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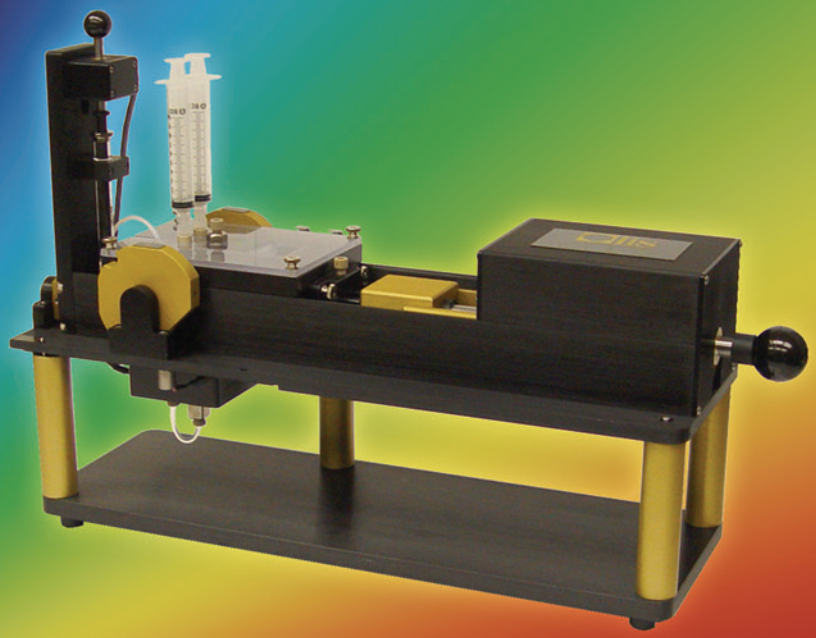
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USA Stopped-Flow Manual

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USA Stopped-Flow Manual

Mixing device for preparation of reactive soilutions in milliseconds

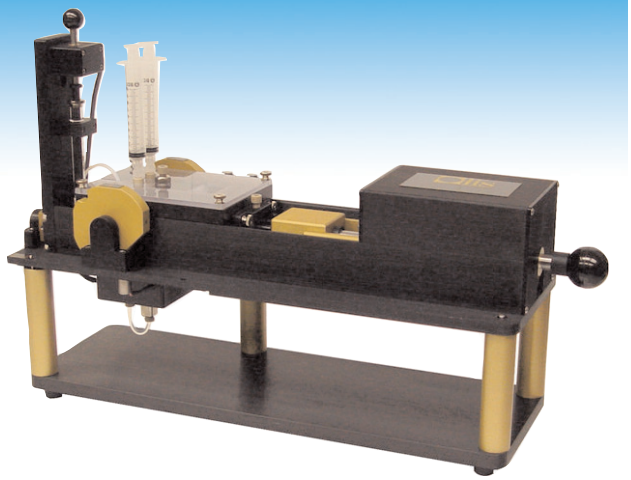
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Stopped-Flow Maintenance

The user can perform most maintenance procedures required to keep the OLIS USA Stopped-Flow in good working condition. Routine maintenance practices are described below.



PROPER USE

Do not use your stopped-flow unit outside of the temperature range for which it is intended.

The temperature range is determined by the type of seals. OLIS has an ambient-range model (10-80°) and two models (low-temperature and fluorescence) that stretch the low end of the range to 0°C. Identify the type you have. Operating a stopped-flow at temperatures outside of its range causes damage that is expensive to repair!

Do not use low-temperature syringes at temperatures above 6°C. The low-temperature syringes are designed to provide a very tight fit for low-temperature operation. At temperatures above 6°C, however, when the syringe shaft is at normal state (i.e., not contracted), retracting the syringe shaft will cause the safety washer on the tip to fall off. If you need to use your low-temperature stopped-flow at temperatures above 6°C, replace the low-temperature syringes with standard OLIS stopped-flow syringes. **Using low-temperature syringes at temperatures above 6°C will void your warranty.**

The following procedure may help to prevent dislodging the safety washer on the tips of low-temperature drive syringes: When retracting the shaft of the drive syringe, use your other hand to push down on the corresponding fill syringe at the same time. Simultaneously pushing down on the fill syringe eliminates the vacuum produced by pulling out on the drive shaft, which reduces the friction that causes the safety washer to become dislodged. In this manner, you are using the fill syringe to "push out" the shaft of the drive syringe with the safety washer intact.

CLEANING

The importance of keeping your stopped-flow unit clean cannot be overemphasized. Treat your stopped flow just as you would any sophisticated piece of laboratory equipment: keep it in good working order by cleaning it frequently.

1. Use distilled water to flush valves and syringes. Refer to the attached flow path diagram.
2. If your OLIS stopped-flow is an anaerobic model, flush valves and syringes with de-gassed distilled water after flushing with distilled water to minimize introduction of oxygen.
3. Use a toothbrush, cotton swab, test tube brush, or clean cloth as necessary to remove any dust, corrosion, or other type of contamination from all parts of the stopped-flow unit.
4. Gently wipe the stopped-flow unit using a clean cloth dampened with soapy water.
5. After the unit has been completely cleaned, finish by spraying furniture polish on a clean cloth and rubbing the cloth over the unit. Furniture polish protects the unit's anodized surfaces from chemicals and moisture.
6. Maintain clean water bath fluid at all times (see the following section on "water bath fluid mixture"). The water bath should be emptied and cleaned occasionally using a cotton swab or clean cloth. If the stopped-flow is to be left idle for some time (such as during a holiday), drain and clean water bath chamber and leave it empty during the period in which the stopped flow will be left idle.

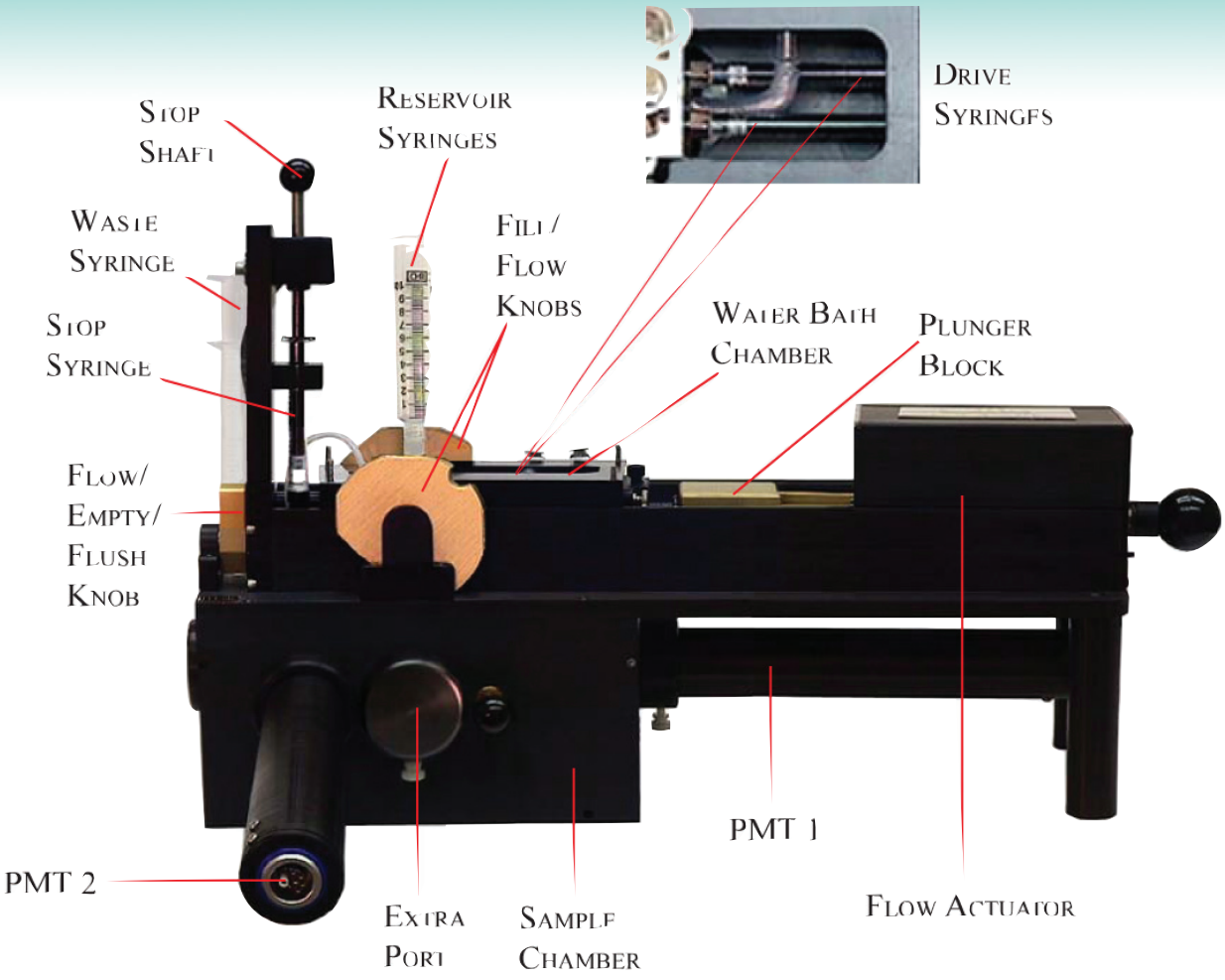
The OLIS USA Stopped Flow mixer will provide many years of service if properly maintained. **Failure to maintain your OLIS stopped-flow may void your warranty.**

WATER BATH FLUID MIXTURE

Distilled water should be used as the stopped-flow water bath fluid. If you are using the stopped-flow at temperatures below freezing, add Methanol or antifreeze as needed to keep the water bath from freezing. As a general rule, approximately one part alcohol or antireeze to ten parts distilled water is sufficient to prevent the fluid from freezing when the stopped-flow is used at temperatures to approximately -2°.

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Frontal View of the USA Stopped-Flow



Components of the Stopped-Flow

RESERVOIR SYRINGES
These two syringes hold the unmixed reagents before they are introduced into the drive syringes. These syringes can be plastic (as shown), tonometers, or specialized fluid holders.

FILL/ FLOW KNOBS
These knobs control the valves between the reservoir syringes and the drive syringes. There are two fill/flow knobs, one for each drive syringe. Putting the knobs in "fill" position opens the valve between the reservoir and drive syringes to allow the drive syringes to be filled with reagent and closes the valve between the drive syringes and the mixing chamber to keep the reagents in their respective syringes prior to the firing of the stopped-flow. Putting the knobs is in "flow" position closes the valve between the reservoir syringes and the drive syringes and opens the valve between the drive syringes and the mixing chamber to allow the reagents to flow into the mixing chamber upon firing of the stopped-flow.

DRIVE SYRINGES
These two syringes are actuated by the pneumatic drive to introduce the reagents into the mixing chamber. Reagent ratios of 1:1 to 1:11 are achieved through the use of same or different sized syringes.

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FLOW ACTUATOR
The pneumatic cylinder which depresses the drive syringe plungers when the stopped-flow is fired, pushing the reagents from the drive syringes into the mixing chamber.

FLOW/ EMPTY/ FLUSH KNOB
This knob controls the valve between the mixing chamber and drive syringes and between the stop syringe and purge syringe. Putting the knob in "flow" position opens the valve between the mixing chamber and the stop syringe, allowing the reagents to complete the flow path when the stopped-flow is fired. Putting the knob in "empty" position opens the valve between the stop and purge syringes, allowing the spent reagents to be emptied. Putting the knob in "flush" position opens the valve between the drive and purge syringes, allowing the drive syringes and observation chamber to be flushed between experiments.

SAMPLE CHAMBER
The compartment of the host spectrometer which houses the sample cell of the stopped-flow, the incoming light (measuring beam and/or actinic beam) and the photomultiplier tube(s). When provided by Olis, is normally the 6 x 6 x 4" box shown in most photographs in this document.

EXTRA PORT
A port in the sample chamber which can house the detector for emission detection or a light source for a photolyzing flash input. Not necessarily available in non-Olis host spectrometers.

PMTS
The recommended detectors are end-on photomultiplier tubes for single or dual beam detection throughout the 190-900 nm spectral range; specialized detectors are available for other spectral ranges. PMT 1 is the straight-through position for the absorbance signal and PMT 2 is set at 90° to the sample for optional reference detection. For emission readings, the sample PMT is in the position labeled 'extra port' and a reference or ratio detector can be placed between the light source and sample chamber to allow monitoring of the excitation light.

PLUNGER BLOCK
The block between the flow actuator and the drive syringes which pushes the drive syringe plungers, thus initiating the mixing process.

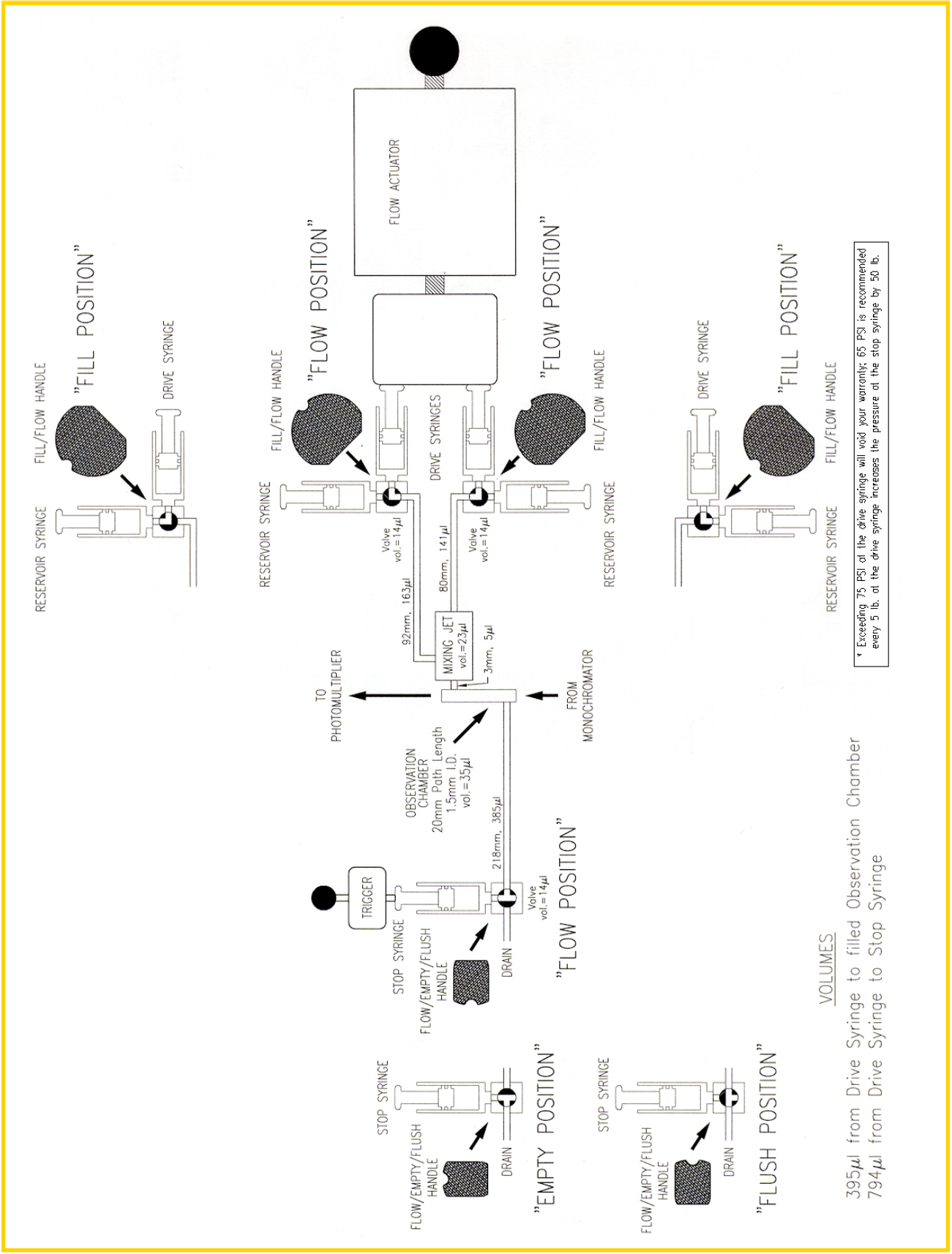
The photos below show the stop shaft in the process of passing through the stop sensor field. In the photo on the left, the machined groove has not yet reached the sensor field. In the middle photo, the machined groove has just entered the sensor field, activating the stop sensor. In the photo on the right, the flow has stopped and the top of the stop syringe plunger rests against the underside of the sensor housing.



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USA Stopped-Flow Block Diagram

Withpathlengths and flow volumes



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Changing Syringes

Changing the stopped-flow syringes is required when a syringe breaks or when you want to switch to variable-ratio mixing.

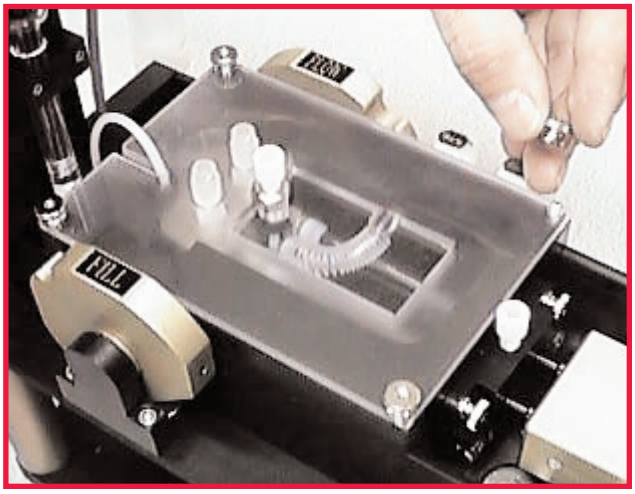
Changing syringes is not difficult, but does involve the risk of damaging your stopped-flow. The following instructions take you step-by-step through the process and provide illustrative photos for most steps. Before changing the syringes, read all the instructions to ensure that you understand the process and to assess your level of comfort in performing the task.

Please note that overtightening a syringe during installation may severely damage the syringe and associated valve, rendering the stopped-flow inoperable and requiring expert repair by OLIS personnel. If you prefer not to change the syringes yourself, or if syringe replacement is not permitted by your institution, OLIS will be happy to replace syringes for you at not cost except the price of new parts and shipping and handling fees both ways. This warning is given not to discourage you from attempting to change the syringes yourself, but to ensure that you are aware of the risk of damage involved and to offer you the alternative of having OLIS take the responsibility.

The tools needed to change stopped-flow syringes are included in tool kit provided with purchase.

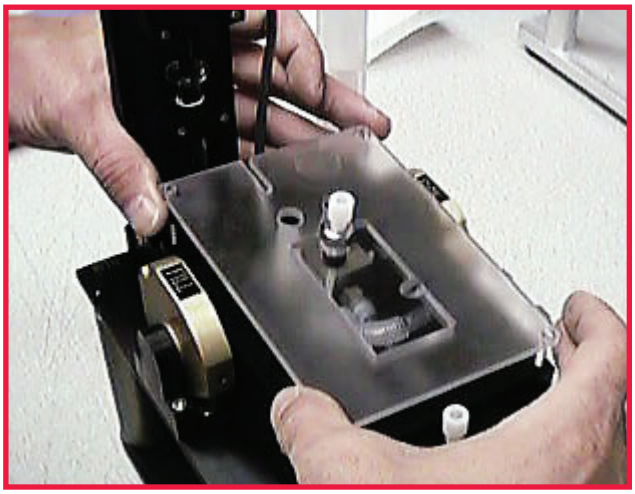
Step 1

Remove the four thumbnuts from the cover of the water bath chamber.



Step 2

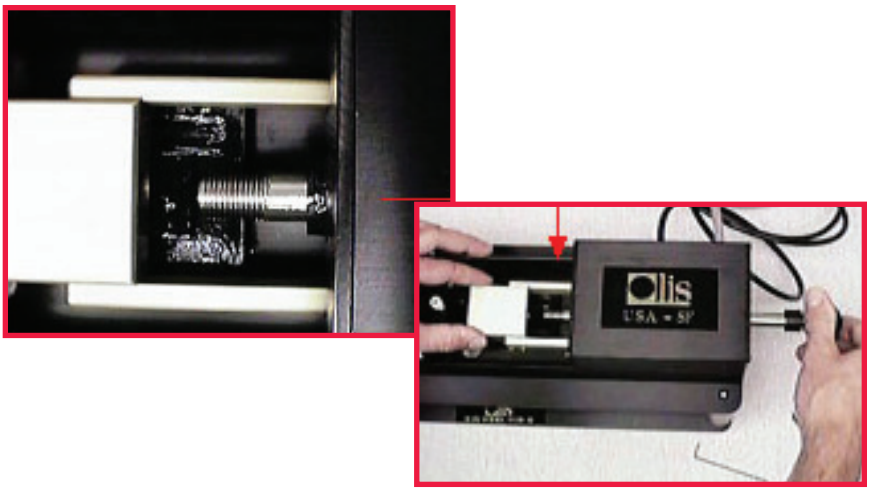
Gently remove water-bath cover, easing it carefully over syringe extenders.



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Step 3

In your right hand, grasp the black knob at the end of the cylinder shaft; in your left hand, hold the plunger block steady. Unscrew the cylinder shaft from the plunger block by turning the knob counterclockwise.



Step 4

Lift the plunger block assembly as shown in photo 4a and move it out of the way; do not disconnect the wires, but allow the plunger block assembly to rest against the side of the air cylinder cover. To keep the plunger block assembly out of the way, you may secure it using a rubber band, as shown in photos 4b and 4c.

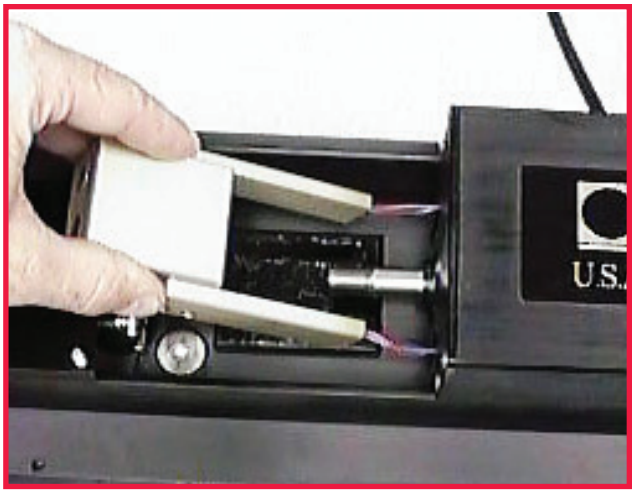


Photo 4a

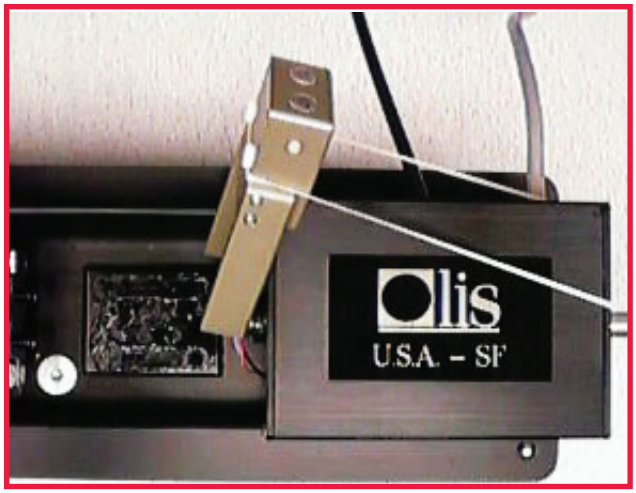


Photo 4b

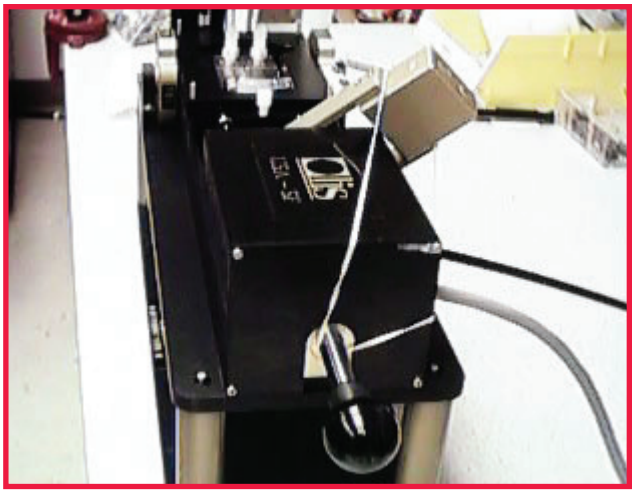
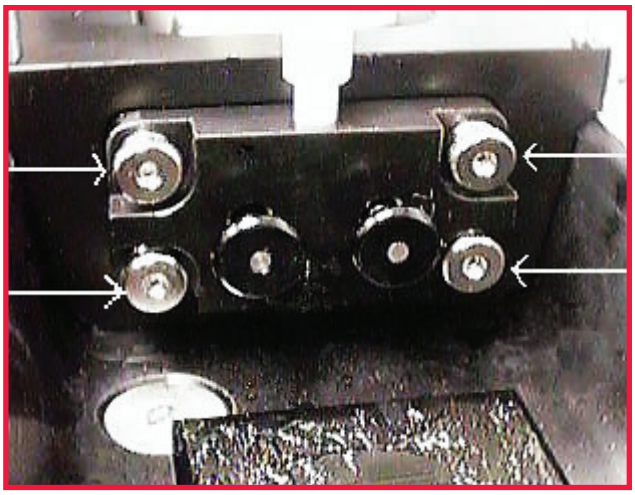


Photo 4c

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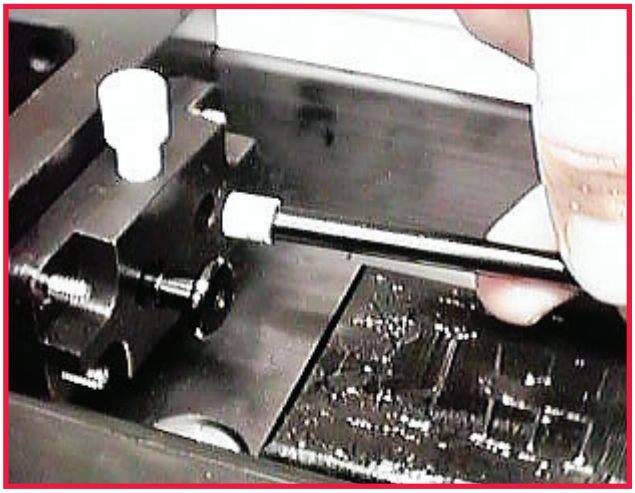
Step 5

Remove the four thumbnuts from the purge cap.



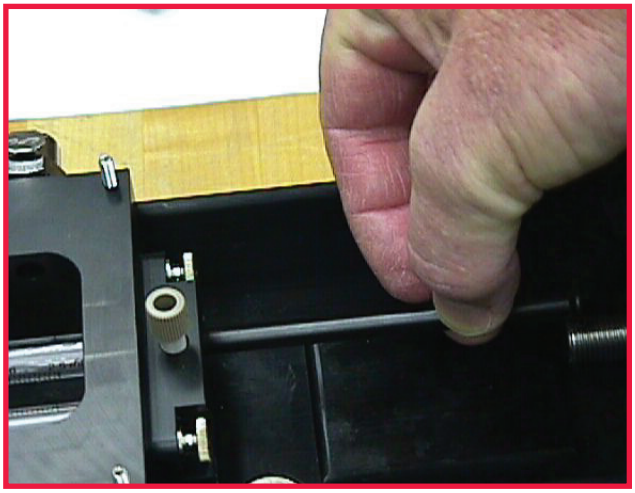
Step 6

Remove the plunger from the body of the syringe by gently pulling it straight out.



Step 7

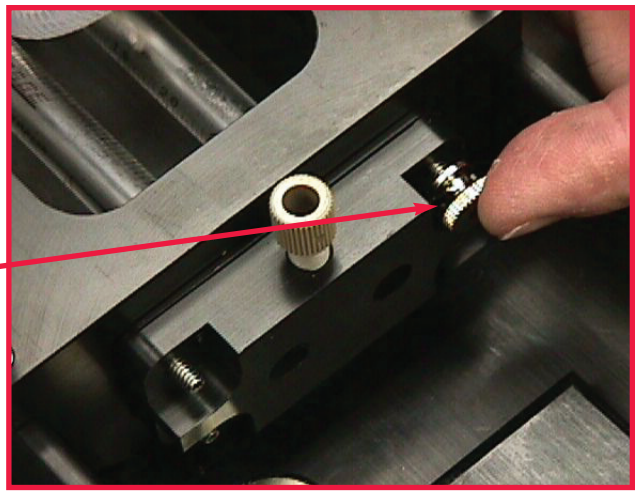
Remove syringe plungers.



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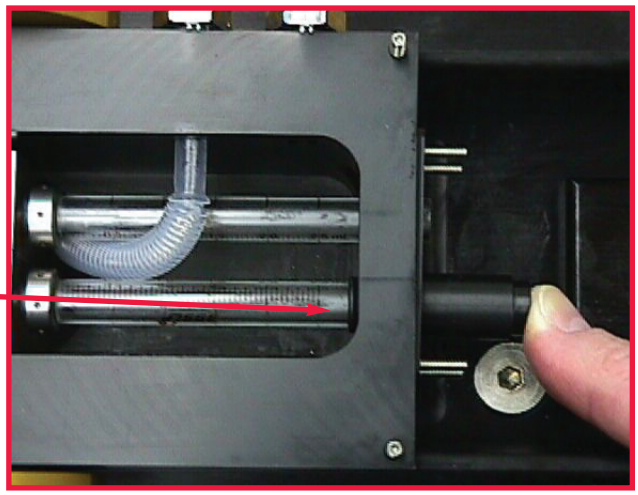
Step 8

Remove four thumb nuts and remove purge cap.



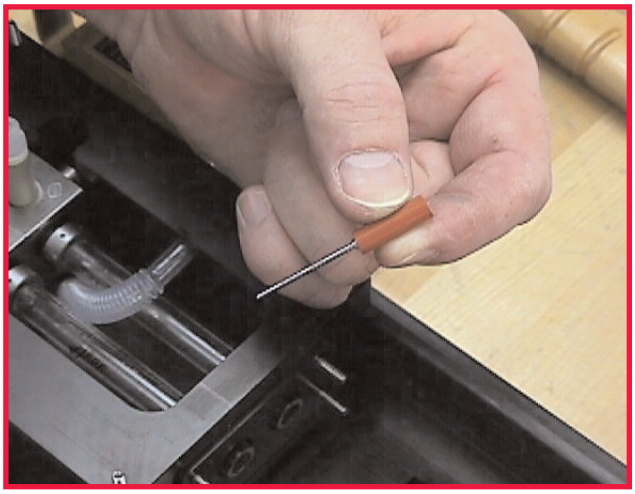
Step 9

Use long side of O-ring tool, push O-ring forward into bath.



Step 10

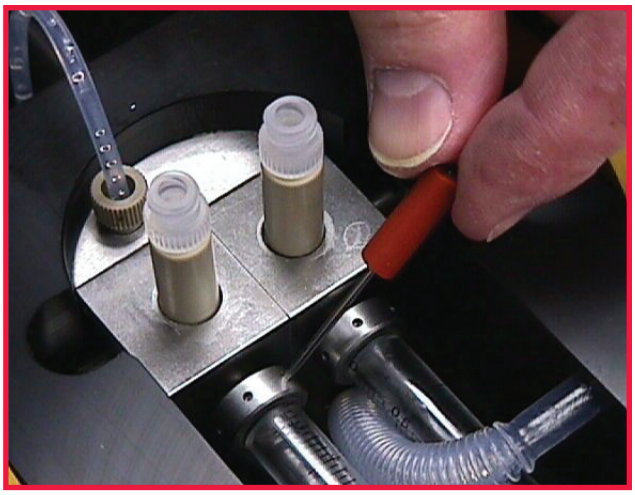
1/16” pin wrench used for installing or removing syringes.



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Step 11

Use 1/16” pin wrench to remove or install syringes. **Counter clockwise to remove and clockwise to install.** Note: Over tightening will cause damage to valves; use caution when installing. You should hand tighten, then use the pin wrench about a 1/4 turn to complete installation.



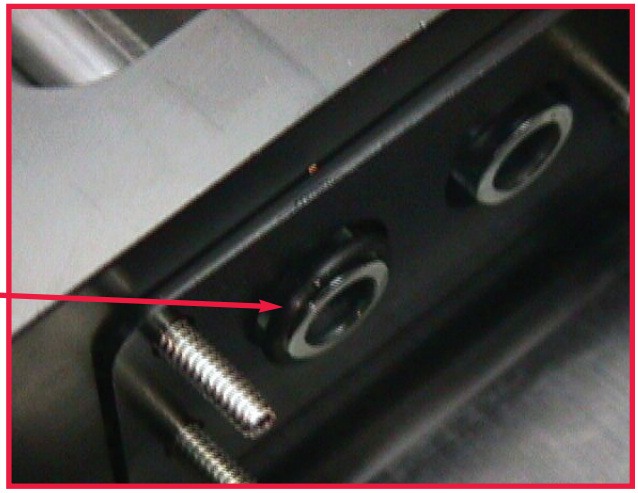
Step 12

A thin layer of lube should be added to O-rings prior to installing.



Step 13

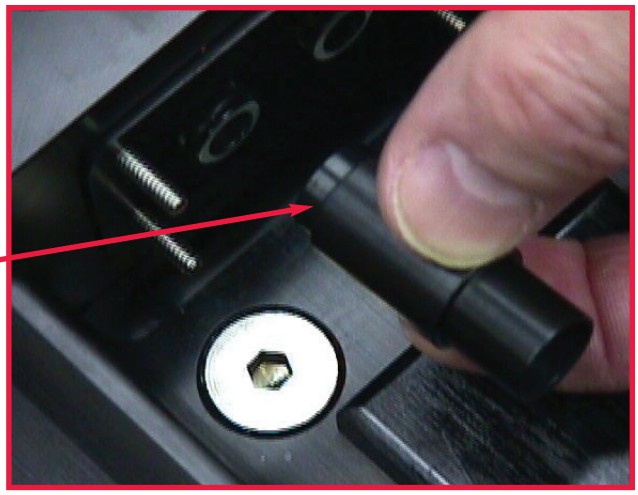
Place one of the lubed O-rings on the syringe barrel.



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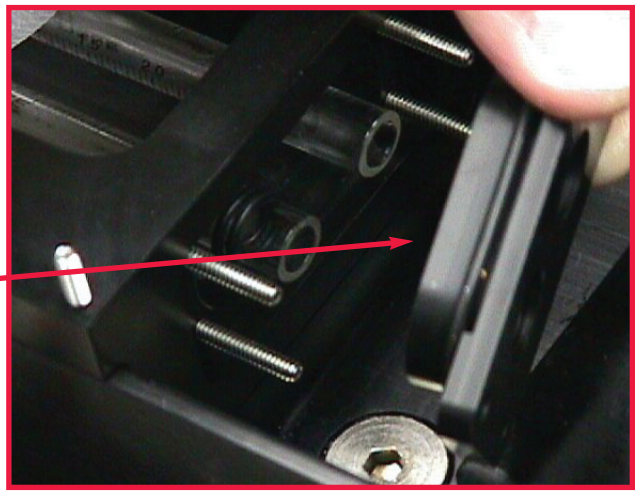
Step 14

Use small side of O-ring tool to push the O-ring into place. There are two O-rings per syringe.



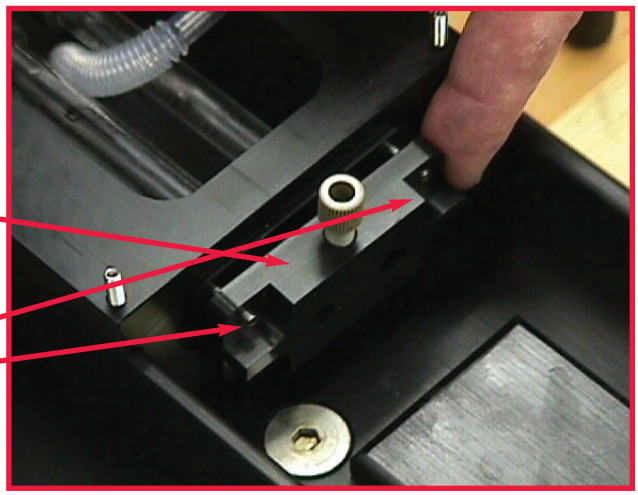
Step 15

Check condition of O-ring on bath syringe clamp.



Step 16

Install purge cap and four thumb nuts..



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Timing Sequence of the USA Stopped-Flow

Data Collection Period

Pre-Experiment Data Collection Period

Delay 1

The first delay is an optional user-set period during which useful 'pre-trigger data' are collected; it ends with the issuance of the "fire stopped-flow" command by the Olis software.

Start data collection; user presses "C" to initiate data collection

Delay 2

The second delay is a fixed instrument-imposed period during which the stopped-flow hardware prepares for firing (valves open, gas begins to flow, pressure builds) and fires (reagents flow, mixing occur); it ends with the activation of the stop sensor.

"Fire Stopped-Flow" command is issued.

Adjustment Period*

The adjustment period of the stopped-flow timing sequence is the time it takes for the stop shaft to rise from the position shown in the middle photo (stop sensor activation) to the position shown in the right photo (stop of flow); the range is typically a few milliseconds.

Stop switch is activated.

Experiment Time

User-set period for collection of reaction data. User fires stopped-flow. Flow actuator pushes plunger block. Plunger block pushes drive syringe plungers, forcing approximately 100 ml of each solution into mixing chamber. Stop is caused by stop syringe. Observation is made. Concurrently, any previously mixed solution (spent solution) is forced from observation cell into stop syringe. Prior contents of stop syringe are now in waste syringe.

Start data collection; user presses "C" to initiate data collection

As the mixed reagents are pushed into the stop syringe, forcing the stop shaft upwards, the shaft passes through the sensor field. When the machined groove in the shaft enters the sensor field, the stop sensor is activated and the software is alerted to the impending stop of flow. Approximately 2–5 milliseconds later, the plunger in the stop syringe strikes the underside of the sensor housing and the flow stops.

* The adjustment period is a user-determined period between activation of the stop sensor and the actual stop of flow; it is provided to enable the user to synchronize the start of reaction data collection ("experiment time") with the stopping of reagent mixing ("flow stops"). This adjustment period is needed because of the difficulty of synchronizing the two events, which are separated in time; differences in ambient conditions make the interval between the two events variable in different OLIS stopped-flow mixers. By setting the length of the adjustment period, the user is able to establish the ideal timing sequence for a particular stopped-flow.

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