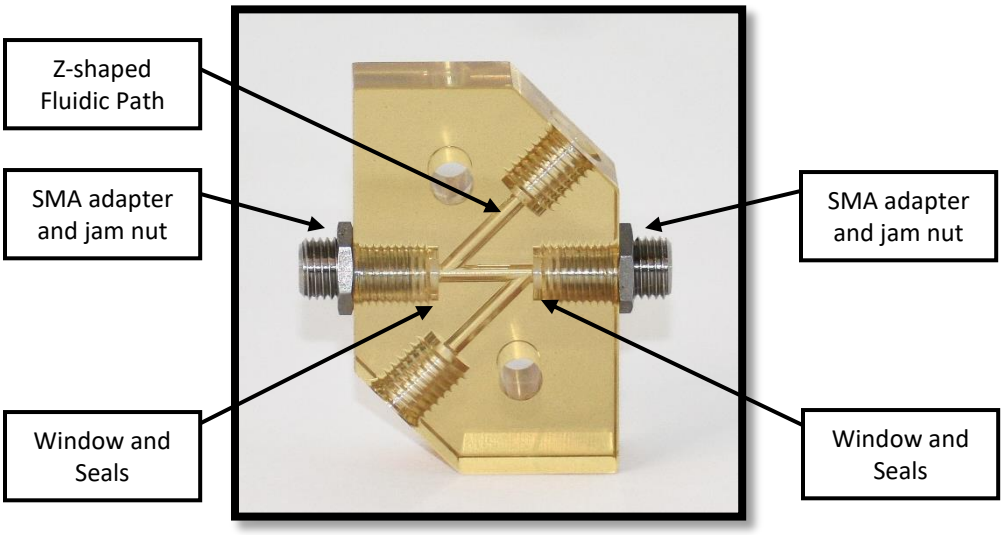

Figure 1: SMA-Z Cell Design

The flow cell optical path contains polished UV fused silica windows found at each of the cell's two fiber optic junctions. Non-corrosive and chemically resistant, these windows efficiently transmit light in the 170nm-2000nm range. Each window is sandwiched between two ring shaped Teflon seals, eliminating the possibility of fluid leakage through these ports.

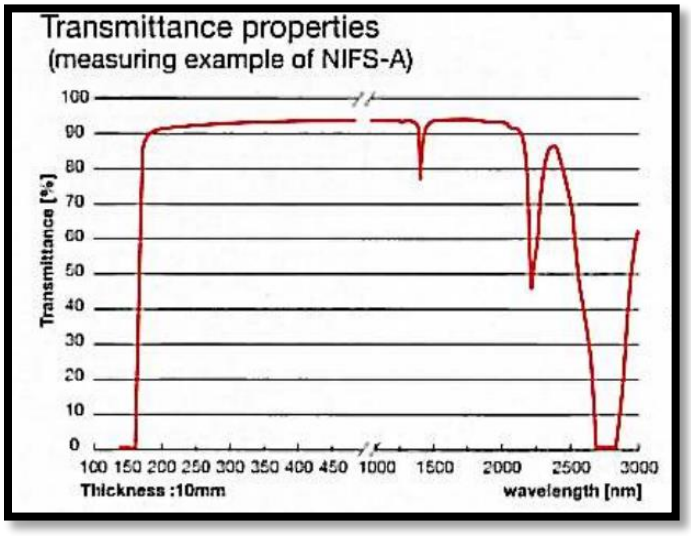

Figure 2: Transmission of UV/Silica Windows

¼-36 thread Stainless Steel SMA-905 connectors attach to the two optical ports on the flow cell and serve both to connect to external fiber optic cables and to hold the seals and windows in place.

2 SETUP

STANDARD SMA-Z CELL

To use the SMA-Z cell, connect tubing to the cell's fluidic ports. Cleanly cut the supplied tubing to the desired length and place a colored nut onto the tubing following with a blue ferrule (with the cone side of the ferrule pointing towards the nut). Screw the tubing into the flow cell so that approximately 5mm of tubing protrudes from the end of ferrule. It is CRITICAL that the tubing is not blocking any portion of the light path. Repeat this procedure for tubing connecting to the other fluidic port.

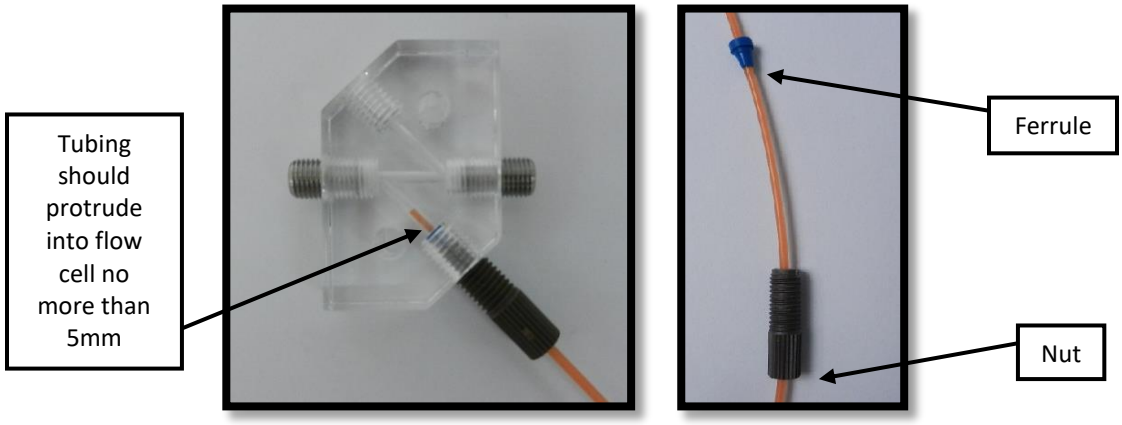

Figure 3: Inserting tubing into Z Cell fluidic path

MICRO-VOLUME SMA-Z CELL

The fluidic path of the microvol SMA-Z line is designed in such a way so that tubing cannot protrude into its light path. To connect tubing to the cell's fluidic ports, cleanly cut the supplied tubing to the desired length and place a colored nut onto the tubing, following with a blue ferrule (with the cone side of the ferrule pointing towards the nut). Insert the tubing into the flow cell until you feel resistance and then screw nut to tighten. Repeat this procedure for the other fluidic port. Tubing should be pushed into the flow cell as far as it will comfortably go in order to minimize dead volume.

Whether using a micro-volume model or not, fiber optic cables connect to each of the flow cell's SMA connectors[1]. One should connect to a light source and the other to an absorbance detector.

[1] A FIAlab DCON-LED may be used on one side of the flow cell instead of a fiber optic cable as a direct connect light source.

Mount the flow cell in such a way that the light path is tilted upward and fluid enters from the bottom, exiting at the top. This is done to minimize the likelihood that air bubbles will get caught in the light path as measurements are being taken.

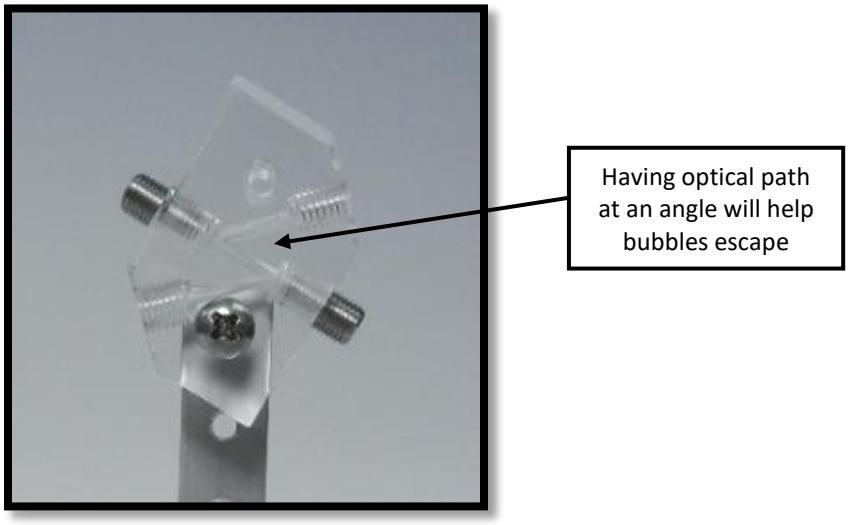

Figure 4: Mounting SMA-Z Flow Cell

3 USE AND TROUBLESHOOTING

To test for leakage, before putting any chemicals through the cell, pump water through for at least ten minutes at or as close to the pressure and temperature you intend to use for your measurements. If leaks are observed coming from the fluidic ports, tighten the nut and ferrule more securely. If leaks are coming out of the junction between the SMA connector and the flow cell, tighten the SMA connector according to the instructions for replacing windows on page 6.

Whenever measurements are made, take care to make sure that the fiber optic cables don't move. If your fiber optic cables move at any point during the course of measurements, light throughput may vary.

Lastly, the most common problem with any type of flow cell is that of bubbles getting stuck in the light path. Bubbles cause significant deviations from accurate measurement as they absorb a substantial amount of the light introduced to the cell. Furthermore, since bubbles do not always remain in the flow cell throughout the course of measurements, not all samples will be influenced by their presence leading to even greater inaccuracy across the run.

If bubbles are of a small size and are entering the flow cell in streams, the source is likely in the temperature of the reaction or the reaction chemistry. This may be helped by adding a back pressure coil to the fluidic outlet of the cell. A back pressure coil can be made from 10ft of 0.02"ID Teflon tubing, wound tightly into a coiled pattern. Note that using a back pressure coil will affect the pressure throughout the system and thus pump rate or tightness of pump tubing may need to be adjusted to give the same flow rate.

Figure 5: Waste Coil

To prevent large more sporadic bubbles from adhering to the flow cell wall, wash the flow cell first by pumping a soapy solution into the cell, letting it sit for a few hours and then rinsing well. It will also help to add a small amount of detergent to the reagents being used. Make sure that the detergent selected is compatible with the chemistry you are performing. 1gram detergent per liter of reagent is a common ratio.

For more assistance on bubbles, please see the bubble troubleshooting guide at the end of this manual.

4 WINDOW REPLACEMENT

When windows get stained, many users wish to replace them. Windows will also need to be replaced if for any reason they become cracked. To replace flow cell windows:

Unscrew one of the SMA fittings from the flow cell and tap the cell against the surface of a table until the seals and window fall out. Repeat this process on the other side. If windows do not come out on one or both sides, use a piece of 1/16th inch OD PEEK tubing cut at a sharp angle and insert into the flow cell to push them out. As an alternative, the flow cell can be soaked overnight in DI water.

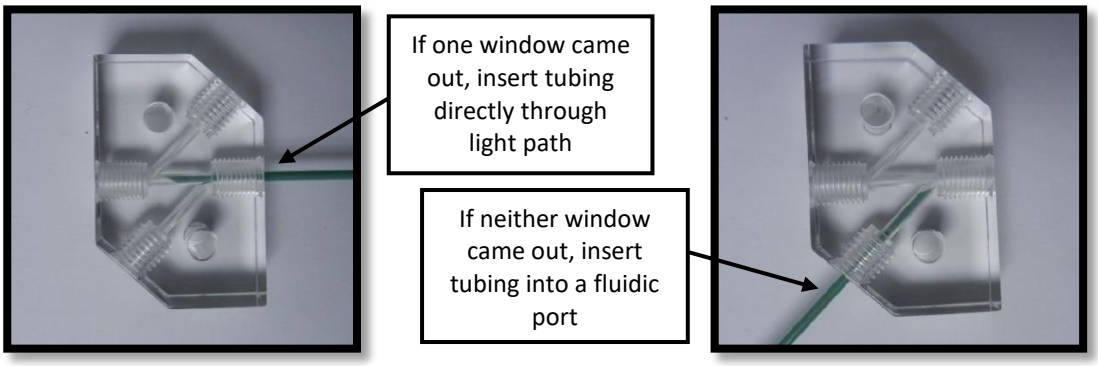

Figure 6: Use of PEEK tubing to remove flow cell windows

Make sure to clean the flow cell well with DI water or a 0.1% solution of detergent followed by a thorough rinse before replacing windows. Dry the flow cell completely, it may be helpful to use a Kimwipe and then also leave the flow cell to air dry overnight.

When dry, insert a new Teflon seal into one of the open ports. Follow this with a silica window and then again, another Teflon seal. To hold these parts in place gently screw in a SMA connector (the ones previously on the cell, if they have been maintained, can be reused) until it touches the seal but make sure to not tighten. Repeat this on the other side of the flow cell.

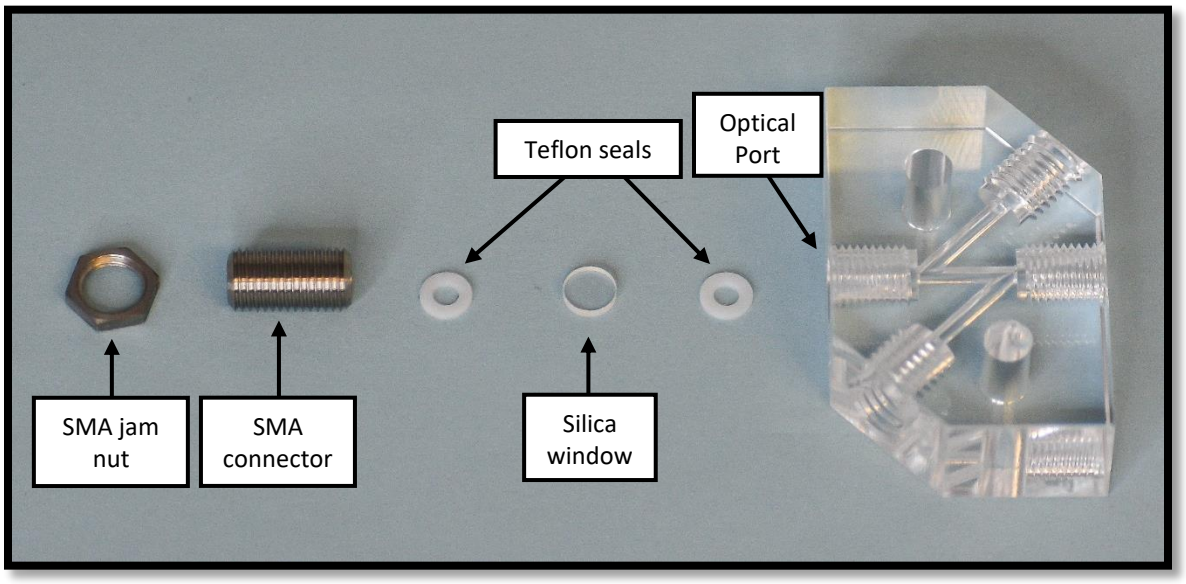

Figure 7: Components into flow cell optical port – Standard SMA-Z

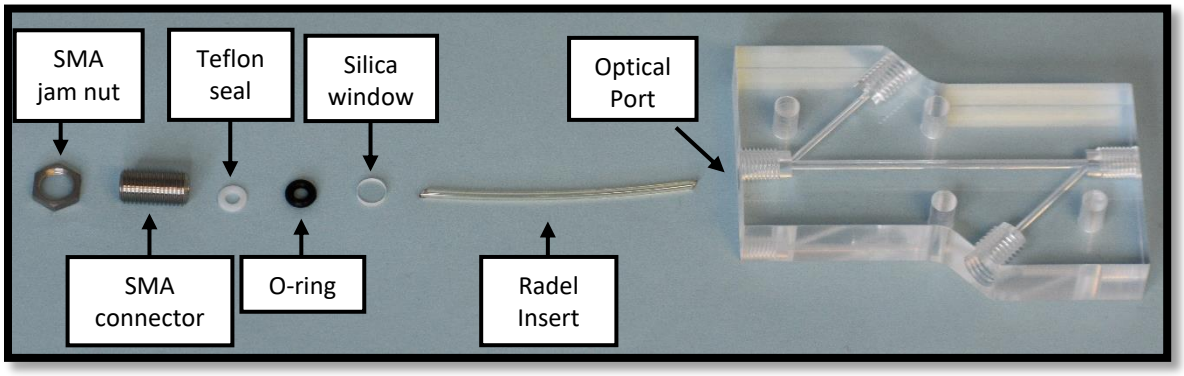

Figure 8: Components into flow cell optical port – Microvol SMA-Z

For microvol flow cells, there is an insert inside the optical path of the flow cell. The insert is reusable and does not need to be cleaned. If removed during the cleaning process, use a piece of PEEK tubing to align the insert inside. When dry, insert the silica window first, then a black Viton O-ring, and then a Teflon seal. To hold these parts in place gently screw in the SMA connector (the ones previously on the cell, if they have been maintained, can be reused) until it touches the seal but make sure not to tighten. With one side assembled, use the PEEK tubing to align the insert with fluidic opening, making sure not to obstruct the Z shaped fluidic path. After aligning the insert, assemble the other side of the flow cell.

Place a piece of large ID Tygon tubing snugly around one SMA connector and use pliers to tighten. Repeat on the other side. Add the jam nut onto both sides and use a wrench to tighten, do not over-tighten.

To ensure the optical port is sealed off from the fluidic path, perform a leak test using a luer syringe and plug fitting. Insert a piece of plug tubing on one fluidic port of the flow cell and an empty syringe (extended) to the other fluidic port. Push on the syringe's piston and check if it bounces back to ensure that the cell is airtight. Hold the flow cell up to your eye in room light and make sure you can see a clear path to check that the windows have not cracked in the replacement process.

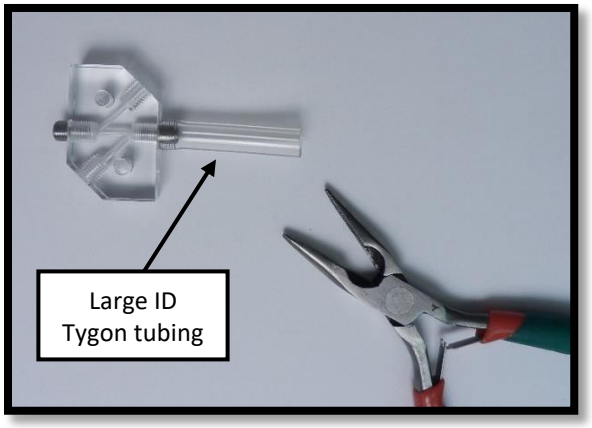

Figure 8: Tightening SMA connectors

5 SMA-Z CELL SPECIFICATIONS

Internal Volume (µL)

Path Length (mm)	**2.5**	**5**	**10**	**20**	**50**	**100**
Standard (µL)	2	13	26	52	130	260
Micro-volume (µL)	2	3	6	12	30	60

Physical Parameters

	PEEK	**PS**	**ACRYLIC**	**ULTEM**	**TEFLON**	**STEEL**
Temperature (C)	100	100	100	100	100	300
Pressure (psi)	100	100	100	100	100	100

Chemical Compatibility (common solvents – room temperature)

R = recommended; NR = not recommended

	PEEK	**PS**	**ACRYLIC**	**ULTEM**	**TEFLON**	**STEEL**
Acetic Acid	R	R	NR	R	R	NR
Acetone	R	NR	NR	-	R	R
Acetonitrile	R	NR	NR	-	R	R
Ammonia	R	R (<30%)	R	-	R	NR
Benzene	R	NR	NR	NR	R	R
Chlorinated Hydrocarbons	NR	NR	NR	NR	R	-
Detergent Solutions	R	R	-	R	R	R
Hydrochloric Acid	R	R (<20%)	R (<35%)	R	R	NR
Hydrofluoric Acid	NR	NR	NR	NR	R*	NR
Hydrogen Peroxide (<40%)	R	R	R (<30%)	-	R	R
Isopropyl Alcohol (<50%)	R	-	NR	R	-	-
Methanol (pure)	R	R	NR	R	R	-
Nitric Acid (20-70%)	R	R	R	R	R	R
Phosphoric Acid (<10%)	R	R	R	R	R	NR
Potassium Hydroxide (50%)	R	R	R	R	R	R
Sodium Hydroxide	R	R (<50%)	R	R	R	NR
Sodium Hypochlorite	R	R	R	R	R	R
Sulfuric Acid (<30%)	R	R	R (<15%)	R	R	NR
Phosphate Buffered Saline	R	R	R	R	R	R

*Requires Sapphire Windows

6 BUBBLE TROUBLESHOOTING GUIDE

Because FIAlab flow cells are so versatile, they are used all over the world in many different configurations. Different components upstream of the flow cell in differing configurations lend to differing causes and origins of bubbles – the most common problem encountered in cell measurements.

Make sure the flow cell is oriented so that the path of the fluid always travels upward. The flow cell should always be used in this orientation because here bubbles are more likely to slip easily to the top of the flow cell and out of the optical path.

Large or small bubbles that travel through the flow cell while measurements are being taken will cause inaccuracy and imprecision. In addition, bubbles that get stuck in the optical path will likewise negatively influence results. To fix any bubble problem that may arise it is necessary to first find the bubble(s) origin. Possible origins of bubble formation follow and tips for removing stuck bubbles can be found at the end of this guide.

Chemical causes of bubbles:

Degassed Reagents:

Bubbles will come out of solution during heating as air is less soluble at higher temperatures. Rather than having this occur in a closed system, it is a good idea to degas reagent and carrier water offline prior to running experiments. To easily degas, place DI water under a vacuum and gently heat or stir for 20 minutes. Use this water to make reagents. DO NOT DEGAS SAMPLES.

Refrigerated Solutions:

As air is more soluble in cold temperatures, when a reagent is left in a refrigerator for prolonged periods of time, air will pervade the solution. Do not store solutions in the refrigerator unless necessary. If refrigeration is required, store solutions in sealed glass or metal bottles, when possible, as plastic containers are more susceptible to air permeation.

Reaction chemistry:

It is inevitable that certain reactions will create bubbles when mixed within the system. Certain reactions also need to take place at a high enough heat where even mostly degassed reagents will create bubbles. If solution is actively being pumped into the flow cell, adding a back pressure coil at the flow cells outlet port will increase pressure upstream and force any small bubbles that want to form to remain in solution. A simple and cost-effective back pressure coil can be made by tightly coiling a 20ft length of 0.02” ID Teflon tubing.

Hydrophobic surfaces:

If bubbles get trapped at specific places, hydrophobic surfaces within the system created by salts, contaminants or oil residues may be the cause. Washing the system with a 1% detergent solution is absolutely necessary and it is best if this solution can be left in the system overnight. Rinse the system thoroughly with DI water after washing and make sure to wash out the setup at the end of each day. If compatible with the chemistry being run, adding a small amount of detergent (total 0.1%) to the carrier or reagent solutions will help prevent future bubbles by dramatically lowering surface tension.

Fluidic causes of bubbles:

Fittings:

If bubbles appear to be coming from a specific fluidic junction in the setup, it is likely that the nut and ferrule at this point is either too loose or too tight. If the fitting is loose, air can easily enter the system through the gaps at this connection. Tightening the fitting should eliminate these bubbles but DO NOT OVERTIGHTEN. Overly tight fittings can be a problem as they can collapse and crimp the tubing inside. There is also the possibility that the ferrule or nut is defective. To fix a problem fitting that cannot be remedied by tightening, unscrew the fitting, remove the nut and ferrule and trim the tubing so the portion formerly held by the fitting will no longer be used. Tubing should be cut with a square face tubing cutter. Clean out the port where the fitting was connected with a damp Kimwipe and dry thoroughly. Using a new nut and ferrule, reconnect the tubing and tighten just to the point where everything is securely held in place.

Tubing:

When dissolved gases exit a narrow constriction, bubbles will often come out of the solution as the result of a drop in pressure. Upstream of the flow cell constriction is likely due to kinked or misaligned tubing. Manually push solution through the system with a syringe to test for back pressure issues and replace any tubing that has warped or reconnect any tubing that is misaligned.

Component causes of bubbles:

Selector Valves:

If a port on a component is not used but left open it creates a prime location for air to enter the system as the pressure difference between the system and its surroundings may be significant. A good example of such a situation is a selector valve used in conjunction with a syringe pump where the fluidic path may sweep past an open port when the pump is not dispensing. Make sure all unused ports are securely plugged with a nut, ferrule and solid Teflon tubing.

Syringe Pumps:

Bubbles can enter the syringe of a syringe pump if solution is aspirated into the pump too quickly. Pulling too quickly can create a vacuum that will pull dissolved gases out of the fluid and form a head space. The

speed at which the pump can effectively draw solution is a function of the ID of the tubing that the solution is being pulled through. Change to a larger ID tubing on this pump port or slow down the flow rate when the pump aspirates.

Bubbles can pass through the syringe piston if the Teflon seal at the top has become worn. In this situation bubbles will be seen floating up from the plunger tip and through the syringe. Replace the syringe.

Tips to remove a stuck bubble:

Bubbles will always pass through the system when primed if the tubing in the system was left dry since its past use. If one of these bubbles gets stuck in the flow cell the following tips are easy and efficient ways to dislodge it.

Tap firmly on the side of the flow cell while solution is flowing through. FIAlab flow cells are very robust; do not be afraid to tap vigorously and with medium force. Tapping is more effective when solution is pumped through the flow cell at a high flow rate

Introduce a large bubble to capture the stuck bubble as it travels through the cell's fluidic path. Stop any

flow of solution and with the flow cell oriented so that fluid entering the cell will travel upwards and out, loosen the fitting on the cell's inlet. Slowly a significantly sized bubble will form and begin traveling through the cell. Retighten the fitting and pump solution through as normal.

Replace the outlet fitting of the flow cell with a 1ml luer lock syringe and pump the piston up and down to make the bubble move within the flow cell path. Remove the syringe and replace the outlet fitting. As the bubble is no longer stuck in a single position, it will be more easily removed when solution is pumped through.

7 CONTACT FIALAB

https://www.flowinjection.com/
For reorders: sales@flowinjection.com
For support: support@flowinjection.com
Phone: 206-258-2290

FIAlab Instruments, Inc.
2151 N Northlake Way
Seattle, WA 98103
USA