

**DynaLoop™ 25
and
DynaLoop 90**

**Instruction
Manual**

**Catalog Numbers
750-0451 (25 ml)
750-0452 (90 ml)**

BIO-RAD

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Section 1 Introduction

1.1 DynaLoop Sample Loop Description

The DynaLoop sample loop is a large-volume sample injection loop for use with the BioLogic DuoFlow™ system. Available in 25 ml and 90 ml sample capacities, the DynaLoop is used with the automated AVR7-3 injection valve.

1.2 Operation

The DynaLoop consists of five major components (Figure 1):

- Sample end assembly
- Buffer end assembly
- Glass tube graduated in 1 ml increments
- Sliding seal assembly
- Protective plastic jacket

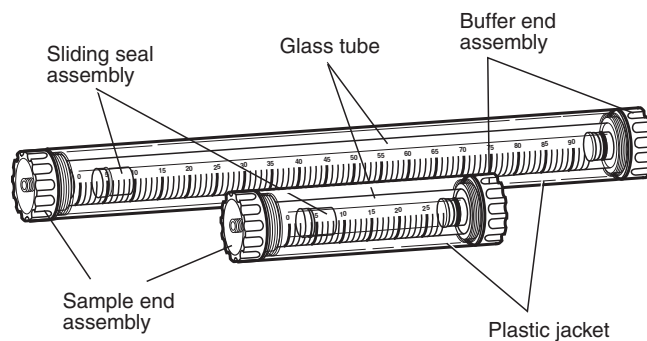


Fig. 1. DynaLoop 25 and DynaLoop 90 sample loops.

The sliding seal assembly divides the glass tube into two distinct chambers:

- Sample chamber
This end connects to port 3 on the AVR7-3 injection valve.
- Buffer chamber
This end connects to port 6 on the AVR7-3 injection valve.

The DynaLoop sample loop delivers sample to the column in a four-phase process:

- Load phase
- Injection phase
- Flush phase
- Reload phase

Load phase With the AVR7-3 injection valve in the LOAD position, sample is injected into the sample chamber end of the DynaLoop via port 3 on the injection valve using a peristaltic pump or a syringe. As the DynaLoop fills with sample, the sliding seal moves toward the buffer end assembly. The sliding seal O-ring and closed check valve within the sliding seal assembly prevent mixing of buffer and sample (Figure 2).

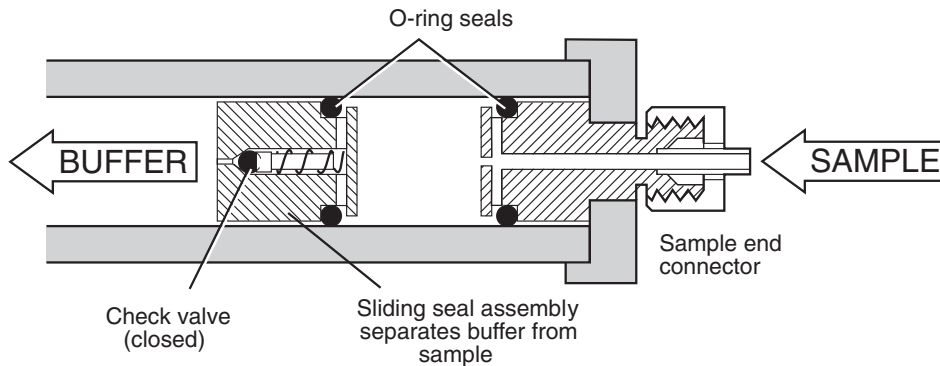


Fig. 2. Sliding seal assembly. (Check valve is closed.)

Injection phase With the AVR7-3 injection valve in the INJECT position, buffer delivered from the BioLogic gradient pump pushes against the sliding seal assembly, and delivers sample to the column. No buffer passes through the seal assembly into the sample chamber while the seal assembly is moving.

Flush phase When the sliding seal assembly contacts the sample end cap, the check valve opens and buffer passes through the sliding seal. Sample is completely flushed from the DynaLoop (Figure 3) onto the column in a concentrated band. When the sample is loaded onto the column and sufficient buffer has flushed all sample from the loop and check valve, the AVR7-3 injection valve will automatically switch to LOAD after the programmed sample load volume is injected. The gradient pump is now connected directly to the column, facilitating accurate gradient elutions.

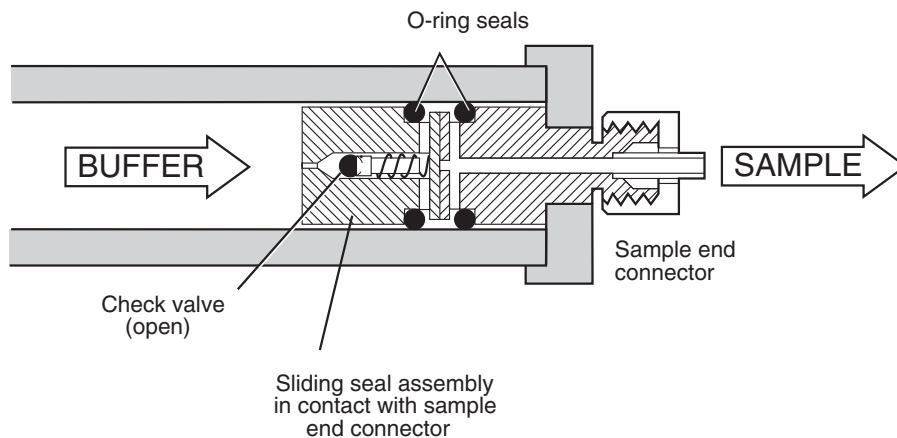


Fig. 3. Flush phase. (Check valve is open.)

Section 2 Installation

2.1 Unpacking

The DynaLoop is shipped fully assembled as shown in Figure 1.

List of components

The shipping carton contains:

- DynaLoop sample loop
- Fittings and tubing to make all necessary plumbing connections
- DynaLoop spare parts
- Instruction manual

2.2 Precautions and Recommendations

This section contains important information concerning the safe operation of this accessory. Please take a few minutes to review it and observe all of the cautions and warnings. Failure to do so can result in damage to the DynaLoop and/or the BioLogic DuoFlow system, and void the warranty.

Maximum Operating Pressure

- The maximum operating pressure of the DynaLoop is 1,000 psi (7 MPa, 70 atm). Set the pump high pressure limit to a pressure below 1,000 psi.
- The DynaLoop has an external plastic sleeve which protects the internal parts from accidental damage. Should the glass tube break, the shield will contain all glass fragments.

Warning: Know the physical properties of the solvents you use. Refer to the Material Safety Data Sheet that accompanies each solvent.

Important Considerations

- Always follow good laboratory practices when handling solvents.
- Use buffers and solvents that are chemically clean and free of particulates; purify and filter all buffers and solvents as necessary.
- The wetted materials of the DynaLoop are Tefzel, glass-filled Ryton (polyphenylene sulfide), PTFE, and PEEK (polyether-ether ketone). The check valve ball and O-rings, which are made from nitrile rubber (Buna-N), are compatible with alcohols, aqueous buffers, and dilute aqueous acids and bases, but will swell when exposed to solvents typically used for HPLC. If in doubt, test for solvent compatibility before using the DynaLoop. Immerse the components in a beaker of solvent overnight and examine for stability.
- The external sleeve is made from polycarbonate, which may lose some of its transparency if exposed to organic solvents or highly acidic or basic fluids.
- Be careful when changing from buffered aqueous solvents to organic solvents. Flush the system with Milli-Q or HPLC-grade water or another appropriate solvent before introducing an organic solvent. Otherwise, the buffer salts may precipitate and block the check valve or other parts of the unit.
- If the DynaLoop is not to be used for several days, remove buffers and store the loop in a 20% ethanol solution. For longer periods of storage, refer to Section 4.2, Cleaning and Storage.
- Do not kink, nick, or sharply bend the tubing. Bent or damaged tubing restricts flow and eventually causes tubing or fitting failure.

2.3 Installation

Overview

This section describes how to connect the DynaLoop to the BioLogic DuoFlow system with an AVR7-3 automated injection valve.

Auxiliary Pump Loading

An auxiliary peristaltic pump can be programmed to automatically fill the DynaLoop prior to sample injection.

General Instructions

Before using the DynaLoop for the first time, clean the unit to remove packing dust and debris. See Section 4.2, Cleaning and Storage, for directions on disassembling, cleaning, and reassembling the unit.

Preparation

1. Attach the DynaLoop to the BioLogic DuoFlow system. Measure the lengths of tubing required to make the desired connection. Allow enough slack so that the tubing is not pulled tightly around sharp corners.
2. Using the tubing cutter supplied with the BioLogic DuoFlow System Fittings kit, cut the tubing supplied with the DynaLoop to the desired length.

Make Connections

1. To make a collet/nut connection at the end of the DynaLoop, slide a collet/nut fitting and ferrule over the end of the cut tubing. Make sure the tapered end of the ferrule faces the tubing end. The blunt end of the ferrule must face the collet/nut (Figure 4).
2. Push the tubing all the way into the connector until the tube bottoms in the fitting.
3. While pressing the tubing securely into the fitting, slide the ferrule and collet/nut toward the DynaLoop and finger-tighten the collet/nut. Do not overtighten.

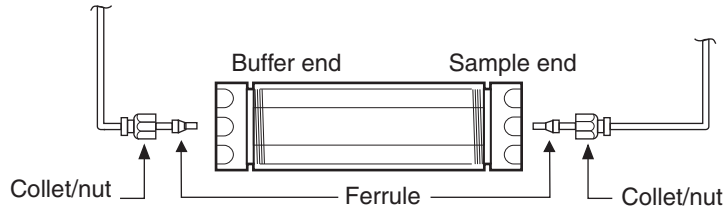


Fig. 4. DynaLoop plumbing connections.

Connection to AVR7-3 Injection Valve

There are two stages to installing the DynaLoop sample loop to a or AVR7-3 injection valve:

- Attaching BioLogic fittings to the DynaLoop
- Purging the DynaLoop of air

Connect Tubing

1. To attach the 1/8" 1/4-28 fittings to the loop for connection to the injection valve, slide a 1/4-28 nut onto the sample end tubing of the DynaLoop. Slide a yellow ferrule over the end of the tubing. The blunt end of the ferrule must face away from the nut (Figure 5). Repeat this procedure for the buffer end tubing.
2. Remove the sample loop currently connected to the injection valve.

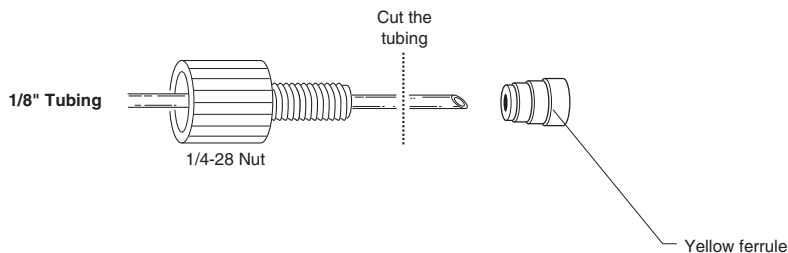


Fig. 5. Injection valve plumbing connections.

3. Connect the sample end of the DynaLoop to port 3 of the injection valve (Figure 6).
4. Connect the buffer end of the DynaLoop to port 6 of the injection valve (Figure 6).

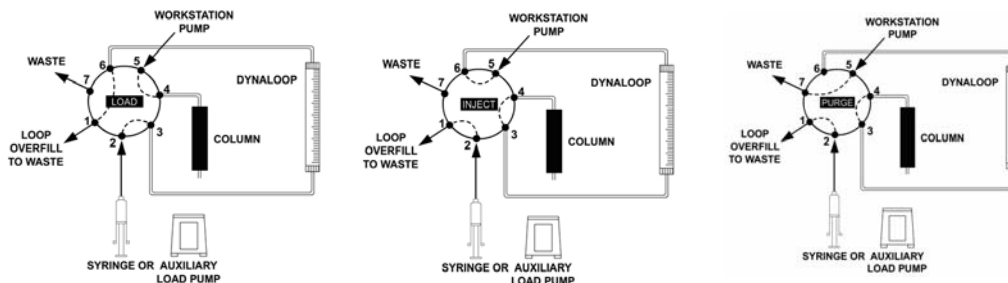


Fig. 6. Injection valve positions.

Purge Lines

1. Disconnect the tubing from the column at the column inlet (port 4) and redirect port 4 to a suitable waste container using a short piece of tubing.
2. Switch the injection valve to INJECT. Set the BioLogic DuoFlow pump flow rate to 10 ml/min. Verify that the DynaLoop fills with buffer.

Caution: The sliding seal must move toward the end of the DynaLoop designated as the sample end. If the seal moves toward the buffer end of the DynaLoop (the end with the white filter), stop the BioLogic DuoFlow pump immediately. The DynaLoop was installed backwards. Correct the plumbing configuration before using the DynaLoop.

3. Continue the flow until the DynaLoop is completely purged of air. Flow into the waste container should form a smooth stream free of bubbles. When the sliding seal assembly contacts the sample end cap, the check valve allows air and buffer to flow out through the DynaLoop sample delivery tube.
4. Use a syringe to deliver a couple of milliliters of buffer into the injection port (port 2) until it flows smoothly from the drain tube outlet (port 1) (Figure 6).
5. With the pump still running, switch the injection valve to LOAD. Allow the pump to run until the flow from the column connection tube makes a smooth stream free of bubbles into the waste container.
6. Stop the BioLogic DuoFlow pump.
7. Reconnect the column inlet tubing to the column. The system is now ready for use. Refer to Section 3, Using the DynaLoop, for applications information.

Peristaltic Pump Setup

There are three stages to installing the Bio-Rad® Model EP-1 Econo™ pump or the Econo gradient pump:

- Electrical connections
- Plumbing connections
- Purging lines of air

Electrical Connections (Model EP-1 Econo Pump)

1. Turn off the BioLogic DuoFlow controller and workstation.
2. Connect the EP-1 Econo pump to the BioLogic DuoFlow workstation using System Cable 7 (mini-DIN to breakout, catalog number 731-8267). Plug the mini-DIN end of System Cable 7 into the Econo pump rear connector labeled AUX. On the back of the BioLogic DuoFlow workstation, connect the red wire to the auxiliary connector 6 (AUX Pump) and connect the blue wire to the auxiliary connector 9 (GND) (Figure 7).

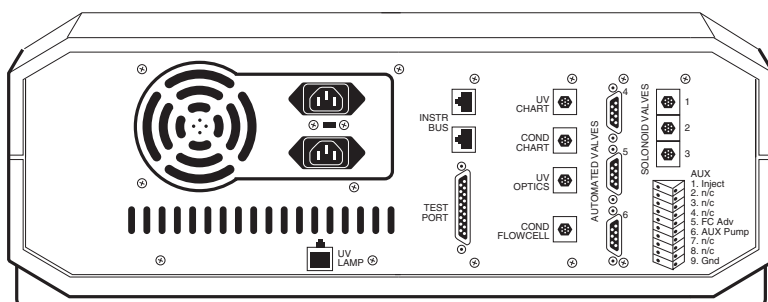


Fig. 7. Rear panel of BioLogic workstation and AUX connector.

Plumbing Connections

1. Use the fittings kit supplied with the EP-1 Econo pump and refer to the Econo pump instruction manual for all peristaltic plumbing connections.
2. To connect the outlet end of the peristaltic pump tubing to port 2 on the injection valve, use a luer to 1/4-28 adaptor (catalog number 732-0113).

Purge Lines

1. Disconnect the tubing from the column at the column inlet connection and redirect port 4 to a waste container.
2. Connect the EP-1 Econo pump inlet tube to a reservoir of buffer. Switch the injection valve to the INJECT position. Set the BioLogic DuoFlow flow rate to 10 ml/min. Verify that the DynaLoop fills with buffer.

Caution: The sliding seal must move toward the sample end of the DynaLoop. If the seal moves toward the buffer end of the DynaLoop (the end with the white filter), stop the pump immediately. The DynaLoop was installed backwards. Correct the plumbing configuration before using the DynaLoop.

3. Manually start the EP-1 Econo pump. Buffer should flow from the pump to the drain tube at port 1.
4. Continue running the BioLogic DuoFlow pump and the EP-1 Econo pump until the DynaLoop and all tubing associated with the Econo pump are completely purged of air. Flow into the waste containers should form a smooth stream free of bubbles. When the sliding seal assembly contacts the sample end cap, the check valve inside the seal will open allowing air and buffer to escape.
5. With both pumps still running, switch the injection valve to LOAD. Flow from the peristaltic pump will move the sliding seal towards the buffer end of the DynaLoop. Flow from the BioLogic DuoFlow pump will go through the injection valve directly to port 4 and then temporarily to waste.

6. Some air may still be trapped in the sample chamber of the DynaLoop. If so, switch the injection valve to the INJECT position and again empty the sample chamber of the DynaLoop to the column inlet waste container.
7. Continue to purge all the air from the system by switching between LOAD and INJECT as described in steps 5 and 6. Terminate this purging procedure with the valve in the LOAD position and with the sliding seal assembly in contact with sample end fitting. Stop the BioLogic DuoFlow and the EP-1 Econo pump. Reconnect port 4 to the inlet of the column. The system is now ready for use.

Electrical Connection (Econo Gradient Pump)

1. Turn off the BioLogic DuoFlow controller and workstation.
2. The Econo Gradient Pump (EGP) is connected to the DuoFlow workstation with bus communication cable (System cable 17, catalog number 750-0650) (Figure 8). For a complete discussion of the Econo Gradient Pump, refer to its separate instruction manual.

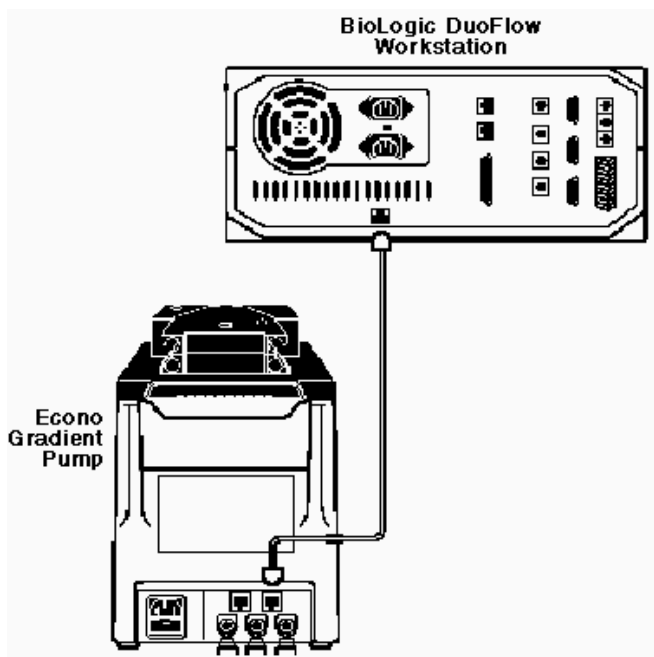


Fig. 8. Connecting the Econo Gradient Pump to the BioLogic DuoFlow workstation using System Cable 17.

Plumbing Connections

1. Use the fittings kit supplied with the Econo gradient pump and refer to the Econo gradient pump instruction manual for all peristaltic plumbing connections.
2. To connect the outlet end of the peristaltic pump tubing to port 2 on the injection valve, use a luer to 1/4-28 adaptor (catalog #732-0113).

Purge Lines

1. Disconnect the tubing from the column at the column inlet connection and redirect Port 4 to a waste container.

2. Connect the Econo gradient pump inlet tube to a reservoir of buffer. Switch the injection valve to the INJECT position. Set the BioLogic DuoFlow flow rate to 10 ml/min. Verify that the DynaLoop fills with buffer.

Caution: The sliding seal must move toward the sample end of the DynaLoop. If the seal moves toward the buffer end of the DynaLoop (the end with the white filter), stop the pump immediately. The DynaLoop was installed backwards. Correct the plumbing configuration before using the DynaLoop.

3. Manually start the Econo gradient pump. Buffer should flow to the drain tube at port 1.
4. Continue running the BioLogic DuoFlow pump and Econo gradient pump until the DynaLoop and all tubing associated with the Econo gradient pump are completely purged of air. Flow into the waste containers should form a smooth stream free of bubbles. When the sliding seal assembly contacts the sample end cap, the check valve inside the seal will open allowing air and buffer to escape.
5. With both pumps still running, switch the injection valve to LOAD. Flow from the peristaltic pump will move the sliding seal towards the buffer end of the DynaLoop. Flow from the BioLogic DuoFlow pump will go through the injection valve directly to port 4 and then temporarily to waste.
6. Some air may still be trapped in the sample chamber of the DynaLoop. If so, switch the injection valve to the INJECT position and again empty the sample chamber of the DynaLoop to the column inlet waste container.
7. Continue to purge all the air from the system by switching between LOAD and INJECT as described in steps 5 and 6. Terminate this purging procedure with the valve in the LOAD position and with the sliding seal assembly in contact with sample end fitting. Stop the BioLogic and the Econo gradient pump. Reconnect port 4 to the inlet of the column. The system is now ready for use.

Section 3

Using the DynaLoop

This section describes some techniques for using the DynaLoop.

3.1 Injecting Sample Volume

This section describes the procedures for:

- DynaLoop preparation
- Loading sample manually
- Loading sample automatically

Preparation

Purge air from all system components. To purge the DynaLoop quickly, disconnect the column inlet and set the BioLogic DuoFlow pump to a higher flow rate. Purge the system with the valve set to INJECT. Remember to stop the pump and reconnect the column after the system is completely purged.

Load Sample Manually (via a syringe)

1. With the injection valve in the INJECT position, allow the sliding seal to contact the sample end fitting. Set the valve to the LOAD position. Load sample into the DynaLoop by using a syringe at the filler port (port 2). Hint: use a 1/4-28 – Female Luer adaptor at port 2 (part no. 910-4159). As sample is loaded into the loop it displaces the buffer that was used to purge the unit.
2. When sufficient volume of sample for a run or series of runs is loaded into the DynaLoop, stop the filling process. For a series of partial volume injections after one loading sequence, fill the DynaLoop with about 2 percent extra sample.

Leave the syringe inserted in the injection port to minimize the introduction of air into the DynaLoop.

3. The sample is now ready for injection.

Load Sample Automatically

1. To load the sample automatically using an auxiliary pump such as the Model EP-1 or Econo gradient pump, you must select AUX Pump in the BioLogic setup editor.
2. In the Protocol screen, program an **Isocratic Flow** step that is long enough for the auxiliary pump to load the desired quantity of sample into the DynaLoop. For example, if you are loading 25 ml of sample at an auxiliary pump flow rate of 5 ml/min, you will need at least a 5-minute step prior to the sample loading step. Otherwise the protocol will fail the automatic validation done prior to the run.

The flow rate of the BioLogic DuoFlow pump is not critical, so you may set a low flow rate (e.g., 0.1 ml/min) to minimize buffer waste. It is important that either the time or volume length of this first step is of sufficient duration to allow the auxiliary pump to fill the DynaLoop.

3. In the Protocol screen, select **Load/Inject Sample** to program the BioLogic DuoFlow system to automatically fill and inject the DynaLoop sample. From the Load/Inject Sample window,
 - a. Select the **Dynamic Loop** as the type of loop to be used.
 - b. Select **Fill Before Inject**. This instructs the auxiliary pump to load the sample into the DynaLoop.

Note when using the EP-1:

The auxiliary pump flow rate is not under the control of the BioLogic DuoFlow. The flow rate is used by the system when validating the protocol before the run.

Because the BioLogic DuoFlow system only starts/stops the EP-1, the correct flow rate must be set at the EP-1. It is recorded in the yellow data entry boxes of the Fill Sample section of the Load/Inject Sample dialog box. Note that the rinse function is not available when the DynaLoop is being used.

Note when using the Econo gradient pump:

The auxiliary pump flow rate is under the control of the BioLogic DuoFlow. Note that the rinse function is not available when the DynaLoop is being used.

- c. In the Fill Sample Loop area of the window, select the sample to be loaded and enter its volume and the flow rate of the auxiliary pump.

- d. In the Inject Sample area of the window, select the Injection Buffers, the buffer composition, the flow rate of the BioLogic DuoFlow workstation pumps, and the sample volume to be injected onto the column.

The BioLogic DuoFlow system will now automatically control the loading and injection of the sample. Note that the rinse function is not available when the DynaLoop is being used.

4. Continue writing your desired separation protocol.

The AVR7-3 Injection Valve Function

During the run, the AVR7-3 injection valve functions as follows (Figure 6):

- Load. While in this position the valve connects ports 5 and 4 for equilibration of the column and for sample elution. In this position, sample loop is loaded to the desired volume via port 2 and buffer is expelled from the dynamic loop through port 1.
- Inject. While in this position the valve connects ports 5 and 6 and ports 3 and 4 for applying the sample onto the column. The flow from the workstation pump forces the sliding piston to expel the sample onto the column.
- Purge. While in this position the valve connects ports 5 and 7 and allows purging or buffer changes of the workstation pump without the need to remove the column from the system.

Section 4 Care and Maintenance

4.1 Troubleshooting

Table 1. Troubleshooting the DynaLoop

Problem	Possible Cause	Corrective Action
High backpressure when loading or injecting	Plugged filter	Replace filter (Section 4.3)
Leakage at the compression screw end fittings	Loose or worn ferrules	Tighten or replace ferrules
Leakage into the plastic shield	Worn O-rings on sample or buffer end	Replace O-rings (Section 4.4)

4.2 Cleaning and Storage

Refer to Figure 9 as you follow these instructions.

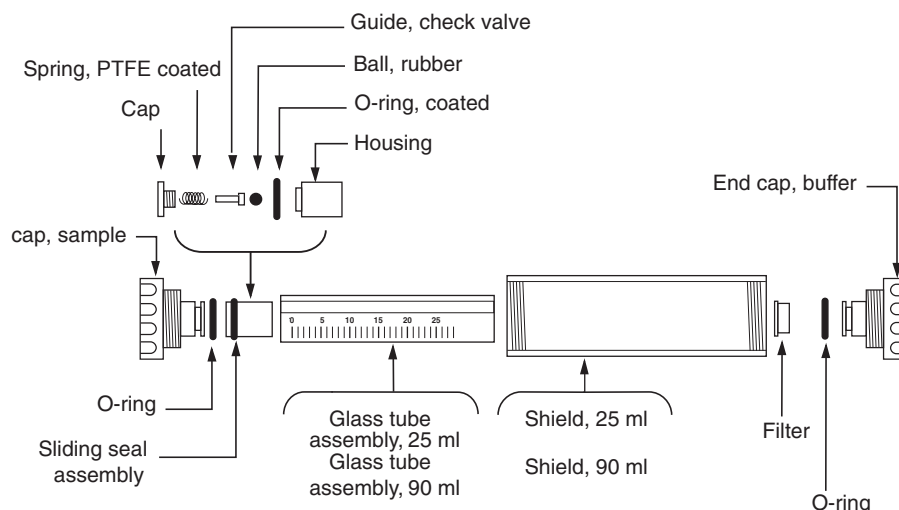


Fig. 9. DynaLoop cleaning.

Cleaning and Storage

With the DynaLoop in the fluid path, use the BioLogic DuoFlow pump to deliver the cleaning solutions to the DynaLoop.

1. Rinse with 1 volume of a dilute acid such as 0.1 M acetic acid.
2. Flush with 10 volumes of HPLC-grade water.
3. Rinse with 1 volume of 0.1 M sodium hydroxide. Flush immediately with 10 volumes of HPLC-grade. (Prolonged contact with sodium hydroxide will etch the glass tube.)
4. Rinse with a solution of 10% methanol and 90% HPLC-grade water and dry thoroughly before use.

For storage periods longer than a few days, store the unit disassembled at room temperature.

Disassembly

1. Turn off the BioLogic DuoFlow pump. Remove the DynaLoop from the fluid path.
2. Disassemble the unit by unscrewing the end caps and removing the glass tube.
3. Using a blunt plastic instrument, push the sliding seal assembly out of the glass tube.
4. To disassemble the sliding seal assembly, hold the body of the assembly in one hand and twist the cap that retains the O-ring counterclockwise. The check valve components (spring, guide, round rubber ball), and O-ring can now be removed and inspected.

Reassembly

1. Hold the body of the sliding seal assembly in one hand. Insert, in order, the rubber ball, the check valve guide, and spring into the hole in the center of the sliding seal housing. Place the O-ring in the groove at the top of the housing. Screw the retainer cap onto the body. (Ensure that the spring enters the large hole in the center of the cap.)
2. Insert the sliding seal assembly into the glass tube. The O-ring end of the sliding seal assembly must be oriented in the same direction as the sample end legend on the glass tube.

Caution: If the sliding seal assembly is inserted backwards, the DynaLoop can overpressurize and break when reconnected to the system.

3. Insert the sample end cap into the sample end of the tube. (The sample end cap does not have the white filter.)
4. Slide either end of the protective plastic jacket over the glass tube and screw it onto the sample end cap.
5. Insert the buffer end cap (with the white filter), into the glass column and thread the end cap into the plastic jacket. Hand tighten the assembly.

The DynaLoop is now ready for use. Be sure that the O-ring end of the sliding seal assembly is oriented towards the sample end of the unit. Reconnect the sample and buffer tubing, and purge air from the unit as described in Section 2.3, Installation.

4.3 Replacing the Filter

The filter is an assembly consisting of a filter and a distributor located at the buffer end cap assembly. Replace the filter periodically. If the filter is plugged, you will observe excessive pressures during loading and injection.

Removing Filter

1. Disassemble the unit and remove the old filter. If you cannot pull off the filter with your fingers, use a blade screwdriver. Lay the flat edge of the blade against the black plastic part. Rotate the blade slightly to get the corner of the blade under the white skirt of the filter cap. Pry off the filter by working the screwdriver in the gap between the filter and the plastic lip. Lever the screwdriver against the large white threaded part of the end cap, not the black plastic lip (see Figure 10).

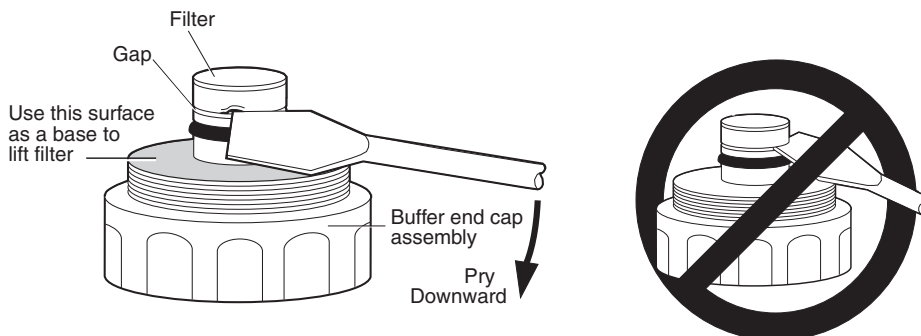


Fig. 10. Removing the filter.

Caution: Do not pry against the black plastic lip on the end fitting that holds the O-ring. The connector tip may break, causing the DynaLoop to leak.

Installing New Filter

1. Check that the new filter contains a distributor and snap the replacement filter onto the black connector tip.
2. Insert the buffer end cap into the glass column and thread the end cap into the plastic jacket by turning clockwise.

4.4 Replacing O-Rings

The DynaLoop contains three O-rings; one on each end cap assembly, and a third on the sliding seal housing. The O-rings on the buffer and sample connectors are identical. The O-ring on the sliding seal assembly is PTFE-coated to reduce friction.

End Cap O-Rings

1. To access the end cap O-rings, unscrew the end caps from the plastic shield and remove them from the glass column.
2. Carefully remove the O-rings from the connector tips. On the buffer end, remove the filter assembly first (see Section 4.3, Replacing the Filter).

Caution: Do not scratch the connector tip where the O-ring sits. If the connector tip is scratched, the DynaLoop will leak.

3. Slide a new O-ring over each end cap. Reinstall the filter on the buffer end cap.

Sliding Seal O-Ring

1. With the end caps removed, use a long blunt instrument to push the sliding seal from inside the glass tube.
2. Unscrew the cap at the O-ring end of the sliding seal housing.
3. Lift off the O-ring and replace it with a new one.
4. Screw the cap into the housing, making sure that the check valve spring enters the hole in the center of the cap.
5. Insert the sliding seal assembly into the glass tube making sure the O-ring end of the assembly is toward the sample end of the tube.

Caution: If the sliding seal assembly is inserted backwards, the DynaLoop can overpressurize and break when reconnected to the system.

6. Insert the sample end cap into the sample end of the tube. (The sample end cap does not have the filter.)
7. Slide either end of the protective plastic jacket over the glass tube and screw it onto the sample end cap.
8. Insert the buffer end cap (with the white filter), into the glass column and thread the end cap into the plastic jacket. Hand tighten the assembly.

Section 5

Appendix A Replacement Parts

Table 2. DynaLoop Replacement Parts

Product Description	Catalog Number
DynaLoop Parts Kit, includes end cap O-rings, sliding seal O-ring, filter, collet/nut fittings, 1/8" ferrules, 1/4-28 nuts and ferrules, and 1/8" PTFE tubing.	750-0450
Luer to BioLogic System Fittings Kit,	732-0113
FEP Tubing, 1/8" pre-pump connections, 15'	750-0603

Section 6

Appendix B Solvent Compatibility

The O-rings and check valve ball, which are made from nitrile rubber (Buna-N), are compatible with alcohols, aqueous buffers, and dilute aqueous acids and bases.

Typical HPLC solvents, such as dioxane, acetone, organic esters, ethers, dimethylformamide, tetrahydrofuran, and concentrated acids and bases, can swell the seal and check valve ball. Test for compatibility with questionable solvents by immersing these components in a beaker of solvent overnight. Then examine for stability.

Luer-Lok is a registered trademark of Becton, Dickson, and Company.

Milli-Q is a registered trademark of Millipore Corporation.

PEEK is a registered trademark of Victrex PLC.

Ryton is a registered trademark of Chevron Phillips Chemical Company LLC.

Tefzel is a registered trademark of the E.I. Du Pont de Nemours Co.



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