Hardware Manual
for the
Heated miniDAWN
Light Scattering Instrument

30 South La Patera Lane, B-7
Santa Barbara, CA 93117

M1200 Rev. B

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1 Introduction

This chapter introduces you to the Heated miniDAWN® laser light scattering photometer, the manual that describes it and available support options at Wyatt Technology Corporation (WTC).

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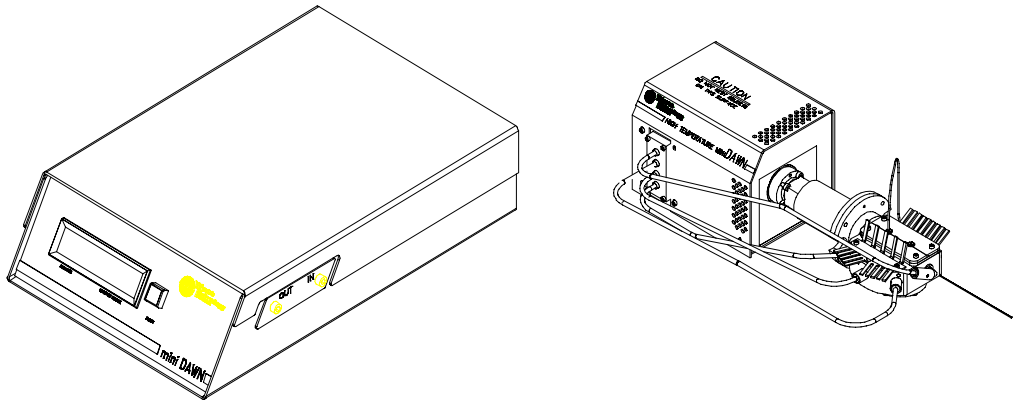
Overview

Figure 1-1: The miniDAWN electronics enclosure and optical bench assembly



The Instrument

The miniDAWN is a universal light scattering photometer for liquid chromatography. It can also be used for stand-alone batch measurements, often detecting aggregation instantly with less than 500 µL of sample.

The Heated miniDAWN has been designed for installation in a Waters 150C or a Polymer Lab's PL210 oven. The Heated miniDAWN consists of two major parts:

- **Electronics Enclosure:** this box contains the electrical components used to control the miniDAWN and interfaces to connect this box to the optical bench assembly and to your PC.
- **Optical Bench Assembly:** this assembly is installed inside the oven and is connected to your HPLC system. The read head anchors to the laser diode, and the flow cell and manifolds bolt directly into the read head to provide a stable optical bench. It can be run at temperatures up to 150 degrees C.

The miniDAWN can determine absolute molar masses from 1000 to over a million g/mol (daltons), and root mean square (*rms*) radii from 10 nm to over 50 nm. It is an excellent tool in such areas as polymer chemistry, protein purification, and process and quality control.

The laser beam is aimed into the flow cell, and passes in the same direction as the flowing stream. The windows that let light pass through the flow cell are recessed in the manifolds, minimizing sample volumes and stray light. The three discrete photodetectors spaced around the flow cell enable simultaneous measurements over a range of angles (for example, with TCB in the cell the range is 48°–132°).

The analog signal from each photodetector is processed by its own DSP (Digital Signal Processor) integrated circuit with 16-bit digital conversion for high signal resolution. In addition, two auxiliary analog inputs (with their own DSP chips) enable interfacing to external instruments such as

differential refractive index and ultra-violet detectors or differential viscometers. A six-pole Gaussian filter in the DSP chip processes each light scattering and auxiliary signal for the greatest possible noise rejection without peak distortion. Since the analog-to-digital conversion is performed on-board the miniDAWN, low light scattering signals are not prone to environmental "noise" or pickup. The digital output transmits to your computer through the RS-232 serial port.

The Software

Wyatt Technology offers the following software package for collecting and analyzing data from the miniDAWN instrument:

- **ASTRA® for Windows**
  ASTRA for Windows collects and processes light scattering data as a function of time. From polymers fractionated by size or molar mass, ASTRA calculates the molar mass moments (number, weight and z-average) along with the rms radius moments of the molecules in solution. A concentration sensitive detector is needed for the molar mass calculations.

About This Manual

The *Heated miniDAWN Hardware Manual* describes how to set up and use the Heated miniDAWN laser photometer. Because the DAWN software is used for stand-alone applications and ASTRA primarily for HPLC, you need to consult their respective user's manuals for guidelines in sample preparation and auxiliary hardware setup.

Manual Conventions

Whenever we point out internal components and their location, we assume you are facing the front of the instrument.

Often you can use either a hex wrench (Allen wrench) or a Ball driver for the same task, in which case we will refer to the tool as a *hex wrench*.

The IUPAC Definition Committee specifies the term *molar mass* for the sum of the atomic weights of all atoms in a mole of a molecule. This term can be used interchangeably with *molecular weight*.

How the Manual Is Organized

The chapters and appendices in the *Heated miniDAWN Hardware Manual* are organized as follows:

Chapter 1—Introduction - gives a general overview of the miniDAWN instrument and this manual, and describes the support options available from Wyatt Technology.

Chapter 2—Setup - supplies the steps necessary for unpacking, connecting and testing the miniDAWN.

Chapter 3—miniDAWN Components - takes you on a guided tour of the miniDAWN's exterior and interior.

Chapter 4—Maintenance - gives general maintenance guidelines and the procedure for cleaning the flow cell.

Chapter 5—System Startup and Shutdown - tells you how to connect the fluid lines and heat up the system to its operating temperature. It also tells you how to cool it down for servicing of the 150C/PL210.

Appendix A—Accessories describes recommended accessories for various light scattering applications.

How to Contact Wyatt Technology Corporation

If you have a question about your miniDAWN, first look in this manual or consult the online help that comes with your ASTRA software. If you cannot find an answer, please contact Wyatt Technology Technical Support.

Corporate Headquarters

Wyatt Technology Corporation
30 South La Patera Lane, B-7
Santa Barbara, CA 93117
USA

Sales Department

Wyatt Technology Corporation Sales Hours are 8:30 A.M. to 5:00 P.M. Pacific Time.

Sales Phone: (805) 681-9009

Sales Fax: (805) 681-0123

Technical Support

Wyatt Technology Corporation offers a variety of support options to help you get the most from your miniDAWN.

For users outside the United States, the Wyatt Technology distributor should be contacted for assistance.

Before contacting technical support, try to resolve any problems through the ASTRA® for Windows on-line help system and this manual.

Internet

Visit Wyatt Technology's world-wide-web site to e-mail requests for assistance.

World-Wide-Web URL: http://www.wyatt.com

Electronic mail address: support@wyatt.com

FAX

Please fill in the answers to the items listed on page 1-6, and fax it to:

Wyatt Technology Corporation Technical Support Fax: (805) 681-0123

Questions or comments may be faxed at any time.

Mail

Please fill in the answers to the items listed on page 1-6, then mail it to our corporate headquarters.

Telephone

To speak to our support personnel directly, please call between 8:30 A.M. and 5:00 P.M. Pacific Time, Monday through Friday. Calls made during non-business hours will be handled by a voice mail system. When the call is placed please at the instrument and have the documentation at hand if possible. Please be prepared to provide the following information:

- The miniDAWN instrument serial number (located on the back panel).

If the problem is software related:

- The operating system name and version number.
- The WTC software version number. The software version number is located on the original distribution diskettes, or can be determined by selecting **About** from the software Help menu.
- The exact wording of any messages that appeared on the computer screen.

Then, for either a hardware or software problem:

- The type of computer hardware being used.
- Details of what was happening when the problem occurred.
- Efforts attempted to solve the problem.

Wyatt Technology Corporation Technical Support Phone Number:

(805) 681-9009

2 Installing the miniDAWN

This chapter guides you through unpacking the miniDAWN and the setting up and testing of the instrument.

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Unpacking the Instrument

This section lists the accessories and spare parts that were shipped with the instrument. Please read this section and check that everything has arrived in good condition.

1. Carefully examine the shipping container. If it has been mishandled, CONTACT THE SHIPPING CARRIER IMMEDIATELY.
2. Unpack the instrument.
3. Check that the boxes contain three kits together with the following items:

Table 2-1: Parts List

	Part Number
miniDAWN electronics enclosure	
miniDAWN optical bench assembly	
Documentation:	
This Heated miniDAWN Hardware Manual	
Software disks and manuals	
Cables:	
Interface cable adapter	P3796-0925
RS-232 interface cable	P4045-03
Main Power Cord	P4100-120 (or 220)
Auxiliary Input/Output Cable	117100
DB25 Interface Cable	124002
Fuses:	
Spare fuses	3616-00500 (or 3616-00250)
Fluid Fittings and Tubing:	
2 Flow cell windows	116007
2 Window O-rings	P6504-2004
2 Flow cell O-rings	P6504-2009
2 Backing rings	200609
6 Stainless Steel Fittings and Ferrules	P6406-10/P6455-10
Tubing, inlet (pre-bent, 0.005” ID)	P6600-005040
Tools:	
1.5 mm Ball driver	(9004
2.5 mm Ball driver	P9005
4.0 mm Ball driver	P9008
Jeweler's loupe	P8404
Anti-static wrist strap	P9012

	Part Number
Lens Tissue	P8052
Standards Kit	P8401
Installation Tool Kit	See packing list
Final Assembly Kit	See packing list

4. Place the miniDAWN on a level surface and inspect the instrument. If any damage is apparent, CONTACT THE CARRIER IMMEDIATELY.

Installing the Instrument

The initial installation of the Heated miniDAWN should be performed by installation personnel from Wyatt Technology. If you deinstall the Heated miniDAWN as described in “Deinstalling for Service” on page 4-13, you may reinstall it using the procedure in “Installing the Optical Bench Assembly” on page 2-5.

Preparing the Oven

To prepare the 150C oven for the Heated miniDAWN installation, several holes must be drilled in the side of the oven. This procedure should be performed by installation personnel from Wyatt Technology. The procedure is described here so that you will understand the changes to be made to the oven. Before the holes are drilled, the columns, the RI detector, and the oven thermocouple may have to be removed.

Note: The PL210 oven is prepared by Polymer laboratories.

To drill the proper holes in the oven, you will need the following materials most of which you will find in the Installation Tool Kit:

- Positioning template
- Gluestick
- 1/8" centerdrill
- 1/8" Cobalt steel twist drill (for drilling pilot holes)
- #29 Cobalt twist drill (for self tapping screws)
- 1/4" Cobalt steel twist drill (as guide for the hole saw)
- 1 7/8" Carbide tipped hole saw
- Small hand drill (brought by installer)
- Spring loaded centerpunch
- Round end file
- Shop vacuum machine (not provided)

To prepare the oven, do the following:

1. Remove the lid flange from the left edge of the oven wall.

   Unscrew the three mounting screws, then prop open the oven compartment by wedging two of the blue stoppers into the oven hinge.

2. Cut the template to shape and place it over the edge of the oven wall.

   The template should be on the inside of the left side wall of the oven. Before final positioning, be sure that the when the drill is pressed against the oven wall, there is enough room to drill the holes.

3. Affix the template to the oven wall.

Apply the gluestick to the back of the template and press it firmly in place. The glue holds the template securely in place and allows it to be easily removed after the holes are drilled (the glue is water soluble).

4. Cover the inside of the oven with paper or foil to catch the chips drilled out from the wall.
5. Use the centerpunch to create a small dimple at the center of each of the ten indicated hole positions on the inside of the oven.

   There are eight for the self tapping screws and one at the center of each of the large holes.
6. Use the center drill to start each of the holes at each of the dimples.

   Be careful not to let the drill wander as the hole is started. Press the drill firmly and let the drill bit cool after each hole. Minor wander may be corrected afterwards by using the round end file to shape the hole until it is correctly positioned relative to the template.
7. Drill through the inside wall only using the 1/8" drill.
8. Using the 1/4" drill, extend the hole at the center of the two large holes.

   Work from the inside out and drill all the way through the oven wall. Take care to insure that this hole is perpendicular to the wall.
9. Drill with the hole saw from the inside of the oven through the inner two layers of the stainless steel and the associated insulation.

   Use the existing 1/4" pilot hole as a guide. At each step, stop and remove any chips captured by the saw. Allow the saw to cool between steps.
10. Repeat step 9 from the outside of the oven.

    It may be necessary to clean up any rough edges with either a small file, sandpaper, or a Dremel tool.
11. Drill the eight holes on the inside oven wall for the self-tapping screws using the #29 twist drill.
12. Remove the template from the oven wall.

    Any excess glue can be removed with water.
13. Replace the lid flange.

Installing the Optical Bench Assembly

During the initial installation, Wyatt Technology will install the optical bench assembly in the 150C or PL210 oven. If you later deinstall the Heated miniDAWN as described in “Deinstalling for Service” on page 4-13.

You will need the following materials and tools:

- 1.5 mm Ball driver
- 2.5 mm Ball driver

- 4 mm Allen wrench (ground down)
- ¼" socket driver
- 8 #8x1" self-tapping screws
- electrical tape
- aluminum foil
- laboratory jack
- top Heat Sink
- 3 M3 screws
- heat sink compound
- 2 hollow foam stoppers
- vacuum grease
- stainless steel Retaining Plate
- exterior Foam Clamp
- Dress Plate

To install the optical bench assembly, do the following:

1. Remove all four optical fibers from the read head.

   First remove the Top heat sink and the Read head cover plate using the 2.5 mm Ball driver (if they are installed, see Figure 2-1 and Figure 3-5). Loosen the four set screws using the 1.5 mm Ball driver and pull each fiber out of the Read head. Do not remove the fibers from their respective photodiodes.

2. Cover all exposed holes in the read head with electrical tape to keep dust from entering the read head.

   Also cover the ends of the optical fibers using aluminum foil securing the foil with tape.

3. Disconnect the Post Assembly from the Optical Chassis Assembly (Figure 2-1 and Figure 3-3).

   Use the 4 mm Allen wrench to unscrew the four cap screws holding the precision flanges together and separate the flanges (do not loosen the flange o-ring, Figure 2-1). Note that the read head and post assemblies should still be connected.

4. Cover the two exposed flange surfaces using aluminum foil securing the foil with electrical tape to keep the flanges clean.

5. Lower the Read Head/Post Assembly into the oven and attach the assembly to the oven wall using four self tapping screws and the socket driver (Figure 2-1).

   Carefully slide the post assembly end through the oven wall before attaching it.

6. Place the Optical Chassis Assembly on a laboratory jack and raise it until its flange matches the Post Assembly flange.

7. Insert the optical fibers through the hole in the oven.

   The order of the fibers from top to bottom is F, 1, 2, 3 (Figure 2-1). Insert them with the F fiber on top, the #3 fiber towards the front, the #2 fiber on the bottom, and the #1 fiber towards the rear of the instrument.

8. Reattach the Post Assembly to the Optical chassis assembly using the four cap screws and the 4 mm Allen wrench.

   First remove the aluminum foil. Make sure the flange o-ring is in place.

9. Install the fibers in the read head.

   First remove the foil from the fibers and the tape from the read head. Lock the fibers into place using the set screws and the 1.5 mm Ball driver.

10. Install the top Heat sink (Figure 2-1) using three M3 screws and the 2.5 mm Ball driver.

    First spread a thin layer of heat sink compound on the bottom of the Heat sink. The holes are the same as for the set screws.

11. Install the hollow Foam stoppers around the optical fibers to block air passage through the wall (Figure 2-1).

    First apply vacuum grease to the inside of the Foam stoppers, then wrap one of them around the external part of the fibers and the other one around the internal part of the fibers with the tapered ends facing the wall. Push both Foam stoppers into the hole in the wall making sure that the internal one is flush with the inside wall.

12. Install the stainless steel Retaining plate (Figure 2-1) over the internal Foam stopper securing it with two self-tapping screws using the socket driver.

13. Install the exterior Foam clamp (Figure 2-1) over the external Foam stopper securing it with two self-tapping screws using the socket driver.

    First split the two halves of the clamp.

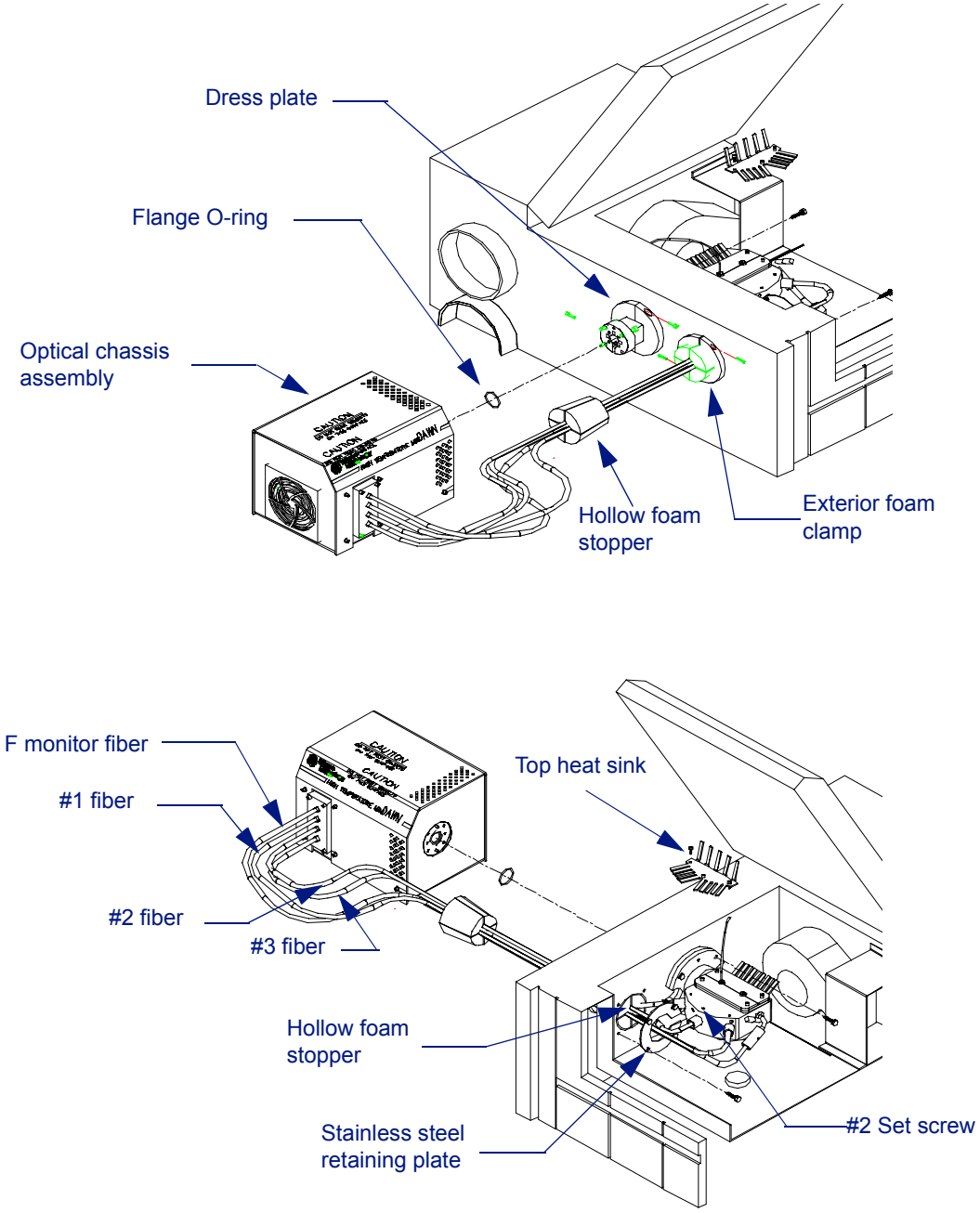
14. Install the Dress plate (Figure 2-1) over the external part of the Post assembly securing it using the 1.5 mm Ball driver.

    First split the two halves of the plate.

You should now reinstall the columns, the RI detector and the oven thermocouple which were removed initially, before drilling the oven. Also connect the column outlet to the Heat exchanger inlet line (Figure 3-5).

See “System Startup and Shutdown” on page 5-1 for further information about how to connect the fluid lines and heat up the oven to its operating temperature, but first read “Installing and Connecting the Electronics Enclosure” on page 2-8.

Figure 2-1: Outside and Inside views of the optical bench assembly during installation



Installing and Connecting the Electronics Enclosure

The installation procedure for the miniDAWN involves some initial tests to verify that everything is working properly. These test should be done while the oven of the 150C/PL210 is still at room temperature.

To install and connect the miniDAWN electronics enclosure, do the following:

1. Plug the power cord into its connector on the back panel, the other end to an AC outlet.

   Make sure the ON/OFF switch (Figure 3-1) is in the off position.

2. Connect one end of the DB25 Interface cable to its port on the back panel of the Electronics enclosure, the other end to its port on the Optical chassis assembly.

   Put on the anti-static wrist strap before performing this procedure. Remove the Shorting connector from the Optical chassis assembly before connecting the Interface cable.

3. Connect one end of the RS-232 serial interface cable to its port on the instrument's back panel, the other end to the computer's serial port.

   This is the 9 pin interface cable that connects the SERIAL DATA OUT connector on the back panel of the miniDAWN (see Figure 3-2) to a serial port on the back panel of your computer. We recommend COM2 for most systems.

Note: If you use an interface cable other than the one supplied by WTC, make sure it is a 9 pin RS232 serial cable with all 9 wires wired straight through.

4. Switch on the electronics enclosure and let it warm up for 30 minutes before proceeding to the next step.

   The main power switch is on the back panel, next to the power connector.

5. Press the SELECT button to switch the channels on the front panel display, and compare the voltages with the dark offsets in the miniDAWN Certificate of Performance.

Channel Setting	Value Displayed
1, 2, 3	Detector 1, 2, or 3 voltage
F	Forward laser monitor voltage

   If your voltage readings for detectors 1, 2 and 3 are different from the Certificate of Performance, check the temperature in your laboratory. The dark offsets on the detectors may differ from the Certificate of Performance by as much as 10 mV per ×C. For example, if your laboratory temperature is at 20×C and the QC laboratory temperature was at 23×C, your current dark offsets may be 30 mV different. If you see a greater difference, contact Wyatt Technology Technical Support.

   The forward laser monitor is set to 0mV at the factory and should be within ±1 mV.

6. Using the supplied WTC software (ASTRA) perform the appropriate steps to configure the instrument to communicate with the software (see the appropriate software user's guide for instructions to configure communication with the instrument.)

7. With the laser turned on, wait at least 30 minutes for the laser to warm up and stabilize.

Note: The laser in the miniDAWN is software controlled and will automatically be turned on by the software once the communications have been established with the instrument.

8. Compare the channel values in the software with the solvent offsets listed on the Certificate of Performance.

Note: The LM value is only available while viewing the numeric real-time channel values in the software (press the View button in the Collect/ Instrument dialog, see the appropriate software user's guide.) The F value is only available on the front panel.

The solvent offsets were measured at the factory with toluene, and the flow cell was filled with toluene and sealed before shipment, so the solvent offsets you are viewing should be very close to those on your Certificate of Performance. More than 20 mV difference between these values and those on the Certificate of Performance (for channels 1, 2, and 3) may indicate air bubbles in the manifolds, in which case you will need to flush the cell with filtered toluene and recheck the solvent offsets. If your dark offsets were different from the Certificate of Performance (see step 5) the solvent offsets should be off by the same amount.

9. Calibrate the miniDAWN using the solvent shipped in the cell (toluene) and the WTC software (ASTRA).

   See the appropriate user's guide for instructions on how to perform the calibration measurement.

10. Compare your calibration result with the value from the Certificate of Performance.

    Your calibration result should be within 5% of the value on the Certificate of Performance.

11. When you have confirmed that the instrument is in good working order, connect the auxiliary devices (described next.)

12. Keep the miniDAWN Electronics Enclosure on a flat, clean surface, standing on its feet and positioned to allow air convection through the back of the instrument to keep its electronics cool.

    See "Maintenance" on page 4-1 for more information about how to keep your miniDAWN in peak condition. System startup is described in "System Startup and Shutdown" on page 5-1.

Auxiliary Devices

The Auxiliary connector is used in a chromatography setup. It has four cables for different applications:

- You must connect one concentration-sensitive detector (usually the DRI inside the 150C/PL210). The red and white wires of the AUX1 cable connects to the integrator output on the side panel.
- You must use the AUTO INJECT cable to sense an injection from the auto injector in the 150C/PL210. The red and black wires from the cable connect to the closure switch on the side panel. This switch should be normally open.
- You can connect the 90 DEG. cable to your existing data collection system or a chart recorder for recording the 90° output signal.

Attaching the Auxiliary Connector

To attach the Auxiliary connector and its cables, do the following:

1. Attach the Auxiliary connector to the AUXILIARY input/output port on the rear panel of the miniDAWN (see Figure 3-2).
2. Connect the appropriate cable to your other device(s) as follows:
   a. Red wire to the positive terminal.
   b. White wire to the negative terminal.
   c. Black wire to the ground terminal (usually not needed).
   d. Clear wire to the shield terminal (usually not needed).
3. If you are connecting the AUX1 or AUX2 cable to a concentration detector and experience electronic drift or noise, you may try connecting the ground wire and/or the shield wire.
4. If you are connecting the AUTO INJECT cable to an auto injector, make sure that an injection closes the circuit.

   You can monitor the status of the auto inject circuit in ASTRA. Some injectors require programming for this closure to happen.

Adjusting the Auxiliary Gain

The miniDAWN is shipped with an AUX gain setting of 1¥, which can be adjusted via the DIP switch on the main PCB.

To adjust the gain settings:

1. Remove the miniDAWN top cover.
   a. Disconnect the power cord.
   b. Using a 2.5 mm Ball driver, remove the two screws at the top of the back panel.
   c. Lift the back end of the cover, slide the cover back ½" and then lift it off.

2. Locate the DIP switch at the front left corner of the main PCB).

   The switch settings should all be "ON" for a 1× gain.

3. Set the switch according to Table 2-2.

   We recommend a gain of 10x for most refractometers with a full scale output of 1V. The gain adjustments for AUX1 and AUX2 are independent of one another, so, for example, you could set the gain at 10 for AUX1 and at 1 for AUX2.

Table 2-2: AUX gain settings

	AUX1	
GAIN	**SW1**	**SW2**
1	ON	ON
10	ON	OFF
100	OFF	ON
1000	OFF	OFF

	AUX2	
GAIN	**SW3**	**SW4**
1	ON	ON
10	ON	OFF
100	OFF	ON
1000	OFF	OFF

3 miniDAWN Components

This chapter shows you the interior and exterior of the miniDAWN. Read it before making any measurements to familiarize yourself with the various parts and their functions.

Under normal operating conditions you should not need access to the internal components other than to remove the flow cell for cleaning or set the AUX gains.

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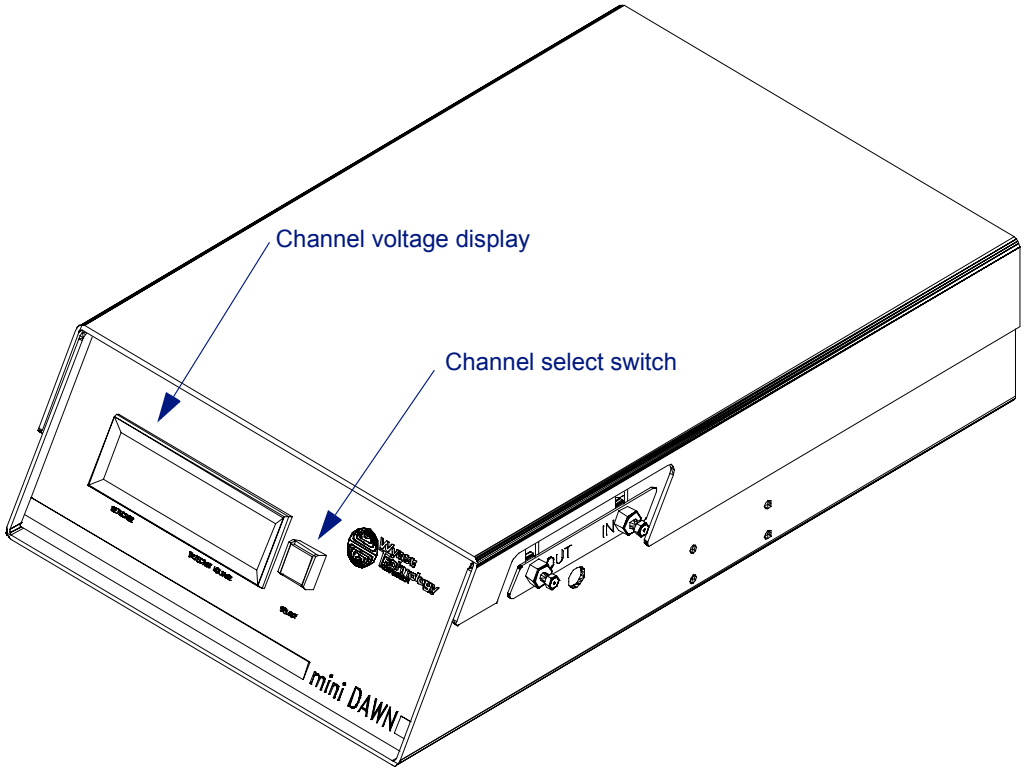
Electronics Enclosure Front Panel

The front panel of the electronics enclosure contains the channel voltage LED display and the SELECT button for switching channels. As you press SELECT, the voltage displays the selected channel.

Table 3-1: Channel types

Channel Number	Value Displayed
1, 2, 3	Detectors 1, 2 or 3 voltage
A1, A2	Auxiliary detectors 1 or 2 voltage
F	Forward laser monitor voltage

Figure 3-1: Electronics enclosure front panel

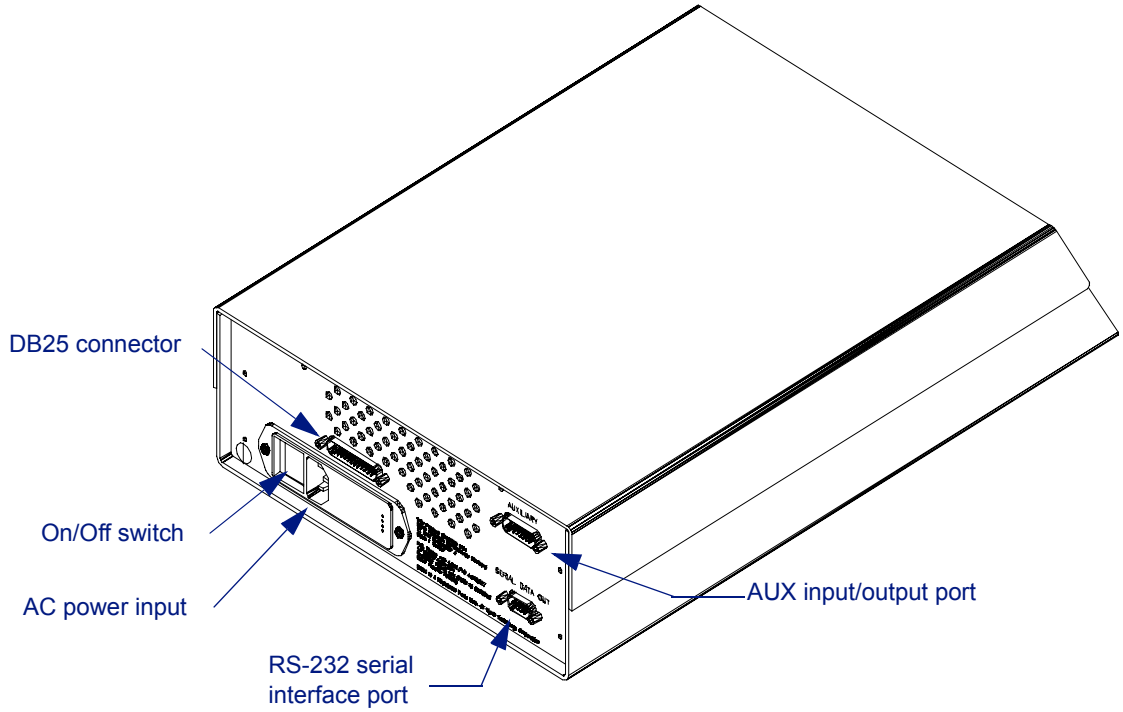


The side panel contains Outlet and Inlet ports. These are not used for the Heated miniDAWN because all the plumbing is done within the oven.

Electronics Enclosure Back Panel

The back panel contains the Main Power switch and the main AC power connector, DB25 data connector, Auxiliary Input/Output port, and RS-232 serial data port. The fuse holder is located next to the Main Power switch.

Figure 3-2: Electronics enclosure back panel



The back panel also contains information about the laser. No direct lateral radiation is received by the user.

The miniDAWN Tristar is configured for the AC power listed on the instrument identification label, and may be used anywhere in the world without reconfiguration.

Changing a Fuse

What you need to change a fuse:

- Tool for prying the AC Power module cover off.
- Fuse from the spares supplied in the accessory kit.

To replace a fuse:

1. Disconnect the power cord.
2. Open the cover of the AC Input Module using a small blade screwdriver or similar tool.
3. Replace the old fuse with a new one according to the following chart:

Voltage	Amperes	Speed
100/240	0.50	slow

4. Replace the cover of the AC Input Module and reconnect the power cord.

Removing the Cover

The electronics enclosure contains the main circuit board, the power supply, and various electronic components and connectors. With normal use, you will need to remove the cover *only* when changing the AUX gain(s).

What you need to remove the cover:

- 2.5 mm Ball driver

To remove the cover, do the following:

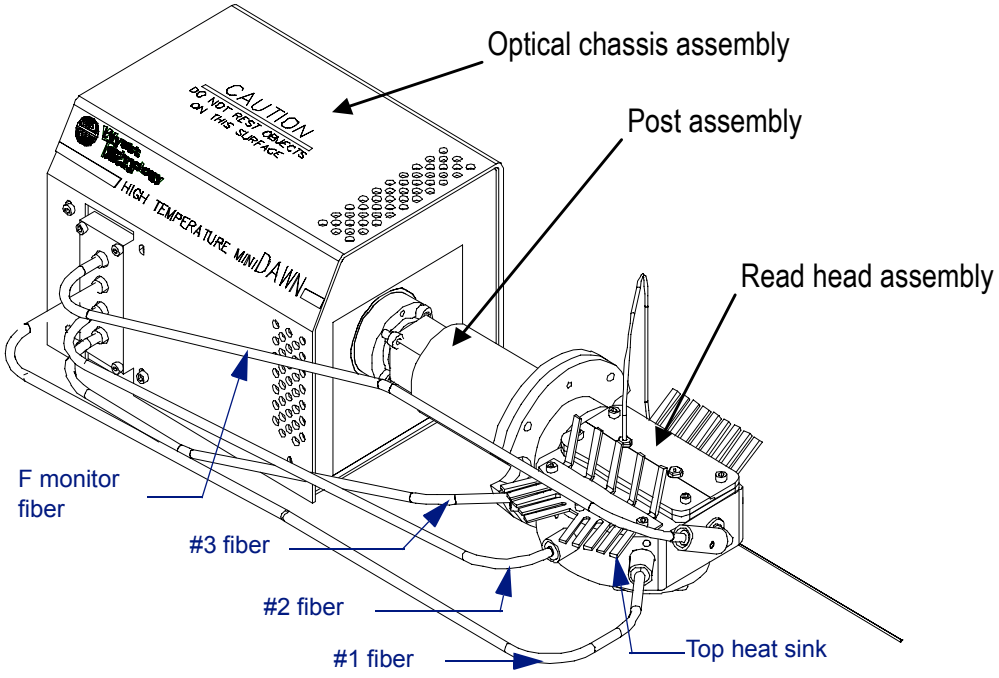
1. Turn the miniDAWN power off (see Figure 3-2).
2. Disconnect the power cord.

   All AC power circuits are protected by safety shields; however you should ALWAYS remove AC power from the instrument before opening the top cover as an additional safety measure.
3. Remove the four screws that fasten the top cover to the instrument using the Ball driver.
4. Lift the back end of the cover, slide it back slightly, then lift off.

Optical Bench Assembly

The portion of the heated miniDAWN that is installed in the oven wall of the 150C/PL210 is called the "optical bench assembly." The portion of this assembly that contains the laser and electronics is installed outside the oven, and is called the "optical chassis assembly." The portion of this assembly that contains the flow cell and read head is installed inside the oven, and is called the "read head assembly." These two assemblies are connected by the "post assembly."

Figure 3-3: Optical bench assembly



Laser

The 30 mW semiconductor diode laser provides the exceptional light source for the system. The special laser system provides very high power density at the illuminated sample by means of a special, narrow beam diameter. The laser is positioned so that the incident beam is vertically polarized. It has a laser light output monitor (LM), which you can view in the ASTRA software (see the software User's Guide for the procedure to view the channel outputs.)

Laser Beam Warning

It is good laboratory practice with any laser source, irrespective of its power, to AVOID LOOKING INTO THE BEAM. Figure 3-4 shows the warning label affixed to the read head.

Figure 3-4: Laser beam warning label

Laser Monitors

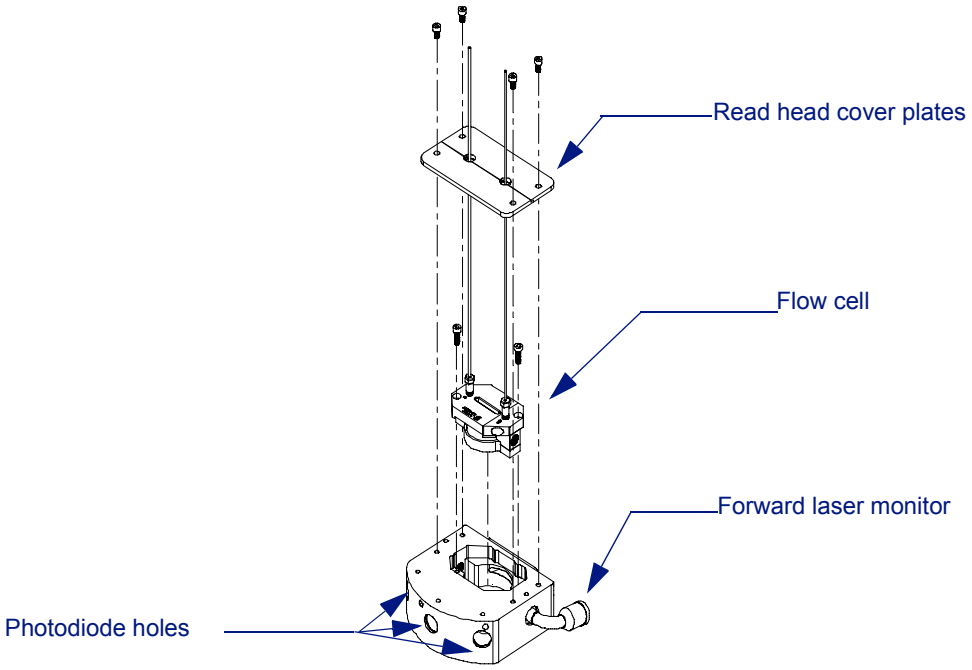
Two photodiodes in the miniDAWN monitor the laser beam intensity. The software uses the rear laser monitor (LM) to normalize the scattering signals relative to incident laser beam power fluctuations. The conventional method involves splitting the incident beam and dividing background corrected light scattering signals by the split signal. This is the system used for the miniDAWN.

The forward laser monitor provides the miniDAWN with the ability to measure transmitted light through the flow cell and sample. (The software does not use this value.) It is useful for determining whether the flow path has obstructions such as air bubbles, which may reduce the signal intensity to near zero volts. It is also useful for estimating how much light is absorbed by a sample containing chromophores that absorb at 690 nm. It appears on the front panel as CHANNEL F. This is the default setting. This setting allows the signal from a concentration detector to be received by the AUX2 connector. This data is then passed to the software along with the data collected by the DAWN.

Read Head and Detectors

Figure 3-3 shows the read head as part of the optical bench assembly. Figure 3-5 shows an exploded view of the read head assembly, with the flow cell and cover plates removed.

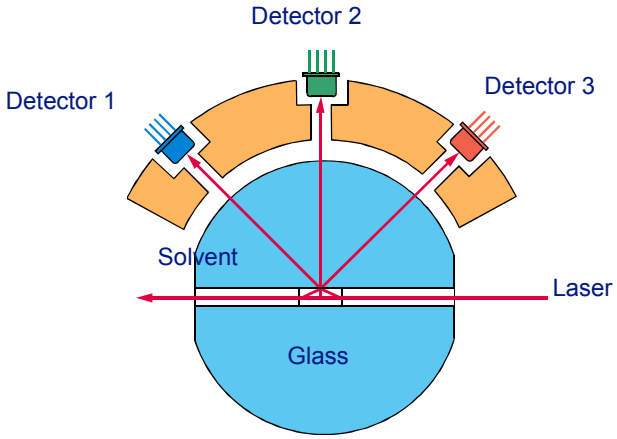
Figure 3-5: Read head assembly exploded



The read head holds the sample cell precisely, collimates scattered light and aligns and holds the detectors in place. Optical fibers, which connect to four photodiodes (three detectors and the forward laser monitor), limit the sample field of view at each detector, and minimizes stray light effects. Since each detector's field of view is limited by its own collimator, only the center of the illuminated sample scatters light into a given detector. Note that the Read head, Post, and Optical chassis assemblies make up a *single* optical bench structure called the Optical bench assembly. After its installation, the Optical bench assembly is solidly attached to the 150C/PL210 oven for maximum stability.

The photodiodes are mounted securely in the read head and require a special tool for removal. The three scattering detectors are arranged as shown in Figure 3-6.

Figure 3-6: Flow cell, laser beam, and detector positions in read head



The photodiodes are actually located on a mounting block inside the Optical chassis assembly; the optical fibers connect the photodiodes to the read head.

The detectors are arranged around the read head to point to the center of the cell. The laser beam passes in the same direction as the flow. The two vertical lines in the bore mark the scattering volume—less than 1 mL.

As with all high-performance electronics, the detectors are susceptible to damage from electrostatic discharge (ESD) and should not be handled unless you are electrically grounded.

The optics have been aligned at the factory and are not user adjustable. Under no circumstances should you alter the alignment settings; to do so will void the warranty and may cause damage to the instrument.

There are no internal fuses or circuit breakers.

Flow Cell

Flow Cell Design

The patented flow cell is at the heart of the miniDAWN, and is critical to the instrument's unique measuring capabilities.

In many applications, such as chromatography, the ability to measure small samples is crucial, so cell volumes must be minimal. The total volume of the cell from the manifold inlet to the manifold outlet is about 70 µL. The actual scattering volume—the illuminated part of the sample that is viewed by the detectors—is less than 1 µL.

To minimize stray light, the laser passes in the same direction as the solvent/sample flow and the cell's windows are recessed in the manifolds, away from the scattering volume. Any stray light from the air/window/solvent interfaces is therefore normally not seen by the detectors. The windows protrude into the flowing stream at the manifolds to minimize debris buildup on their flat ends. As a result, the detectors measure scattering only from the sample and not from the windows.

Figure 3-7: Flow cell assembly

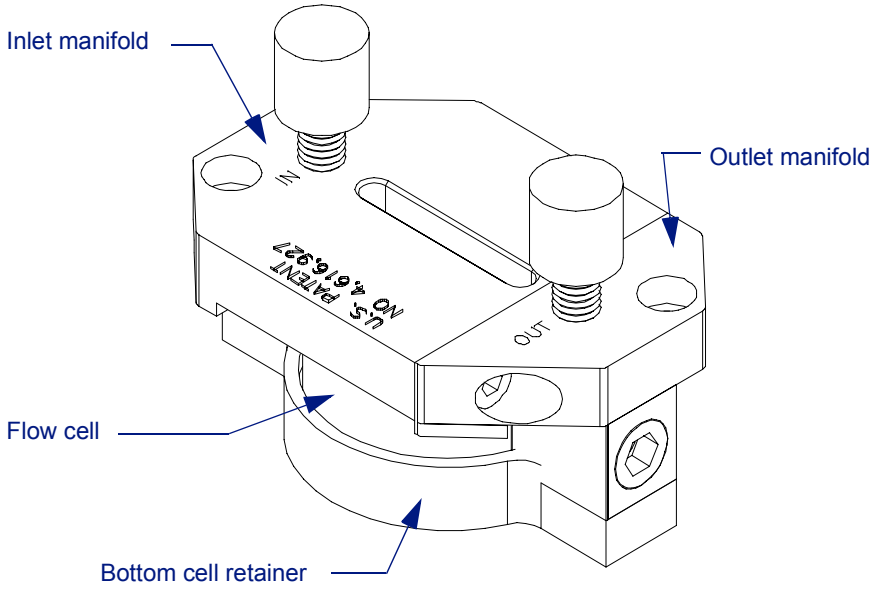
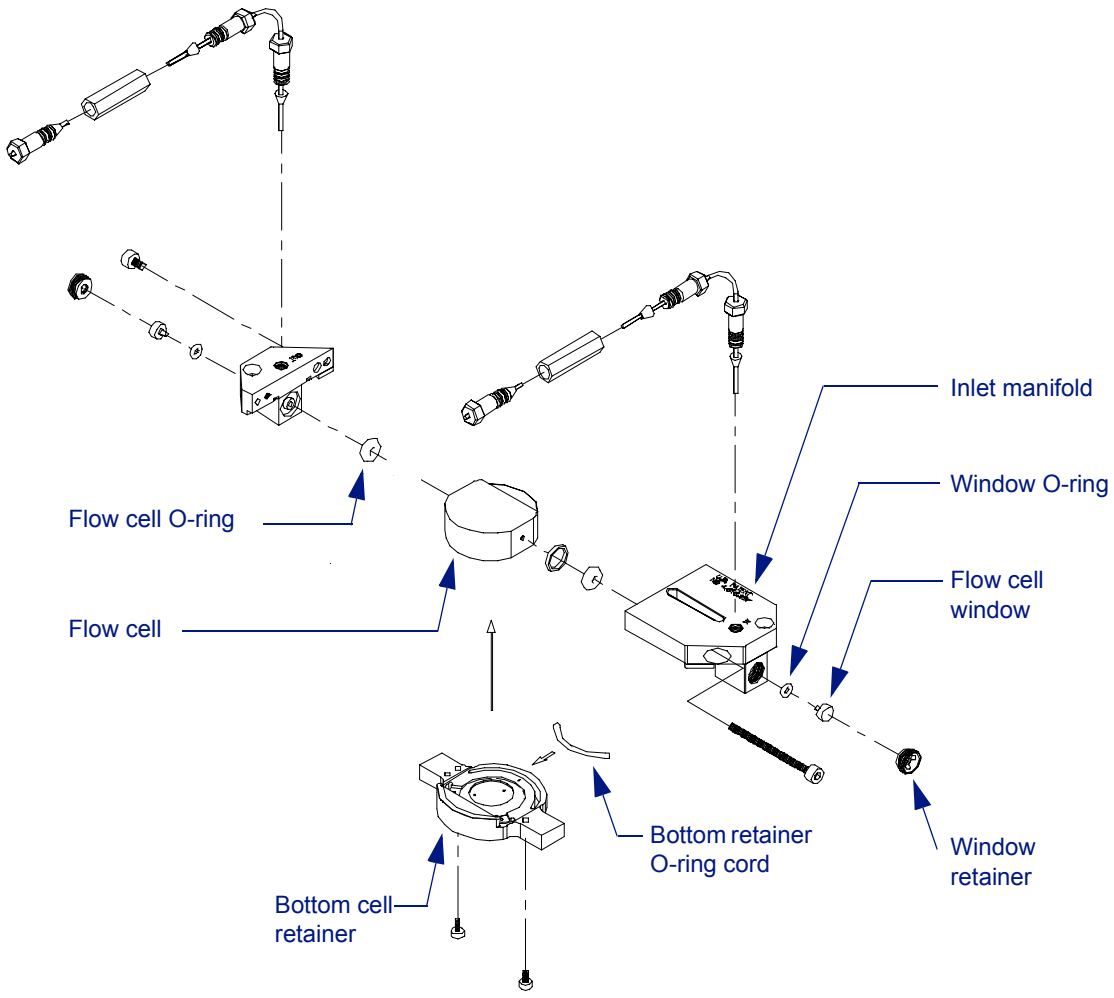


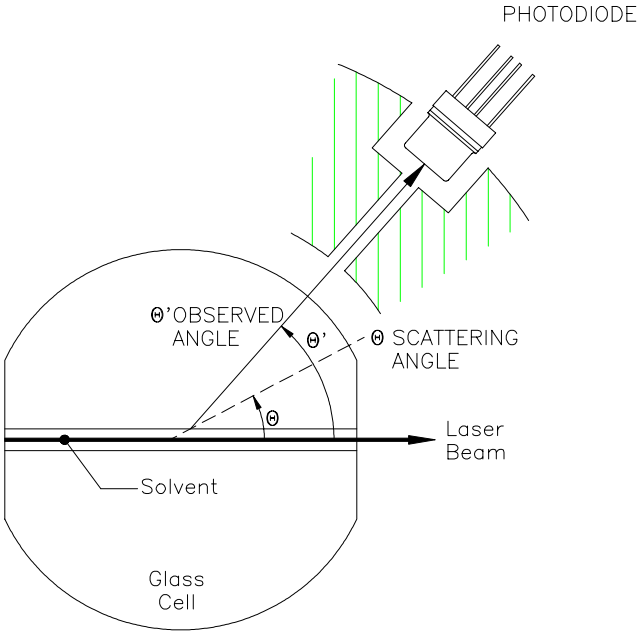
Figure 3-8: Exploded view of the flow cell assembly



Refractive Index Differences—Liquid vs. Glass

The difference in refractive index between the solvent and the surrounding glass cell results in some of the most important features of the flow cell design. As long as the refractive index of the solvent is less than that of the cell glass, it will be possible to obtain measurements of light scattered at relatively small angles, with minimized background contributions. Figure 3-9 shows a detail of the liquid/glass interface and rays scattering from the laser-illuminated sample.

Figure 3-9: Flow cell refractions



The angles are measured with respect to the direction of the laser beam. The illustration shows detector #1.

Applying Snell's Law, the refraction of a ray scattering at angle θ may be determined from

$$n_{liquid}\sin\left(\pi/2-\theta\right)=n_{glass}\sin\left(\pi/2-\theta'\right)$$

(1)

where the angle of incidence is $\pi/2-\theta$ and the angle of refraction is $\pi/2-\theta'$. Expanding the sine functions in Equation (1) results in

$$n_{liquid}\cos(\theta)=n_{glass}\cos\left(\theta'\right)$$

(2)

The detectors are set to detect light at an angle θ, collimated to be centered in the cell. As a result of refraction, the light detected is the light scattered at an angle θ. In this way a greater angular range of scattered light can be detected. The miniDAWN's controlling software handles these calculations automatically. All you need be aware of is the refractive index of the solvent you are using.

Table 3-2: Scattering angles for 3 detectors in toluene, water, TCB; F2 glass installed

Detector Number	Toluene	Water	TCB (135C)
1	44.7°	37.3°	45.1°
2	90.0°	90.0°	90.0°
3	135.3°	142.7°	134.9°

4 Maintenance

The Heated miniDAWN needs little maintenance. This chapter gives general guidelines for keeping the instrument in good working order and provides procedures for cleaning the flow cell.

CONTENTS **PAGE**

General Guidelines

Here are some general operation and maintenance guidelines for keeping the miniDAWN in good working order.

- Keep the miniDAWN electronics enclosure on a flat, clean surface, with space behind it and standing on its feet to allow proper air ventilation.
- Keep the case clean. Use a cloth dampened with water to clean it.
- Allow the electronics enclosure to warm up for 30 minutes before taking measurements.
- Keep the unit closed at all times.

  In addition, you will need to follow the procedure in “Flow Cell Maintenance” on page 4-3 to keep the flow cell clean.

Flow Cell Maintenance

The quality of the light scattering results you obtain from your miniDAWN depends critically on the cleanliness of the flow cell. If you follow the guidelines here, you should rarely need to disassemble your flow cell for cleaning.

However, eventually most cells need to be removed from the read head and disassembled for cleaning of their individual parts (described in "Cleaning the Flow Cell and Windows" on page 4-4).

Particles in the Cell

Here is a list of symptoms of particles in the cell and what you can do to dislodge them.

- Bright stationary spots when viewing the cell bore from above (this can be done at room temperature only).
- Higher voltage baselines than normal.
- Wavy baselines.
- Distorted chromatography peaks especially for detector 1 (dips below baseline, shoulders on right side of peaks, apparent shifts in elution volume).

On-line Cleaning

To keep the flow cell free of particles, we recommend regular maintenance as described here.

At All Times

- Use solvents that are HPLC grade.
- Keep well filtered solvent pumping continuously through the cell. Lower the flow rate if you do not plan to use the miniDAWN for a while.
- Change flow rate in small steps over a period of 10 minutes or longer.

After the GPC system has been brought to room temperature

- With the flow cell still in place, disconnect the miniDAWN from your HPLC system. Inject filtered toluene (0.02 mm) to flush the cell. We recommend filtered ethanol be left in the cell.
- Do not flush the cell from OUTLET to INLET since the inlet uses 0.005" ID tubing which is easily blocked.
- 6M nitric acid may be used to flush the cell.
- A mild detergent solution may also help clean the cell.

Cleaning the Flow Cell and Windows

When the flow cell is dirty, light scatters excessively, which shows up as high voltage, unstable baselines, and distorted chromatography peaks. The flow cell cleaning procedure can be broken down into five major steps:

Step 1—Removing the flow cell

Step 2—Disassembling the flow cell

Step 3—Cleaning the flow cell and windows

Step 4—Reassembling the flow cell

Step 5—Reinstalling the flow cell

What you will need for flow cell cleaning:

- A sheet of clean white paper taped down to your work surface.
- Anti-static wrist strap.
- Ball drivers: 1.5 mm, 2.5 mm and 4 mm.
- Lens tissue. Fold several pieces in finger-width strips for handling and cleaning.
- Lint-free gloves
- Oral-B SuperFloss
- Compressed gas. (Photographic supply stores carry this. At Wyatt Technology we use "Tech Spray" from Com-Kyl distributors in Santa Barbara, (805) 520-1731.)
- Filtered methanol, ethanol, or isopropanol
- Tweezers
- Optional: Sonicating bath

Caution: The flow cell you are about to remove constitutes a substantial amount of the purchase price of the DAWN. Its parts are carefully machined and are expensive. If you have any doubts whatsoever about the safest procedure for handling the cell structure, do not hesitate to call Wyatt Technology.

Step 1—Removing the Flow Cell Assembly

In this first step you will remove the cell assembly from the read head. Before proceeding, complete steps 1 and 2 in “Deinstalling for Service” on page 4-13.

1. Disconnect the Cell inlet tubing from the Heat exchanger outlet tubing.

   Leave the zdv union attached to the cell inlet tubing.

2. Put on the anti-static wrist strap.

   This is an important step. The strap protects the instrument from static discharge, in particular the photodiodes.

3. Switch off the Electronics enclosure and disconnect the power cord.
4. Use the 2.5 mm Ball driver to remove the four M3 screws holding the Read head cover plate in place, then lift off both sections of the cover plate.
5. Pull up the foam so that the flow cell assembly is visible.
6. Use the 2.5 mm Ball driver to remove the two M3 screws.
7. Lift the cell assembly (including the tubing) up and out of the read head; see Figure 4-1.

IMPORTANT: DO NOT PRY THE CELL OUT WITH A SCREW DRIVER OR ANY OTHER TOOL!

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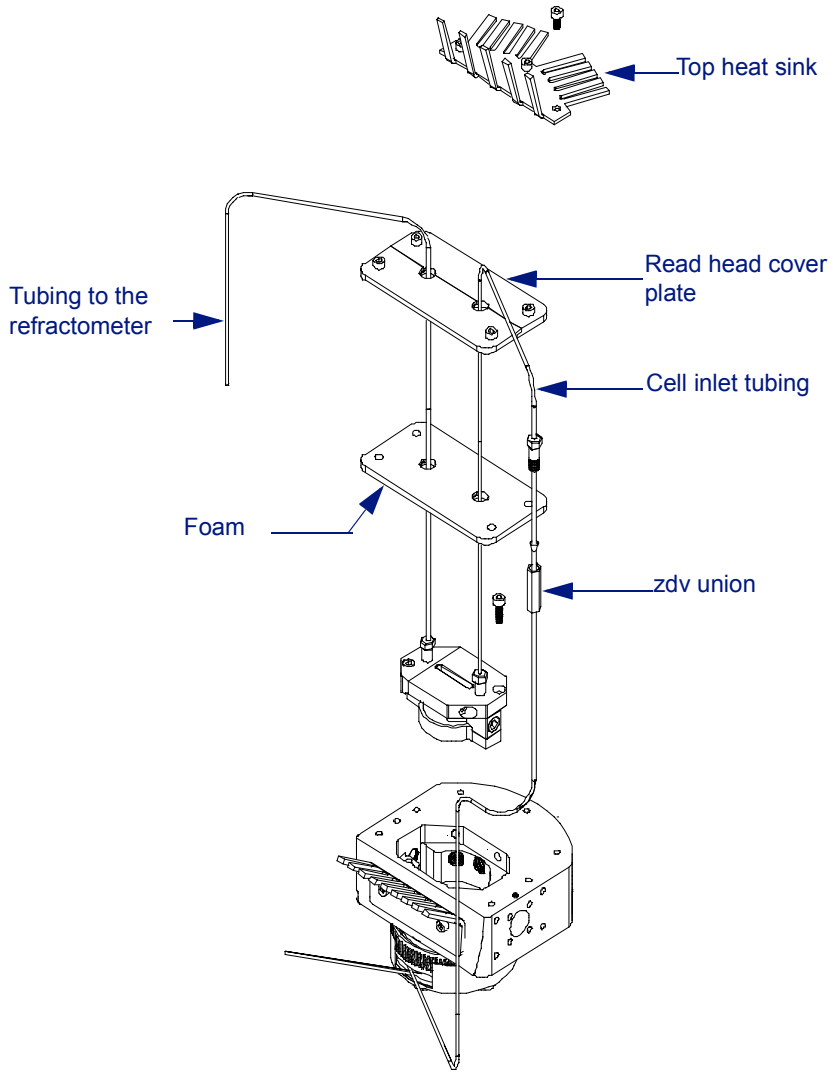
8. Carefully remove the two connecting tubings from the flow cell, taking care not to twist or bend them.

Figure 4-1: Removing the flow cell from the read head.



Step 2—Disassembling the Flow Cell

The different parts that make up the flow cell assembly are shown in Figure 4-2.

1. Separate the stainless steel manifolds from the flow cell:
   a. Use the 1.5 mm Ball driver to unscrew the two M2 screws holding the bottom cell retainer in place. Remove the bottom cell retainer taking care not to lose the two tiny screws and the bottom retainer O-ring and cord.
   b. Use the 2.5 mm Ball driver to remove the M3 screws that connect the two manifolds.
   c. Gently pull apart the manifolds, taking care not to drop the glass cell or touch its curved optical surfaces.
   d. Place everything on your paper-covered work surface, taking care not to lose the O-rings sealing the manifolds to the cell.

      If the DAWN is configured for use below 80 °C, there is a backing ring outside each 6 mm flow cell O-ring. If the DAWN is configured for use at or above 80 °C, there is a 9 mm flow cell O-ring (but no backing ring) on each side.
2. Use the 4 mm Ball driver to remove one window retainer at a time.

   Figure 4-6 illustrates the window-mount and how it is housed in the manifold.
3. Lightly tap the assembly ONCE against a flat clean surface. The cell window and O-ring should fall out if the cell is dry.

   If the window does not fall out easily, you could carefully apply a very mild burst of pressurized air to dislodge it or you could try gently pushing it out from the opposite side with a small piece of Teflon tubing. If necessary, put some filtered alcohol in all the manifold openings and soak overnight.
4. Repeat Step 3 for the other window.

Figure 4-2: Flow cell assembly, exploded view

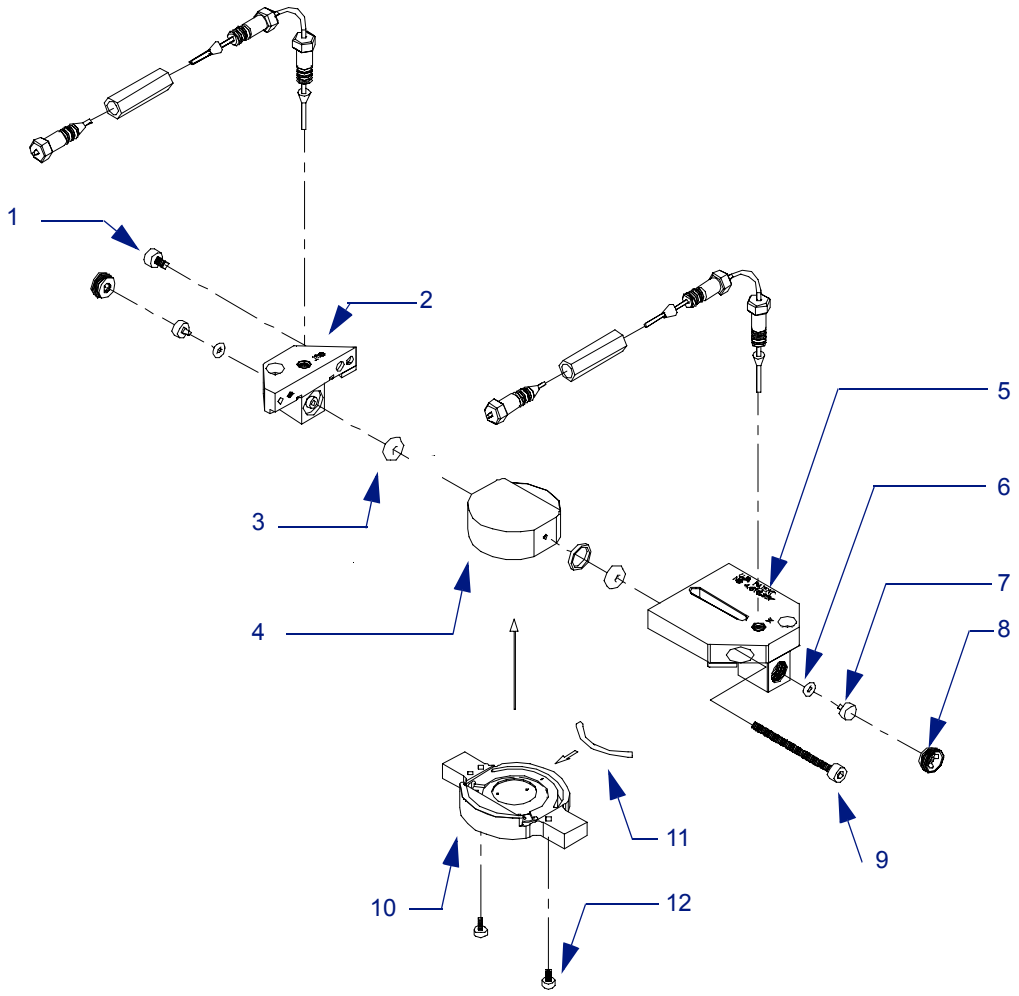


Table 4-1: Flow cell assembly, parts list

Item	P/N	Description
1	S5002-3004	M3 screw
2	200694	Manifold, out
3	P6504-2009	Flow cell O-ring (2)
4	212095	Flow cell
5	200690	Manifold, in
6	P6504-2004	Window O-ring (2)
7	116007	Flow cell window (2)
8	212073	Window retainer (2)
9	S5002-3030	M3 screw
10	211048	Bottom flow cell retainer
11	S6501	Bottom retainer O-ring cord
12	S5002-2006	M2 screw (2)

Step 3—Cleaning the Flow Cell

From here on, you must be fastidious in your handling and cleaning of the flow cell parts. The smallest particle on the flow cell window or inside the bore can introduce stray light and distort your measurements.

Tip: For more thorough cleaning of the optical parts (glass cell and windows), we suggest you use an ultrasonic cleaning unit. If you do, place the parts in a small beaker and cover with filtered alcohol. Fill the ultrasonic unit with enough water to reach part way up the side of the beaker. Place the beaker in the unit and sonicate the parts for about five minutes. Let the flow cell dry on a piece of lens tissue with the bore in a vertical position. Having done this you may not need to clean the cell through-bore as described in Step 2, which follows.

1. Clean your hands thoroughly or wear lint-free gloves.

   When you disassemble the cell, be careful not to handle the glass cell's curved optical surfaces (the sides).

2. Clean the cell through-bore.

   a. Cut a ½" strip of lens tissue and roll it into a thin wick. Or, you may use "Oral-B SuperFloss", available in most pharmacies. The floss is a better tool, as it cannot leave any fibers behind.

   b. Insert the wick all the way through the cell bore, then moisten it with a small amount of filtered ethanol.

   c. While the wick is in the cell bore, untwist it slightly, move it back and forth to clean the cell, then pull it out.

   d. Immediately flush the bore with a stream of ethanol for 10–15 seconds.

      The ethanol stream flushes out any fibers that may have been left behind by the tissue wick.

   e. Blow out the ethanol for 10–15 seconds with compressed gas or let the glass bore drain in a vertical position.

   f. Examine the bore with a magnifying loupe.

      Look through the bore, focusing on the bore exit. Repeat from the opposite side. (See the **Note** on page 4-10.)

3. Clean the outside of the cell. (See Figure 4-3.)

   a. Pick up the cell with a folded lens tissue; touch only the flat surfaces.

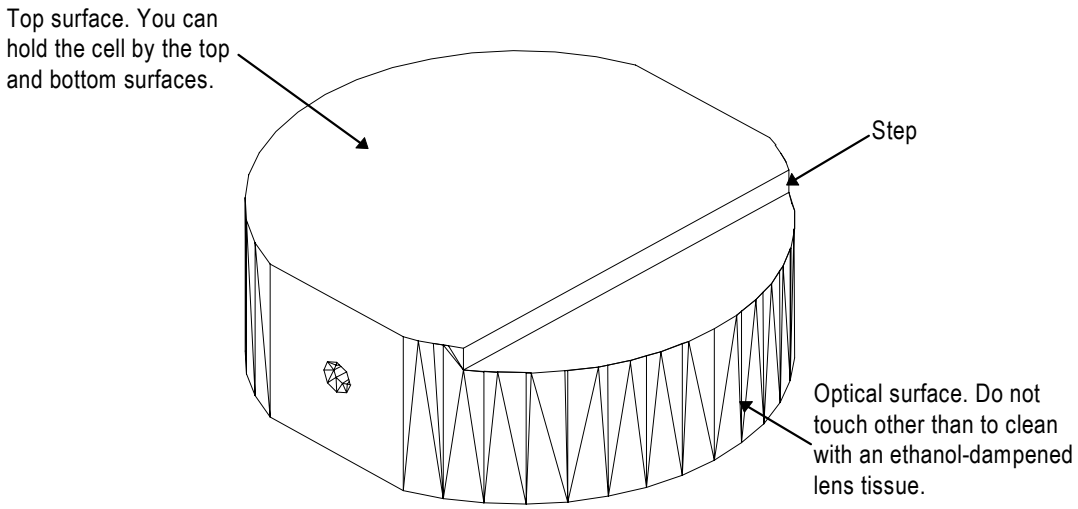
   b. Wipe the curved optical surfaces with another folded lens tissue moistened with alcohol.

   c. If needed, wipe the ethanol off the optical surfaces with dry lens tissue. **Do not** repetitively rub the surfaces since this creates static electricity which attracts particles.

d. Using a magnifying loupe, examine the optical surfaces for any dust. (See the **Note** on page 4-10.)

e. Also, check the bottom and top surfaces for dust and finger marks.

Figure 4-3: Flow cell



4. Clean the window faces.

This is the most important step in cell cleaning. Even the smallest particle left on the window faces will induce stray light and cause signal distortion, especially at low angles.

a. Take a folded lens tissue moistened with a couple of drops of alcohol and hold it against the side of your forefinger with your thumb and third finger.

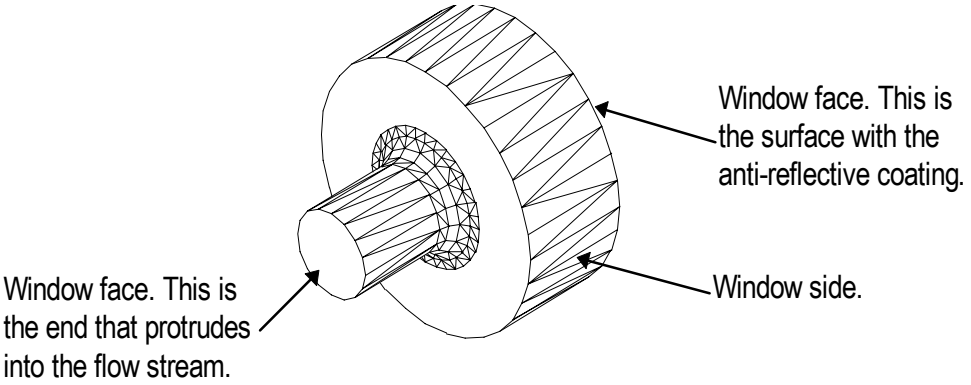
b. Pick up the window with the tweezers. Hold the window by the sides, not by the window faces.

c. Smoothly wipe both window faces across the tissue.

d. Carefully examine both ends of the cleaned window for any particles.

With the loupe look straight through the window from end-to-end. (See the **Note** on page 4-10.)

Figure 4-4: Cell window



This tiny glass part is specially manufactured and is expensive.

Note: By examining the flow cell through-bore and the windows using a bright light, you can, with some practice, easily find where any residue has accumulated. Examine them with a jeweler's loupe while back-lighting the glass at a slight angle. The area next to the light should be dark to provide good contrast. The bright light will illuminate any particles on the glass which, when viewed against the dark background, will show up clearly. Since fingerprints on the glass cell circumference will alter the light scattering characteristics of a sample significantly, we urge you to use great care when handling the cell. Its role is vital in the measurement process and you must be certain to wipe it clean with high quality lens tissue before inserting it again in the cell assembly.

Step 4—Reassembling the Flow Cell

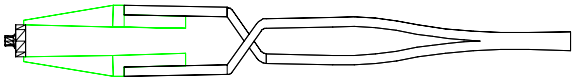
You will need to use new flow cell windows and O-rings each time you clean the flow cell for the heated miniDAWN. The high temperatures used cause the O-rings to degrade and adhere to the flow cell windows.

As you reassemble the flow cell you will clean the washers and O-rings.

Note: Assemble the flow cell in a laminar flow hood if there is one available.

1. Carefully replace the windows with their O-rings, washers and retainers so that the windows are not chipped or over-tightened. (See Figure 4-6.)
   a. Holding the window O-ring with the tweezers, put a drop of alcohol on it, then dry with a burst of pressurized air. Check for particles with the loupe.
   b. Insert the O-ring into the manifold.
   c. Lift the window with the tweezers. (Pick up the window near its back edge as shown in Figure 4-5.)

Figure 4-5: Holding the cell window for reinsertion into the manifold



d. Holding the manifold and window square with one another, gently push the window into the O-ring.

e. Let go of the window with the tweezers, pivoting them before you lift them out of the manifold.

The fit is tight enough that you could dislodge the window if you were to just lift the tweezers straight up.

f. Inspect the tip of the 4 mm Ball driver with the loupe for any particles, and, if necessary, clean with an alcohol-moistened lens tissue before proceeding.

g. Clean the window retainer with alcohol and pressurized air, then place it in the manifold and tighten with the 4 mm Ball driver.

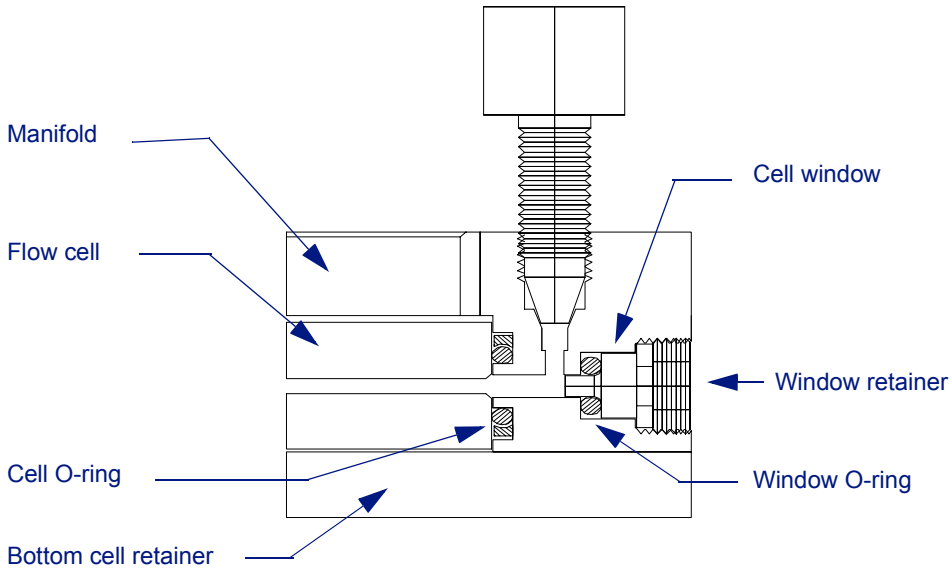
You may need to use your fingers to start the tightening of the retainer.

h. Inspect the window mount with the loupe.

If any particles appear on the window, you need to remove it and its seals and clean again.

i. Repeat steps 1a) through 1i) for the second window.

Figure 4-6: Window mount detail



2. Install the cell in the manifolds.

a. Insert the cell O-rings followed by the backing rings if they were removed in Step 2.1.

b. Holding the cell with lens tissue, place it in the inlet manifold (the larger manifold).

   A step is machined into the top surface of the glass cell; the manifold has two pins to help align the cell properly.

c. Push the cell step against the manifold pins.

d. Make sure that the glass step and manifold pins are matched up well.

e. Place the outlet manifold next to the inlet manifold and push them firmly together.

3. Insert the short M3 screw into the outlet manifold and tighten with the 2.5 mm Ball driver. Then, insert the long M3 screw into the inlet manifold and tighten.

4. View the O-rings through the bottom glass surface (make sure the surface is clean) and confirm that the bore is centered in each O-ring.

   Also examine the alignment pins to make sure they touch the cell glass on each side.

5. Inspect the sides of the cell and apply a burst of air if you see any particles.

6. Replace the bottom cell retainer cord and O-ring and attach the bottom cell retainer to the manifolds using the 1.5 mm Ball driver.

Step 5—Reinstalling the Flow Cell

1. Reattach the inlet and outlet tubing to the cell, making sure the foam is in place.

   If you are not careful, the cell could be reversed. Make sure that the 0.005" ID tubing with the zdv union is attached to the manifold labeled IN (Figure 4-1).

   After several cleanings the fittings may become worn or deformed, at which point the tubing *and* fittings should be replaced. The instrument is shipped with one spare set of tubings.

2. Replace the cell assembly in the read.

   Insert the two M3 screws into the top of the cell and tighten with the 2.5 mm Ball driver.

3. Replace both sections of the Read head cover plate.

   Insert the four M3 screws and tighten with the 2.5 mm Ball driver. Make sure the foam is in place underneath it.

4. Plug in the power cord and turn on the miniDAWN.

   Next proceed to "System Startup and Shutdown" on page 5-1 for directions on how to complete the plumbing and heat up the system to its operating temperature.

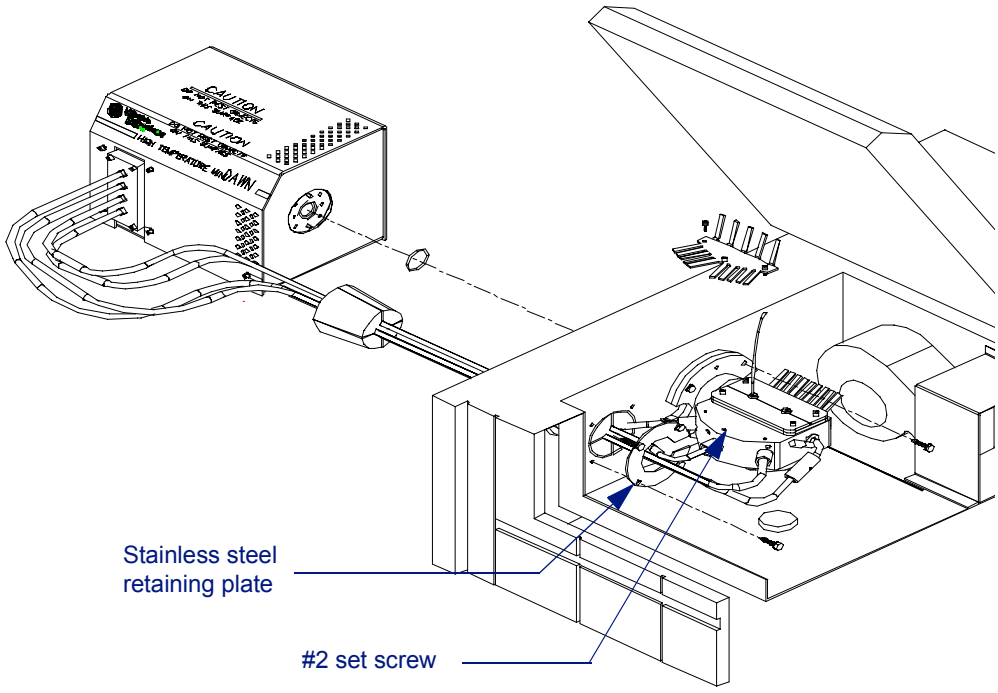
Deinstalling for Service

The laser assembly cannot be aligned while it is installed in the oven. If the laser assembly ever becomes misaligned (for example, if it receives a severe mechanical shock) or if the laser is damaged, you should deinstall and return the assembly for service.

You will need the following materials and tools:

- 1.5 mm Ball driver
- 2.5 mm Ball driver
- 4 mm Allen wrench (ground down)
- ¼" socket driver
- electrical tape
- aluminum foil
- laboratory jack
- Shorting connector
- Anti-static wrist strap

Figure 4-7: Deinstalling the optical bench assembly (view from inside oven)



To deinstall the optical bench assembly, do the following:

1. Cool the 150C/PL210 oven to room temperature.

First reduce the flow rate to 0.1 mL/min. Switch off the oven temperature controller and let it slowly reach room temperature with the oven lid closed.

2. Stop the pump, then open the oven and disconnect the Cell outlet line from the refractometer.
3. Disconnect the Heat exchanger inlet line from the columns.
4. Put on the Anti-static wrist strap.
5. Switch off the miniDAWN Electronics enclosure and disconnect the power cord.
6. Disconnect the Interface cable from the back of the miniDAWN Electronics enclosure and the Optical chassis assembly.
7. Connect the Shorting connector to the Optical chassis assembly.

   The connector shorts the contacts of the laser and protects it from damage from electrostatic discharge.
8. Support the Optical chassis assembly with a laboratory jack.
9. Loosen the 2 screws on the Dress plate using the 1.5 mm Ball driver (see Figure 4-8).
10. Remove the 2 screws from the Exterior foam clamp using the socket driver (see Figure 4-8).
11. Remove the 4 screws holding the Stainless steel retaining plate using the socket driver (see Figure 4-7).
12. Remove the Top heat sink and the Read head cover plate if they are in place.

    Use the 2.5 mm Ball driver.
13. Loosen all 4 set screws that hold the optical fibers in place using the 1.5 mm Ball driver (see Figure 4-7).
14. Remove the four optical fibers from the read head by sliding them back out of their holes.

    The #3 fiber (closest to the oven wall) will not come all of way out of its hole.
15. Use the 4 mm Allen wrench to loosen and remove the 4 cap screws on the precision flanges that connect the Optical chassis assembly to the Post assembly.

    Do not loosen the flange o-ring. The Optical chassis assembly is now resting on the laboratory jack.
16. Cover the open ends of the two flange halves with aluminum foil securing it with electrical tape.
17. Using the socket driver, unscrew the 4 self-tapping screws that mount the read head to the inside of the oven wall.

    Once you have removed these screws, the #3 optical fiber should be free to slide out of its hole.

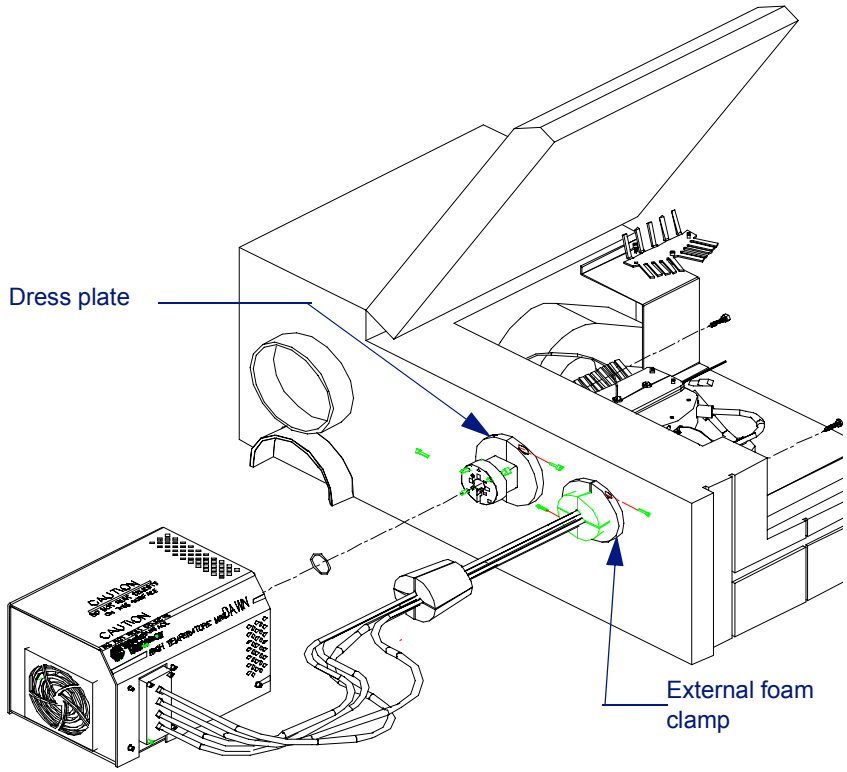
18. Cover the ends of the 4 Optical fibers with aluminum foil securing it with electrical tape.
19. Carefully pull the Optical fibers out through the oven wall.

    Remove the Hollow foam stoppers at the same time.
20. Carefully remove the Read head/Post assemblies from the oven chamber.

    Cover the exposed holes in the read head with electrical tape.

Please call Wyatt Technology for detailed packing and shipping instructions.

Figure 4-8: Deinstalling the optical bench assembly (view from outside oven)



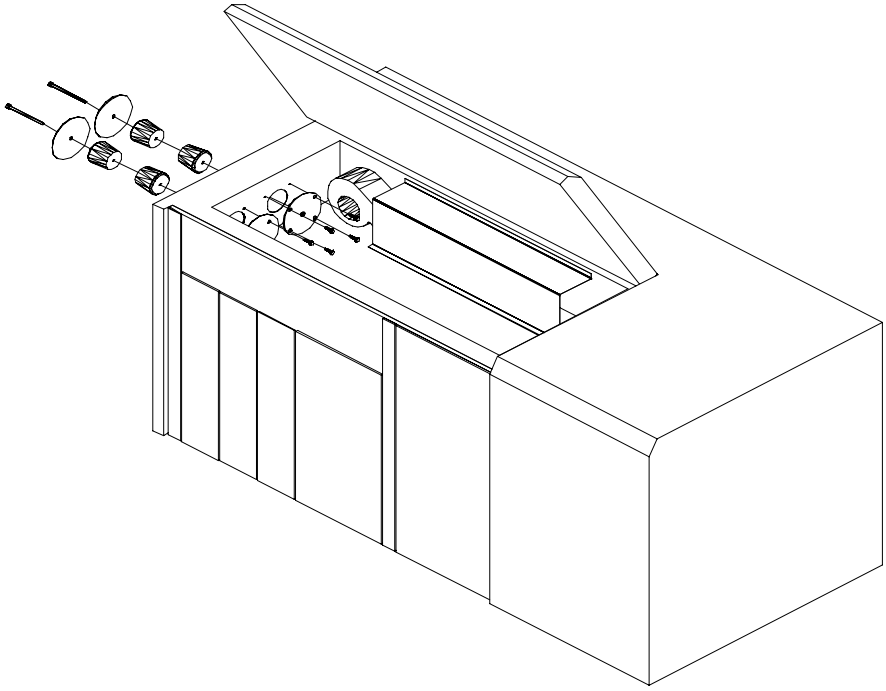
Plugging the Oven Holes

Wyatt Technology provides two Plug assemblies that fill the holes in the oven wall and allow you to use the 150C/PL210 while the Heated miniDAWN is being serviced.

You will need the following materials and tools:

- 3 mm Ball driver
- ¼" socket driver
- 2 Plug assemblies
- 8 self-tapping screws

Figure 4-9: Installing plugs in oven wall



To install the plugs, follow these steps:

1. Unscrew the long M4 screw which connects its two halves together.

   Use the 3 mm Ball driver.

2. Insert the blue foam plugs in the wall of the oven.

   Insert one from the outside and one from the inside.

3. Insert the long M4 screw through the center of the foam plugs and attach it to the inner plate.

4. Tighten the M4 screw until the foam plugs expand and fill the hole in the oven wall.

5. Repeat step 1 through 4 for the second plug.

6. Install the 8 self-tapping screws using the socket driver.

   Each inner plate requires 4 screws.

5 System Startup and Shutdown

Using a Heated miniDAWN is similar to using a non-heated miniDAWN. This chapter gives procedures for heating up and for cooling down the system. Details about data collection are provided in the software manual for the software you are using.

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Differences Between a miniDawn and Heated miniDawn

If you have previously used a ambient miniDAWN, you should be aware of the following differences between the use of that device and the heated miniDAWN:

- Data collection can be performed at up to 150 degrees C.
- The recommended solvent is trichlorobenzene (TCB), which has a refractive index at 150 degrees C of 1.500 at 690 nm.
- The cell type used is F2.
- Each time you clean the flow cell, you should replace the flow cell windows and all four o-rings. The o-rings degrade faster at high temperatures.
- Particles tend to shed from a column during temperature cycling and can accumulate in the flow cell. To prevent this, you should use the procedure described in “System Startup” on page 5-2 to bring the system up to operating temperature and to plumb the flow cell.

System Startup

It is essential to keep the flow cell clean when plumbing it into the flow system. Although the cell plumbing can be performed at room temperature, it is our experience that particles shed from a column during temperature cycling can accumulate in the flow cell. The particles create unstable miniDAWN baselines and cause distortion of the miniDAWN peaks.

Therefore, the preferred method for plumbing the cell is to perform the steps at elevated temperature with the cell empty. This method helps prevent the particle problem.

Instructions for connecting a freshly cleaned cell

We assume that you have completed Step 5 of the cell cleaning procedure in Chapter 4. The oven is currently at room temperature and the cell inlet and outlet tubing are connected to the cell but not connected to the 150C/PL210. The cell inlet tubing should have the zdv union installed.

1. Connect the heat exchanger outlet tubing directly to the RI detector, bypassing the cell.

The short length of 0.005" ID tubing, the cell inlet tubing, is normally connected to the heat exchanger outlet via the zdv union. You should leave it unconnected.

2. If there is solvent in the cell (other than TCB), connect an empty syringe to the cell inlet tubing and withdraw all solvent, then disconnect the syringe.
3. Begin flowing solvent through the system. It is best to let the solvent flow at approximately 0.2 mL/min during the rest of this procedure.

We recommend that you insulate your chromatography columns. The insulation prevents them from cooling too rapidly when the oven is opened to perform the miniDAWN plumbing. (It is good practice to insulate the columns anyway, since it improves their temperature uniformity).

4. Raise the oven temperature to 80C no faster than 1C per minute.
5. After the system has equilibrated, put on heatproof gloves and open the oven lid.
6. Quickly disconnect the heat exchanger outlet tubing from the RI detector.
7. Connect the cell outlet tubing to the RI detector.
8. Connect the heat exchanger outlet tubing to the cell inlet zdv union.
9. Close the oven lid and slowly raise the oven temperature to its operating temperature, no faster than 1C per minute.
10. Slowly increase the solvent flow to the operating rate.

    Increasing the flow rate from 0.2 mL/min to 1.0 mL/min should take at least 30 minutes. Use an even slower increase rate if the columns are new or have not been used for a long time.
11. Wait for the baseline noise to settle down before injecting a sample.

    The peak-to-peak noise on all three detectors should be below 5 mV.

System Shutdown

To shut down the 150C/PL210 for service, follow these directions:

1. Lower the flow rate to 0.2 mL/min.
2. Slowly lower the oven temperature to 80C, no faster than 1C per minute.
3. Open the oven lid and disconnect the cell outlet tubing from the refractometer.
4. Disconnect the heat exchanger outlet tubing from the cell inlet zdv union.

   Plug the zdv union with a stainless steel plug.
5. Connect the heat exchanger outlet tubing to the inlet of the refractometer.
6. Close the oven lid and switch off the temperature of the oven.

   Wait for the oven to reach room temperature before proceeding.
7. Perform the necessary service on the 150C/PL210, then follow the procedures under System Startup to bring the instrument up to temperature again.

Warnings and Cautions

Do not place anything on top of the Optical Chassis Assembly. Adding weight can cause misalignment.

While the power is ON, never disconnect the Interface cable between the miniDAWN Electronics enclosure and the Optical bench assembly.

If you ever need to disconnect the optical bench assembly, make sure you wear an anti-static wrist strap and install the Shorting connector (originally shipped installed) on the Optical chassis assembly. The diode laser is very sensitive to static discharge.

A Accessories

This appendix lists recommended accessories for various light scattering applications. The WTC kits can be purchased directly from Wyatt Technology; other accessory parts can be purchased form the listed vendors.

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Wyatt Technology Kits

Name	Description	WTC Part #
Cell Cleaning Kit	Tweezers, lens tissue, dusting gas, wrist strap	900001
Filter Kit A	In-line filter holder, fittings, filter membranes (aqueous or organic)	900002
Filter Kit B	Syringes, syringe filters (0.02 μm), fittings, PEEK tubing	900003
DNDC Kit	Injection valve, 1 mL injection loop, syringes, PEEK tubing, fittings	900004

Accessory Parts

The following accessories can be used with your miniDAWN. Contact the following vendors for the listed parts:

Vendors	Phone Number
Alltech	800-255-8324
Millipore	800-645-5476
Upchurch	800-426-0191
VWR	800-932-5000
Air-Tite of Virginia	800-231-7762
RAZEL Scientific Instruments	203-324-9914

Syringe Filters

Inorganic media, good for aqueous or organic solvents

Name	Diameter (mm)	Pore size (µm)	Alltech Part Number
Whatman Anotop 10	10	0.02	2172
Whatman Anotop 10	10	0.10	2240
Whatman Anotop 10	10	0.20	2170
Whatman Anotop 25	25	0.02	2132
Whatman Anotop 25	25	0.10	2252
Whatman Anotop 25	25	0.20	2130

Syringes

10 mL, all polymer (good for organic and aqueous solvents)

Importer	Air-Tite of Virginia, Inc.
Available from VWR	VWR Part Number 53548-006

Accessories for Microbatch Work with a Syringe

Name	Description	Upchurch Part Number
Female Luer adapter	from Luer to 10-32 threads	P-642
Finger Tight fittings, extra long	For 1/16" tubing,10-32 threads	F-130
PEEK tubing	0.010" x 1/16" x 5'	1531
PEEK tubing	0.020" x 1/16" x 5'	1532
RAZEL syringe pump	Low cost basic pump	

GPC In-Line High Pressure Filter

For 1/16” Lines

Description	Vendor	Vendor Part Number	Quantity
High Pressure Filter Holder Stainless Steel Diameter = 25 mm	Millipore	XX45 025 00	1 each
Anodisc inorganic membrane filter Pore sizes 0.2, 0.1, or 0.02 µm Diameter = 25 mm	Alltech	2250 2268 2255	50/pkg
Durapore* membrane filter Pore size = 0.10 µm Diameter = 25 mm	Millipore	VVLP 025 00	100/pkg
MF **membrane filter Pore size = 0.025 µm Diameter = 25 mm	Millipore	VSWP 025 00	100/pkg
Stainless Steel fitting 1/8” to 1/8” MPT	Alltech	61038	1 each (2 required)
Reducing ferrules Teflon 1/8” to 1/16”	Alltech	RF-200/100T	10/pkg

* Hydrophilic Durapore - Aqueous and selected organic solvents (Toluene, THF, etc)

** MF Mixed Cellulose - Aqueous and selected organic solvents (Toluene, THF, etc)

B Laser Specifications

The miniDAWN contains a GaAs laser operating at a nominal wavelength of 685nm. The GaAs laser is a single transverse mode heterojunction that emits light between 680nm and 690nm, where the exact wavelength varies from device to device. Typically diode lasers undergo periodic mode hops between different longitudinal modes which have slightly different efficiencies giving rise to sudden changes in intensity, however Wyatt Technology utilizes a patented intensity stabilization method which achieves a typical long term intensity stability of 0.1%.

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Electrical and Optical Specifications

Table B-1: Electrical and optical specifications

	GaAs
Power Output	30 mW
Laser Operating Wavelength	680 nm – 690 nm
Vertical Beam $1.0/e^2$ Intensity Diameter	80 µm
Horizontal Beam $1.0/e^2$ Intensity Diameter	52 µm
Polarization Ratio	> 100:1
Max Power Stability	< 0.5%
Typical Optical Noise	0.1%
Typical Operating Voltage	2.4 VDC
Typical Operating Current	85 mA

Environmental Specifications and Safety Notes

Table B-2: Environmental specifications

	GaAs Operating	**GaAs Non-Operating**
Temperature	-10 to +60 °C	-40 to +85 °C
Relative Humidity	0-95%	0-95%
Shock	1500 G – 0.5 ms	1500 G – 0.5 ms

The lasers used in the miniDAWN are classified as Class 1 Laser Product according to IEC60825-1:1993+A1+A2. This means that under normal operation, no laser radiation should escape from the instrument, and no protective equipment must be worn. However the follow warning applies:

Caution: Use of controls or adjustment or performance of procedures other than specified herein may result in hazardous radiation exposure.

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