INTERFACIAL EXCHANGE CELL
for the DHR/AR Rheometer

Getting Started Guide

Revision A Issued December 2017

Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

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Introduction

Important: TA Instruments Manual Supplement

Please click the TA Manual Supplement link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- TA Instruments Offices

Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2004 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL61010A-2-010:2002 Particular requirements for laboratory equipment for the heating of materials + Amendments.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 1 - Basic immunity test requirements.

For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

Safety

Do not attempt to service this instrument, as it contains no user-serviceable components.

Required Equipment

While operating this accessory, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

Instrument Symbols

The following label is displayed on the accessory for your protection:

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot surface. Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.

Please heed the warning labels and take the necessary precautions when dealing with these areas. This *Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

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Chapter 1:

Introducing the Interfacial Exchange Cell

Overview

The Interfacial Exchange Cell (IEC) accessory for the DHR/AR Rheometer allows for the study of the interfacial properties of a system while varying the properties of the subphase component. The Interfacial Exchange Cell is used in conjunction with the Double Wall Ring (DWR) to measure interfacial properties. The cell consists of a Delrin trough and aluminum base, and mounts onto the standard Peltier Plate. The trough is connected to a syringe pump with PFA tubing and PEEK fittings. The syringe pump controls the equal exchange of the subphase fluid, keeping the interface level (height) stationary throughout the experiment. The inlet/outlet ports for the subphase fluid exchange are designed to minimize the disturbance of the rheological measurement at the interface.

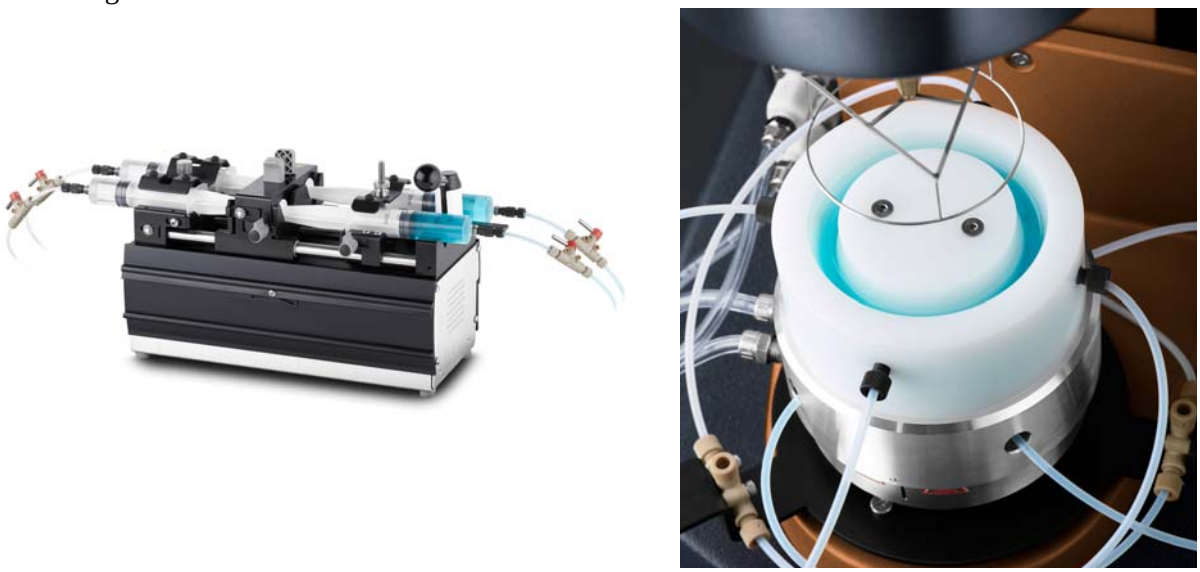

Figure 1 Interfacial Exchange Cell syringe pump (left) and trough (right).

Specifications

Refer to the table below for IEC system specifications:

Table 1: Interfacial Exchange Cell Specifications

Subphase Volume	37.7 mL
Recommended Infusion Rate	6 mL/min
Trough to Tee Fitting Tube Length	280 mm
Maximum Operating Temperature	100°C

Chapter 2:

Installing the Interfacial Exchange Cell

Installing the Interfacial Exchange Cell

The Interfacial Exchange Cell installs on the standard Peltier Plate. The aluminum base has three ball plungers used to locate the cell on the Peltier Plate. The eight inlet and outlet tubes connecting the trough to the tee fittings should be 280 mm in length. 16 precut lengths of PFA tubing are provided in the kit, and 15 feet of uncut tubing is also supplied. Make sure all tubing lengths are equal to ensure a uniform pressure drop between the pump and the inlet and outlet port. The different fittings supplied with the kit are detailed below

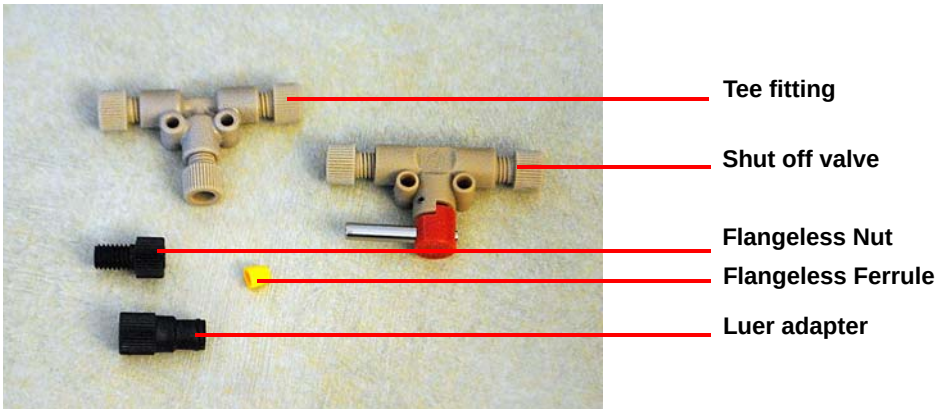

Figure 2 Fittings included.

1 Raise the DHR stress head to maximum height.

2 Remove all upper test geometries as well as any lower Smart Swap™ base attachments.

3 Remove the gold protection cover at the Smart Swap base. Turn counter-clockwise to detach the cover plate.

Figure 3 Remove cover.

4 Remove the three screws using a 4 mm hex key and replace them with the standoffs (P/N 546623.001).

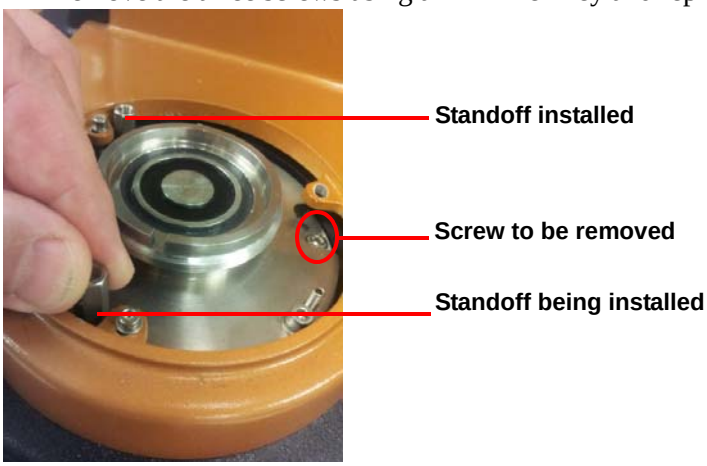

Figure 4 Remove screws and install standoffs.

5 Install tee fittings to the fitting bracket using the supplied M3 screws.

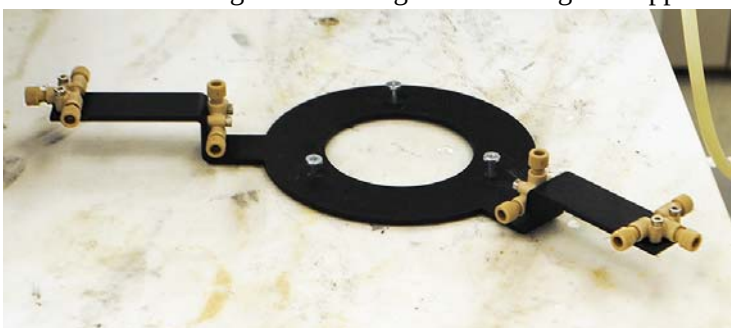

Figure 5 Tee fittings installed on the bracket.

6 Install the fitting bracket to the standoffs, orienting the bracket as shown.

Figure 6 Bracket installed.

7 Thoroughly inspect the mounting surfaces of the Smart Swap™ base and clean off any material that may interfere with mounting the lower accessory.

8 Install the Peltier Plate to the Smart Swap™ base.

9 Separate the Delrin trough from the aluminum base by removing the two M4 screws using the Allen wrench provided.

10 Thoroughly clean the Delrin trough. See "Maintaining the Cell" .

11 Install the flangeless nuts on one end of four of the precut lengths of PFA tubing. Then slide the ferrules onto the tubing. Make sure that the larger diameter end of the ferrule is flush with the end of the tubing

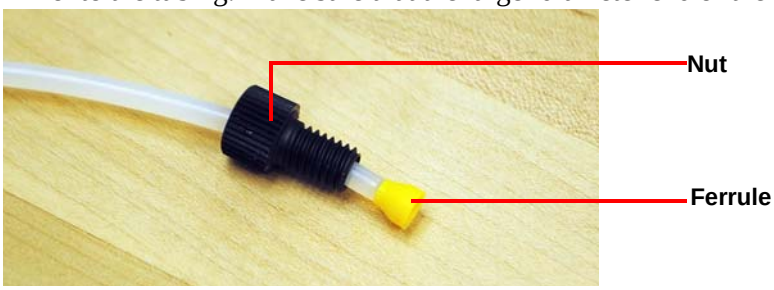

Figure 7 Flangeless nut and ferrule installed on precut tubing.

12 Connect the four lengths of tubing to the four bottom ports on the trough. Make sure these fittings are firmly hand-tightened prior to reinstalling the aluminum base. Note that the fittings must be secure to ensure a proper seal between the ferrules and the threads and prevent leaks from the trough.

Figure 8 Tubing connected to trough.

13 Align the M4 screw holes in the trough with the threads in the base. Guide the four infusion tubes through the slots in the aluminum base. Secure the trough and base with the two M4 screws

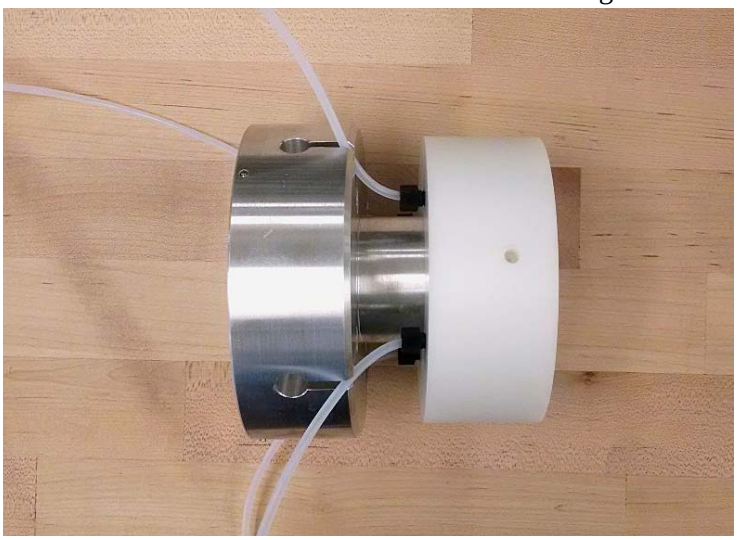

Figure 9 Tubing threaded through aluminum base.

14 Place the aluminum base onto the Peltier Plate, aligning it with the ball plungers on the Peltier Plate.

15 Remove the flangeless nuts and ferrules from two side ports of the infusion tees on the fitting bracket. Install the nuts and ferrules on the infusion tubes (similar to step 2). Connect the tubes to the infusion tees on the fitting bracket, taking care to hand-tighten the nuts to ensure a good seal and avoid leaks.

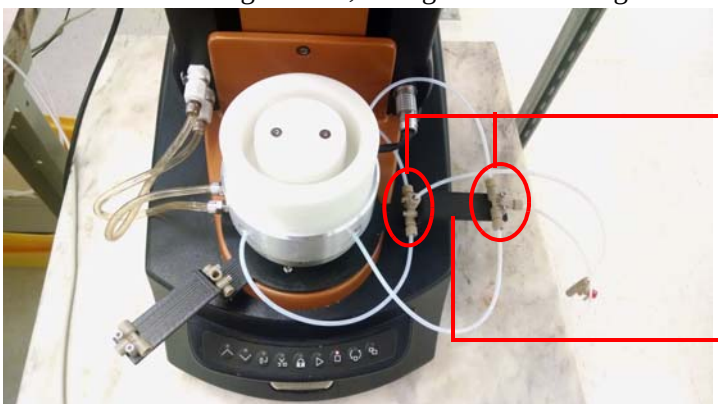

Figure 10 Fitting bracket and infusion line connections.

16 In a similar manner, connect the four withdraw tubes to the two withdraw tees on the left side of the fitting bracket.

Figure 11 Fitting bracket and withdraw line connections. Infusion lines not shown for clarity.

17 Install the flangeless nuts and the ferrules on the four withdraw tubes. Connect the free end of the withdraw tubes to the four top fittings of the trough. Once again, hand-tighten to ensure a firm seal.

Figure 12 Top fittings.

18 Place the syringe pump in a convenient location. Cut four equal length tubes to run from the infusion and withdraw tees to the syringe pump. Use the supplied tubing cutter to ensure the cut ends of the tubing are square.

NOTE: Tubing lengths from the Tee fittings to the syringe pump can vary depending on pump location. Make sure that both inlet and both outlet lengths are equal to ensure even infusion and withdraw.

19 Install the shut-off valves onto the four tubing lengths near the syringes. Connect the shut-off valves to the Luer adapters using a short length of tubing, flangeless nut, and ferrule. Connect the syringes to the Luer adapter.

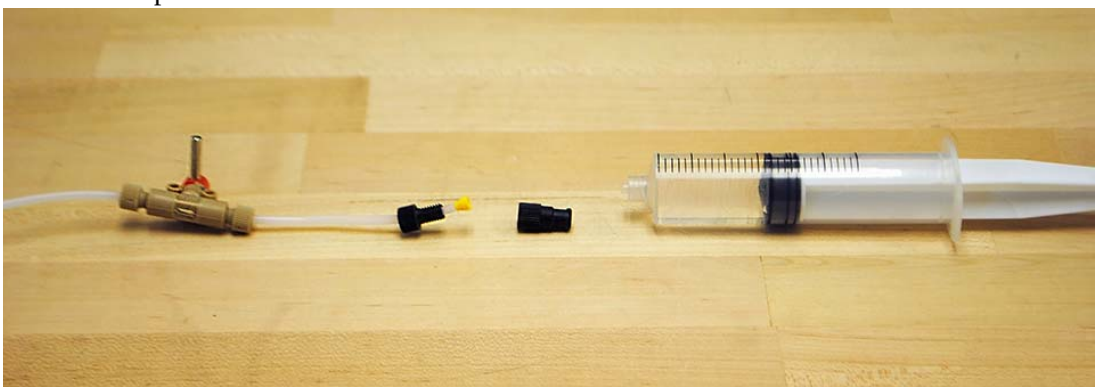

Figure 13 Tubing connection to syringe.

20 Install the upper DWR geometry.

Installing the Syringe Pump

A syringe pump is used to control the equal exchange of the subphase fluid. The TA Instruments supplied syringe pump (PN 271902.001) is fully integrated with TRIOS software and can be triggered within the experimental setup. You can also connect other model syringe pumps and trigger the subphase exchange manually, outside of TRIOS. The following setup steps are for the TA Instruments supplied syringe pump.

1 Connect the syringe pump to the PC with the supplied USB cable.

2 Connect power to the syringe pump and turn it on.

3 Windows will automatically detect the syringe pump, but will not install drivers automatically. Insert the CD supplied with the syringe pump into the PC.

4 Find the syringe pump under **Devices and Printers** in the Windows Start menu.

5 Right-click on the device and select **Properties**.

6 Select the **Hardware** tab, and click **Properties**.

7 Select the **Driver** tab, click **Update Driver**, then select **Browse my computer** for driver software. Navigate to the CD drive and select the **Pump Drivers** folder. Drivers will install automatically, and the pump will be operational.

8 Install the infusion and withdraw syringes into the pump. Make sure the syringe flange and plunger are secured in the retaining brackets. Refer to the syringe pump's manual for additional details

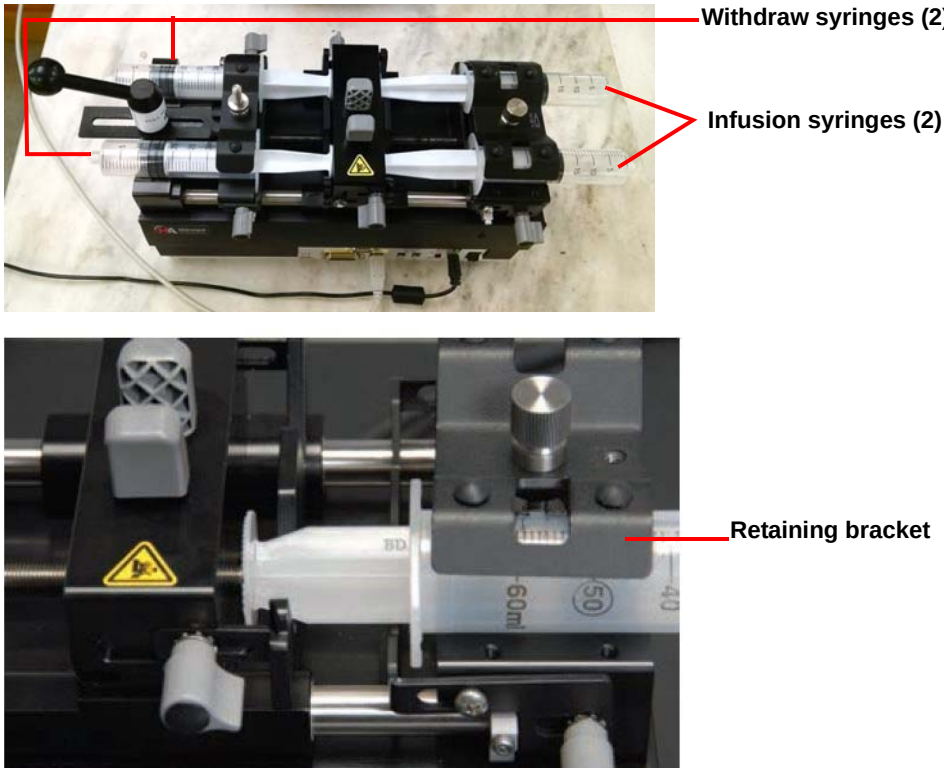

Figure 14 Syringes installed.

NOTE: Make sure all fittings are firmly hand-tightened.

Setting Up the Interfacial Exchange Cell in the Software

Before running an experiment, set up the Interfacial Pump in TRIOS software as follows:

1 Click **Options** > **Discovery HR** > **Interfacial Pump**.

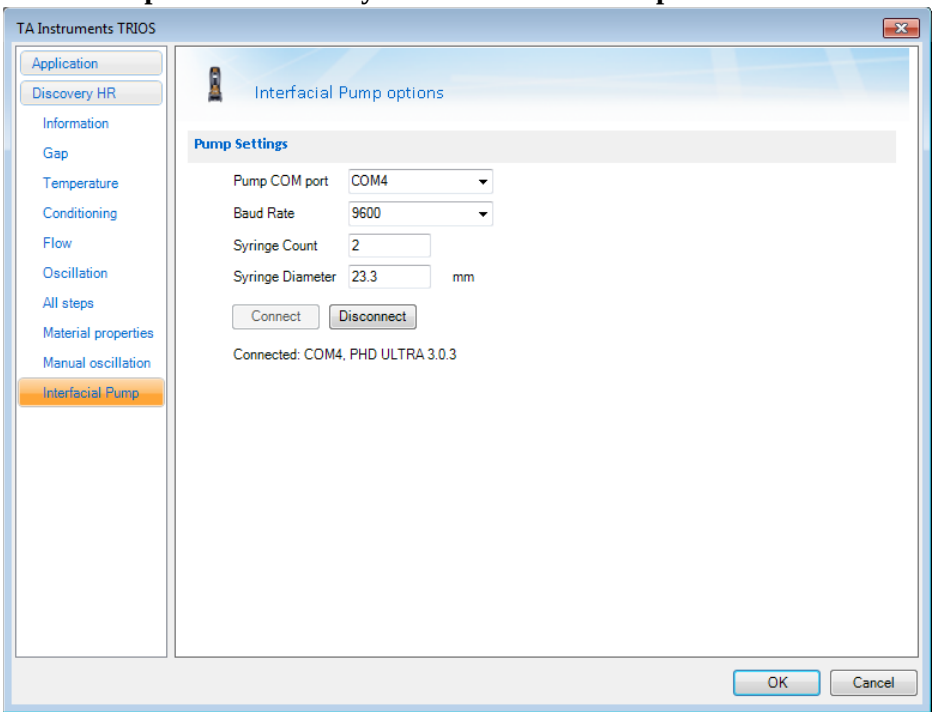

Figure 15 TRIOS Options.

2 Under **Pump Settings**, enter the syringe count and diameter information. Default values for the supplied syringes and TA Instruments supplied syringe pump are shown above in Figure 15.

Chapter 3:

Operating the Interfacial Exchange Cell

Priming Initial Subphase

NOTE: It is important to ensure all infusion and withdraw lines are free from air pockets and bubbles to allow even flow from all ports.

1 Make sure both infusion valves and both withdraw valves are closed. Disconnect the syringes from the Luer adapters.

2 Fill one syringe with the initial subphase fluid and connect to one infusion line. Open the infusion valve and manually inject the initial subphase through the infusion line until all air is removed. Take care to ensure that there are no bubbles visible in the infusion line by tapping the tubing. Close the valve on the infusion line.

3 Repeat step 2 for the second infusion line.

4 Overfill the trough with the initial subphase.

5 Connect a syringe to one of the withdraw lines. Open the valve and manually withdraw the initial subphase until all of the air is removed. Add fluid to the trough as needed to ensure that the fluid level does not fall below the withdraw ports or introduce air into the withdraw lines. Close the valve on the withdraw line.

6 Repeat this process for the second withdraw line. Add fluid to the trough as needed to ensure that the fluid level does not fall below the withdraw ports or introduce air into the withdraw lines.

7 Fill the infusion syringes with the exchange subphase and install them into the syringe pump. Briefly run the syringe pump from the Control panel in TRIOS (see Figure 14) until all air is pushed from the tip of the syringes and a few drops of fluid are pushed out. A flow rate of 10 mL/min is recommended for this step.

8 Briefly open each infusion line valve and allow the initial subphase to drip before quickly closing the valves. This will remove all the air at the end of the fittings and create a wet seal between the infusion syringe and the infusion lines.

Figure 12 Fluid dripping from the infusion line valve.

9 Connect the infusion lines to the infusion syringes.

10 Install the withdraw syringes into the syringe pump and connect the withdraw lines.

11 Finalize the initial subphase level at the step in the trough.

12 Carefully position the DWR at the subphase surface. The upper phase can now be added.

Running an Experiment

Creating an Experiment

To trigger the pump during an experiment, add an Interfacial Pump Conditioning Step.

1 From the Experiment setup page, expand the **Procedure** bar.

2 Click the arrow next to the displayed experimental parameters.

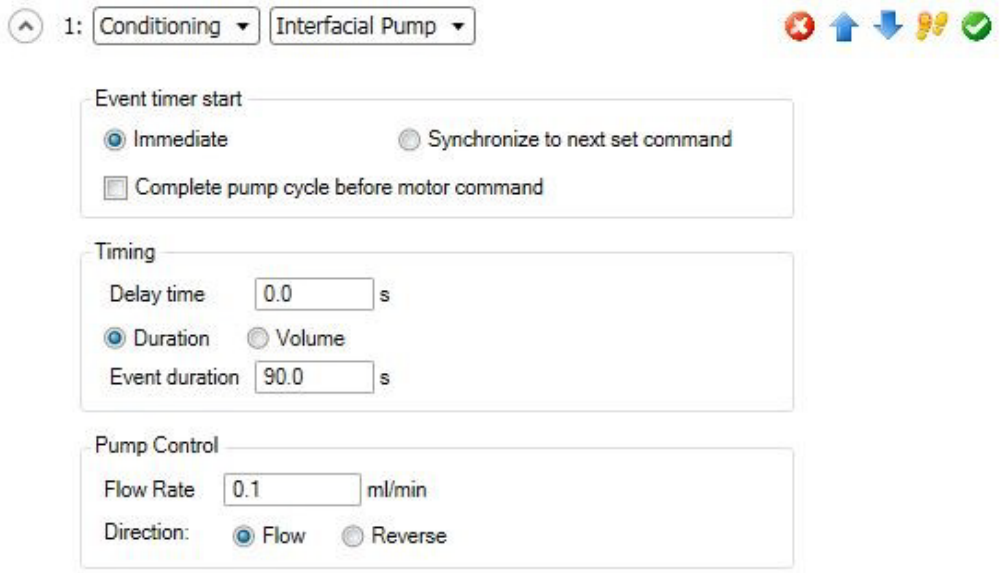

Figure 13 Experimental parameters.

3 In the Test Type drop-down menu, select **Conditioning**.

4 In the Test Mode drop-down menu, select **Interfacial Pump**.

5 Enter the desired experimental parameters.

Using the Control Panel

The syringe pump can be manually controlled outside of an experiment using the Control panel. Enter the Flow rate, Flow duration, and direction and then click **Start**.

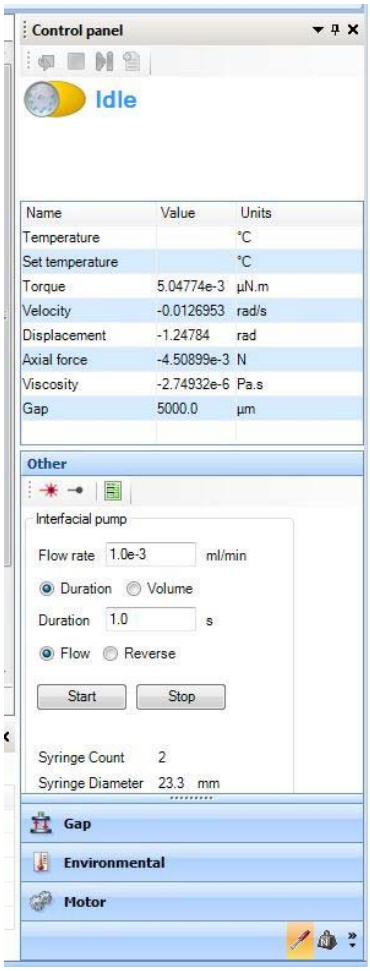

Figure 14 TRIOS Control panel.

Maintaining the Cell

Cleaning the Cell

It is recommended that you thoroughly clean the trough prior to testing. To clean the cell, remove the withdraw lines from the trough. Remove the two M4 screws on the trough and carefully guide the infusion lines to separate the trough from the aluminum base. Undo the infusion line nuts and remove the infusion lines. Plug all eight threaded ports using the supplied M6 set screws. Fill the trough with trichloromethane or other suitable solvent. Allow the solvent to sit in the trough. After a few minutes, remove the solvent and rinse with fresh solvent. After the solvent evaporates, rinse the trough thoroughly with DI water.

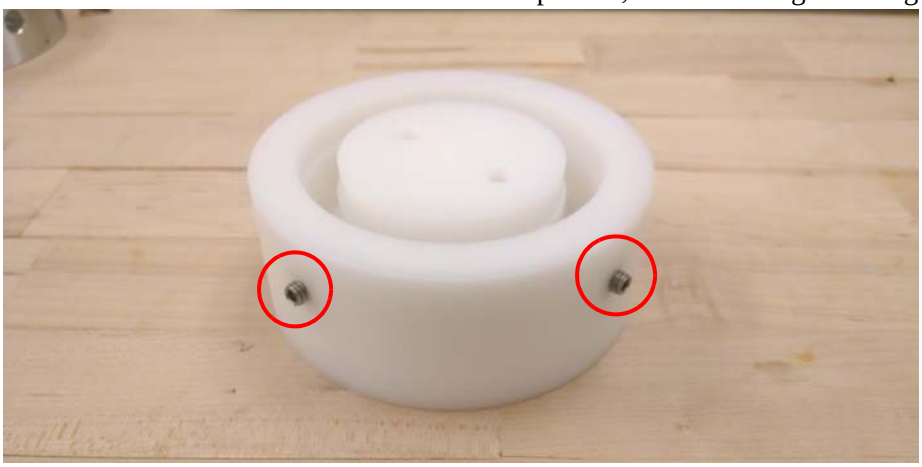

Figure 15 M4 screws.

After an experiment, remove all liquid from the cell with a pipette. Manually flush the infusion lines with a suitable liquid such as DI water and remove the flushed liquid from the cell. Disconnect the withdraw fittings from the Delrin trough. Disconnect the infusion lines from the tee fittings and remove the cell from the Peltier Plate. Disconnect all lines and thoroughly clean all components.

Replacement Parts

Table 2: Interfacial Subphase Exchange Cell Replacement Parts

Part Number	Description
271896.001	M6 flangeless nut
271895.001	Flangeless ferrule
271894.001	Tee fitting
271899.001	Shut off valve
271900.001	Luer adapter
271641.002	30 cc syringe
201688.002	1/8” teflon tubing
271901.001	M6 set screws