

# PDA-100 (USB) PHOTODIODE ARRAY DETECTOR OPERATOR'S MANUAL

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#### PRINTING HISTORY

Revision 01, April 2004 Revision 02, July 2004

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## 1.1 **Product Overview**

The Dionex PDA-100 Photodiode Array Detector is an optical detector capable of measuring the absorbance spectrum from 190 to 800 nm. A deuterium lamp optimizes the UV range (190 to 380 nm) and a tungsten lamp optimizes the visible range (380 to 800 nm).

When using the PDA-100, up to five single wavelengths (2D chromatograms) can be collected without being required to collect 3D data. Collecting individual wavelengths instead of the spectra offers two advantages: it eliminates the need to perform extractions for analyses that do not require spectral data and it conserves disk space.

Key features of the PDA-100 include:

- The deuterium and tungsten lamps ensure low baseline noise, high signal intensity, and applications flexibility in the wavelength range from 190 to 800 nm.
- A 1024-element photodiode array optimizes spectral resolution.
- A built-in holmium oxide filter verifies wavelength calibration accuracy.
- The front panel LEDs indicate detector status at a glance.
- Four analog outputs support data collection on alternate data collection systems.

The PDA-100 is controlled remotely by a computer running the Chromeleon<sup>®</sup> Chromatography Management System (release 6.60 or later). The detector communicates with the computer through USB (Universal Serial Bus).

To take full advantage of the capabilities of the PDA-100, order Chromeleon with the PDA Control option. This option is required in order to perform:

- 3D data presentation and spectral analysis (real-time or post-run)
- Peak purity analysis with selectable criteria
- Spectral library search for positive peak identification

# 1.2 Theory of Photodiode Array Detection

Monochromatic variable wavelength detectors monitor eluting components of the sample at a single wavelength (ideally, the wavelength of maximum absorbance). Photodiode array (PDA) detectors scan a range of wavelengths every few milliseconds and continually generate spectral information. Wavelength, time, and absorbance can all be plotted.

PDA detectors provide three-dimensional information that allows an accurate assessment of peak identity, purity, and quantitation in a single analysis. Software support for PDA detectors includes peak purity and spectral library search functions to help determine peak homogeneity and identity.

### 1.2.1 Advantages of Photodiode Array Detection

PDA detectors are useful in both research and quality assurance laboratories. In the research laboratory, the PDA provides the analyst with a variety of approaches to the analysis. In the quality assurance laboratory, the PDA provides several results from a single analysis, thereby increasing the throughput of the HPLC.

PDA detection offers the following advantages:

#### • Peak measurement at all wavelengths

In methods development, detailed information about the detector conditions required for the analysis may not be known. When a variable wavelength detector is used, a sample must often be injected several times, with varying wavelengths, to ensure that all peaks are detected. When a PDA detector is used, a wavelength range can be programmed and all compounds that absorb within this range can be detected in a single analysis.

#### • Determination of the correct wavelengths in one analysis

After all peaks have been detected, the maximum absorbance wavelength for each peak can be determined. A PDA detector can collect spectra of each peak, after which the PDA software can calculate the absorbance maximum.

#### • Detection of multiple wavelengths

A PDA detector can monitor a sample at more than one wavelength. This is especially useful when the wavelength maxima of the analytes are different. Wavelengths can be selected to analyze each compound at its highest sensitivity.

#### • Peak purity analysis

It is difficult to determine component purity from a chromatogram. However, a PDA detector can analyze peak purity by comparing spectra within a peak. The better the spectra match, the higher the possibility that the peak is pure.

#### • Positive peak identification

In liquid chromatography, peak identification is usually based on relative retention times. When a PDA detector is used, spectra are automatically collected as each peak elutes. The PDA software compares the spectra with those stored in a library to determine the best fit matches; this method increases the likelihood of correctly identifying peaks.

For example, <u>Figure 1-1</u> shows an overlay of pyrene and an impurity obtained from two consecutive chromatographic analyses. Based on 2D data, or if the analyses had been performed with a conventional variable wavelength detector, these peaks might have been misidentified as the same component.

A PDA-100, under the control of Chromeleon with the PDA Control option, correctly identified and differentiated the components. The spectra of pyrene and the impurity (see <u>Figure 1-2</u>) clearly indicate that the two peaks are different components.



Figure 1-1. Overlay of Pyrene and an Impurity



Figure 1-2. Spectra of Pyrene and an Impurity

# 1.3 Overview of This Manual

Chapter 1 Introduction	Introduces the PDA-100 and the theory of photodiode array detection; explains the conventions used in this manual, including safety-related information.
Chapter 2 Description	Provides an overview of PDA-100 operating features and system components.
Chapter 3 Operation and Maintenance	Provides an overview of PDA-100 operation, including guidelines for optimizing detector performance; describes routine preventive maintenance procedures.
Chapter 4 Troubleshooting	Lists problems and presents step-by-step procedures for how to isolate and eliminate the cause of each problem.
Chapter 5 Service	Provides step-by-step instructions for routine service and parts replacement procedures that users may perform.
Appendix A Specifications	Lists the PDA-100 specifications and installation site requirements.
Appendix B Installation	Describes how to install the PDA-100.
Appendix C Reordering Information	Lists spare parts for the PDA-100.

# 1.4 Safety and Regulatory Information

## 1.4.1 Safety Messages

This manual contains warnings and precautionary statements that can prevent personal injury and/or damage to the PDA-100 when properly followed. Safety messages appear in bold type and are accompanied by icons, as shown below.



Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, may result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury.

IMPORTANT

Indicates that the function or process of the instrument may be impaired. Operation does not constitute a hazard.

Messages d'avertissement en français



Signale une situation de danger immédiat qui, si elle n'est pas évitée, entraînera des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures mineures à modérées. Également utilisé pour signaler une situation ou une pratique qui pourrait gravement endommager l'instrument mais qui n'entraînera pas de blessures.

#### Warnhinweise in Deutsch





Bedeutet unmittelbare Gefahr. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.

Bedeutet eine mögliche Gefährdung. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.



Bedeutet eine mögliche Gefährdung. Mißachtung kann zu kleineren oder mittelschweren Verletzungen führen. Wird auch verwendet, wenn eine Situation zu schweren Schäden am Gerät führen kann, jedoch keine Verletzungsgefahr besteht.

Informational messages also appear throughout this manual. These are labeled NOTE and are in bold type:

NOTE NOTES call attention to certain information. They alert users to an unexpected result of an action, suggest how to optimize instrument performance, etc.

#### 1.4.2 Product Labels

The TUV GS, C, US Mark safety label and the CE Mark label on the PDA-100 indicate that the PDA-100 is in compliance with the following standards: EN 61010-1:2001 (safety), CAN/CSA-C22.2 No. 1010.1-92+A2:97 (safety), UL 61010C-1:2002 R8.02 (safety), and EN 61326:1997+A1:1998 (EMC susceptibility and immunity).

These symbols appear on the PDA-100 or on PDA-100 labels.



Alternating current



Protective conductor terminal



Power supply is on



Power supply is off



Indicates a potential hazard. Refer to the operator's manual for an explanation of the hazard and how to proceed.

## 2.1 Front Panel

Figure 2-1 illustrates the PDA-100 Photodiode Array Detector front panel.

- Press the **Power** switch in the lower left corner for on/off control of the detector power.
- The four LED indicators identify the status of the detector power, the deuterium and tungsten lamps, and the analysis (see Table 2-1).

When the corresponding function is in progress, the LED is lighted. If an error occurs, the LED flashes and an error message is logged in the Chromeleon Audit Trail.



Figure 2-1. PDA-100 Photodiode Array Detector

LED	LED Status	Function
Run	Lighted	Data is being acquired by Chromeleon.
	Flashing	The leak sensor in the drip tray was triggered.
	Off	Data is not being acquired by Chromeleon.
Deuterium	Lighted	The deuterium lamp is on.
	Flashing	A command was issued to turn on the deuterium lamp, but the lamp failed to turn on (see Section 4.3).
		the LED will automatically stop flashing.
	Off	The deuterium lamp is off.
Tungsten	Lighted	The tungsten lamp is on.
	Flashing	A command was issued to turn on the tungsten lamp, but the lamp failed to turn on (see <u>Section 4.3</u> ). If a subsequent attempt to turn on the lamp is successful, the LED will automatically stop flashing.
	Off	The tungsten lamp is off.
Power	Lighted	The detector power is on. Press the <b>Power</b> switch for on/off control of the detector.
	Flashing	The detector failed the start-up routine (see Section 4.2).
	Off	The detector power is off. Press the <b>Power</b> switch for on/off control of the detector.

Table 2-1. PDA-100 Front Panel LEDs

# 2.2 Inside Front Panel

Grasp the PDA-100 front panel by the sides and pull it straight off to access the inside front panel (see Figure 2-2).

INDICATORS       Run     Anguing Strain Time Entry       Deuterium     On     Lamp Failure       Tungstein     On     Lamp Failure	Flow Cell Cover	-, }-
Power	Lamj	
	Cove	r

Figure 2-2. PDA-100 Inside Front Panel

- The flow cell cover protects the cell from dust, ambient light, fluctuations in ambient temperature, and vibration.
- The lamp cover enhances the thermal stabilization of the optical bench. The optical bench is housed in a compartment behind the inside front panel.
- Because the flow cell is located behind a cover, leaks from the cell tubing connections are not immediately apparent. To prevent damage to detector components, the bottom of the optical bench contains a built-in leak tray. A sensor in the sump of the leak tray responds to changes in the index of refraction when wet.

When the leak sensor is activated, it triggers an alarm in the detector and the front panel **Run** LED begins flashing (see <u>Figure 2-1</u>). In addition, the leak is reported to Chromeleon and an error message is logged in the Audit Trail.

# 2.3 Optical System

Figure 2-3 is a schematic of the PDA-100 optical system.

Light from the tungsten lamp is focused through an opening in the internal structure of the deuterium lamp. Light from the tungsten and deuterium lamps is then focused through the flow cell by the source lens. After exiting the cell, the light passes through the spectrograph lens to the filter paddle and is focused into the slit. The light then passes through the slit to the grating, where it is diffracted into the component wavelengths. Measurement of the light occurs at the photodiode array. Each diode measures a narrow portion of the spectrum.



Figure 2-3. PDA-100 Optical System

Optical System Component	Function	
Tungsten Lamp	The light source for visible and near-infrared wavelengths (380 to 800 nm). The tungsten lamp is focused through an opening in the internal structure of the deuterium lamp; this allows the lamps to share the optical axis to the source lens.	
Visible Focus Lens	Focuses the visible light from the tungsten lamp to the through- hole of the deuterium lamp.	
Deuterium Lamp	The light source for UV wavelengths (190 to 380 nm).	
Source Lens	Receives the light from the tungsten and deuterium lamps and focuses it so that the beam passes through the flow cell.	
Flow Cell	The measurement site for sample absorbance. The cell has a flat window on each side.	
Spectrograph Lens	Receives the light from the flow cell and focuses it onto the slit.	
Filter Paddle	The motorized filter paddle in the spectrograph has three positions:	
	• <i>Open</i> = Light passes, unobstructed, along the light path when the holmium oxide filter is moved out of the light path.	
	• <i>Blocked</i> = No light passes along the light path; used to measure the dark signal without turning off the lamps.	
	• <i>Holmium</i> = Places the holmium oxide filter in the light path; used to verify wavelength accuracy.	
Slit	The width of the slit (equivalent to 1 nm) optimizes the optical resolution.	
Grating	Diffracts the light beam into its component wavelengths and directs the light onto the photodiode array.	
Photodiode Array	A series of 1024 photosensitive elements. Each element measures a narrow band of the spectrum. A spectrum is obtained by measuring the light intensity of each wavelength and reporting the results over the selected wavelength range.	

#### 2.3.1 Flow Cell

The PDA-100 flow cell assembly (see <u>Figure 2-4</u>) is available in PEEK or stainless steel. Two built-in heat exchangers—one on the cell inlet tubing and one on the cell handle—help stabilize the temperature of the mobile phase before it enters the cell.



Figure 2-4. Flow Cell Assembly

Flow Cell	Cell Path Length	Cell Volume	Heat Exchanger Volume
PEEK (P/N 056346)	10 mm	13 μL	20 µL
Stainless Steel (P/N 056126)	10 mm	13 µL	9 μL

IMPORTANT

Do not touch the cell windows. If you touch a window, clean it with denatured alcohol and a clean lens tissue.

IMPORTANT

Do not use the PEEK flow cell with normal phase or chlorinated solvents; these solvents will damage the cell.

IMPORTANT

Do not use the stainless steel flow cell with high pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

# 2.4 Electronics



Do not attempt to access the PDA-100 electronics. The components on the PC boards cannot be serviced by the user. If servicing is required, it must be performed by qualified personnel and appropriate electrostatic discharge (ESD) handling procedures must be followed.



Ne retirez aucune des cartes électroniques du détecteur. Aucun des composants sur les cartes ne peut être réparé par l'utilisateur. Toute réparation doit être effectuée par un personnel qualifié utilisant des procédures correctes de décharge électrostatique.



Halten Sie sich von der Elektronik des PDA-100 fern. Die Elektronik kann nicht vom Anwender gewartet werden. Falls ein Service erforderlich ist, ist dieser von qualifiziertem Personal durchzuführen. Dabei müssen die geeigneten Verfahren zur elektrostatischen Entladung (ESD) eingehalten werden.

#### **Power Supply**

The power supply module contains connections to the AC mains and provides regulated, low-voltage power for the PDA-100. Power entry components are shielded and filtered to prevent electromagnetic interference (EMI).



SHOCK AND FIRE HAZARD—The power supply module operates at line potentials. Refer all servicing to qualified personnel.



DANGER D'ÉLECTROCUTION ET D'INCENDIE—Le module d'alimentation électrique fonctionne aux potentiels du secteur. Faites effectuer toutes les réparations par un personnel qualifié.



STROMSCHLAG UND BRANDGEFAHR—Das Modul zur Stromversorgung wird mit Netzspannung betrieben. Der Service darf nur von qualifiziertem Personal durchgeführt werden.

#### Lamp Power Supply Board

The lamp power supply controls the deuterium and tungsten lamps. In addition, it supplies power to the variable speed fan that cools the optical bench.

NOTE The fan speed is controlled automatically. Changes in fan speed may be audible, especially when the lamp selection is changed or the ambient temperature fluctuates.



SHOCK HAZARD—Components used to ignite and operate the deuterium lamp are at high potentials.



DANGER D'ÉLECTROCUTION—Les composants utilisés pour allumer et faire fonctionner la lampe au deutérium sont à des potentiels élevés.



STROMSCHLAGGEFAHR—Die Komponenten zum Zünden und Betreiben der Deuteriumlampe führen Hochspannung. Der Service darf nur von qualifiziertem Personal durchgeführt werden.

#### **Preamp Board**

The photodiode array is connected directly to the preamplifier. The preamp gain is automatically adjusted to compensate for system variables such as lamp intensity. Analog-to-digital conversion of the signal occurs on the Preamp board. Preamp output is directed to the Data Processing board.

#### **Data Processing Board**

The PDA-100 Moduleware resides on the Data Processing board. This board provides the digital signal processing and conversion to log ratios when absorbance is measured.

Also located on the Data Processing board are the connectors for USB and TTL/relay communications, as well as four BNC connectors that provide the analog (recorder) outputs.

# 2.5 Rear Panel

Figure 2-5 illustrates the rear panel of the PDA-100.



Figure 2-5. PDA-100 Rear Panel

#### **Power Entry**

The PDA-100 power entry is socketed for a modular power cord (IEC 320 C13). The detector operates from input voltages over a range of 85 to 265 VAC, 47 to 63 Hz power, and requires a grounded, single-phase power source. The typical input power is 100 W and the maximum line draw is 3.5 amps at 110 VAC (when the detector power is turned on). The appropriate line voltage and frequency are selected automatically.



SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the PDA-100 or connect it to AC power mains without an earthed ground connection.



The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the PDA-100 and is easily accessible.



Operation at AC input levels outside of the specified operating voltage range may damage the PDA-100.



DANGER D'ÉLECTROCUTION—Pour éviter toute électrocution, il faut utiliser une prise de courant avec prise de terre. Ne l'utilisez pas et ne le branchez pas au secteur C.A. sans utiliser de branchement mis à la terre.



Le cordon d'alimentation principal est utilisé comme dispositif principal de débranchement. Veillez à ce que la prise de base soit située/installée près du module et facilement accessible.



STROMSCHLAGGEFAHR—Zur Vermeidung von elektrischen Schlägen ist eine geerdete Steckdose zu verwenden. Das Gerät darf nicht ohne Erdung betrieben bzw. an Wechselstrom angeschlossen werden.



Das Netzkabel ist das wichtigste Mittel zur Stromunterbrechung. Stellen Sie sicher, daß sich die Steckdose nahe am Gerät befindet und leicht zugänglich ist.

#### Fuses

The fuse holder is part of the main power receptacle. The PDA-100 uses two 4.0 amp fast-blow IEC 127 fuses (P/N 954763). For instructions on how to replace the fuses, see <u>Section 5.9</u>.



For continued protection against risk of fire or shock, replacement fuses must be the type and rating specified here.



Pour maintenir la protection contre les risques d'incendie ou d'électrocution, remplacez toujours les fusibles par des fusibles du même type et du même calibre.



Zum Schutz vor Feuer und Stromschlägen müssen beim Sicherungswechsel immer Sicherungen des gleichen Typs und mit gleicher Leistung verwendet werden.

#### **Analog Output Connectors**

The four analog outputs are standard female BNC connectors. The output range is 0 to 1 volt. The full-scale absorbance range can be set to 0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale in Chromeleon. For details about programming the analog outputs, see <u>Section 3.2.2</u>.

The analog outputs continue to output data even after the computer running Chromeleon is turned off or is disconnected from the PDA-100. When the detector power is turned on again, the analog output settings will be either the factory defaults (see <u>Section 3.4</u>) or the settings selected the last time the detector was controlled by the computer.

#### **RELAY/TTL Connectors**

TTL inputs control the following detector functions: autozero (TTL1), UV lamp on/off (TTL3), and visible lamp on/off (TTL4). The TTL2 input is reserved.

The two TTL outputs and two Relay outputs can be programmed as part of a PGM file (see <u>Section 3.2.2</u>) in Chromeleon, or controlled manually through a Control panel in the software. The Relay outputs can be programmed to switch any low-voltage control. The switched current must be less than 200 mA and 42 V peak.

For installation instructions for the Relay and TTL connectors, see Section B.3.8.

#### **USB** Connector

The USB connector allows communication between the PDA-100 and the Chromeleon PC. The PDA-100 is usually connected directly to the computer, using a standard USB A-to-B cable. For USB installation instructions, see <u>Section B.3.2</u> and <u>Section B.3.3</u>.

#### Waste Line

Waste exits the detector via a drain tube connected at the rear panel. The drain tube must be routed downward, below the leak tray. Check periodically to verify that the drain tube is not clogged or bowed upward, and that the tubing remains routed below the leak tray. For installation instructions, see <u>Section B.3.6</u>.

#### IMPORTANT

The drain tube must remain routed below the drain port. If the drain tube is above the drain port, the drip tray may back up and overflow inside the detector.

## 2.6 Chromeleon Software

Chromeleon software is used to control PDA-100 operation. Two modes of software control are available: direct control and automated control.

- With direct control, you select operating parameters and commands from the Chromeleon menu bar, toolbars, and Control panels (see below). Direct control commands are executed as soon as they are entered.
- With automated control, you create a PGM file, which is a list of time-precise control commands for automated operation of the PDA-100. PGM files can be created automatically (with the help of a software wizard) or manually (by editing an existing PGM file).

Chromeleon includes many control commands that are available for all detectors. In addition, there are commands that are exclusively for the PDA-100 (see the table below). These commands can be selected from either the default Control panel or the Commands dialog box. (To open the Commands dialog box, press F8 or select **Command** on the **Control** menu.)

PDA-100 Command	Function
Bandwidth	Sets the bandwidth (the wavelength range at which the chromatogram is measured) of the sample wavelength.
BunchWidth	Determines how many nanometers are averaged when collecting 3D data. Bunching (or averaging) the signals of adjacent wavelengths reduces the size of the 3D data stored.
Data_Collection_Rate	Sets the rate at which Chromeleon collects digital data points from the detector.
NegativeAbsorptionLevel	Adjusts the negative absorbance; this permits the baseline and peaks to be seen even when the mobile phase decreases absorbance.
Offset_Level	Sets the offset applied to the recorder output.
RefBandwidth	Sets the bandwidth of the reference wavelength, if one is selected. If RefWavelength is <b>Off</b> , the RefBandwidth setting has no effect.
RefWavelength	Sets the reference wavelength. If RefWavelength is <b>Off</b> , the reference value is the first data point of the sample wavelength and bandwidth after autozero is performed.

PDA-100 Command	Function		
Rise_Time	Sets how quickly the detector responds to a change in signal.		
Step	Sets the step, which is the time interval between two successively stored data points.		
TTL_Input_Mode	Sets the TTL input signal mode.		
UV_Lamp	Turns the deuterium lamp on and off.		
UVLampAge	Reports (or resets) the number of hours the deuterium lamp has been on.		
UVLampPowerOnSetting	Selects the power-up setting for the deuterium lamp (i.e., whether the lamp is turned on whenever the detector is turned on). The default setting is On.		
Visible_Lamp	Turns the tungsten lamp on and off.		
VisLampAge	Reports (or resets) the number of hours the tungsten lamp has been on.		
VisLampPowerOnSetting	Selects the power-up setting for the tungsten lamp (i.e., whether the lamp is turned on whenever the detector is turned on). The default setting is On.		
Wavelength	Sets the sample wavelength (up to five sample wavelengths can be programmed).		
WavelengthCal	Runs the wavelength calibration procedure to improve wavelength accuracy in the UV range.		
WavelengthCalResult	The result of the last wavelength calibration procedure.		
WavelengthVerTest	Performs the wavelength verification test via the holmium oxide filter.		
WavelengthVerTestResult	The result of the last wavelength verification test.		

## 2.7 Mobile Phases

Solvent quality significantly affects both detection limits and instrument performance. To ensure optimal performance of the PDA-100, observe the following precautions.

**IMPORTANT** Strong bases can etch the silica windows of the flow cell. If the mobile phase is a base, make sure the mobile phase concentration does not exceed 0.1 M. If the concentration of the base is greater than 50 mM, disconnect the separator column and flush the system with DI (deionized) water for 5 minutes at 1.0 mL/min immediately after the analysis. If strong base remains in the cell for 1 or 2 days, the cell windows may need to be replaced (see Section 5.5).

- Prepare all mobile phases with spectroscopy-grade solvents, reagent-grade chemicals, and ASTM Type I (or better) filtered, DI (deionized) water.
- Degas all mobile phases before use and maintain them in a degassed state.
- Mobile phase pH affects not only the retention time of the separation, but the sample absorbance and the background absorbance of the mobile phase. If an analysis employs chemical suppression and compound detection techniques, compare the UV cutoff of the mobile phase before and after the suppressor to see whether it is advantageous to locate the PDA-100 ahead of the suppressor. This location also limits suppressor exposure to backpressure.
- When changing from a buffer to a different operating mobile phase, be sure the solvents are miscible and will not induce precipitation of the buffers. Flush the cell with DI water immediately after the analysis.

# 2.8 Solvent Delivery System

- The pumping system should deliver continuous flow while maintaining a consistent mobile phase composition (if gradient elution is used). Fluctuations in system backpressure may cause baseline disruptions. High sensitivity applications require a smooth, low-pulsation pump. To ensure optimal results, follow the maintenance schedule recommended in the pump user's manual.
- All materials in the solvent delivery system must be fully compatible with the mobile phases required for the analysis. For example, IC and BioLC analyses frequently use mobile phases with extreme pH levels or high salt concentrations. A PEEK pumping system and PEEK flow cell are required when running these mobile phases because the presence of stainless steel in the flow path would contaminate the system.
- Reversed-phase solvents (methanol, moderate concentrations of acetonitrile, etc.) are acceptable for a PEEK system. Other solvents (and high concentrations of acetonitrile) require a stainless steel system.
- All tubing connections should be Teflon<sup>®</sup>, Tefzel<sup>®</sup>, PEEK, stainless steel, or titanium as required for the specific operating pressures and application.
- All mobile phase reservoirs should be compatible with the solvents used with your application. Some plasticizers in blow-molded components leach and are strong UV absorbers.
- Cleanliness is very important. After operating in detection modes that do not require optically clean reagents (such as conductivity), the solvent delivery system may need to be thoroughly cleaned. A basic cleaning procedure is described below; for additional information, refer to the pump user's manual.

#### IMPORTANT

To avoid contamination, always wear gloves when handling pump components in the flow path. Contamination may cause baseline disruptions, spurious peaks, and inconsistent results, especially in sensitive applications.

#### To clean the pump and system between applications:

- 1. Remove the column and the suppressor (if installed).
- 2. Connect the line exiting the injection valve directly to the flow cell.
- 3. Flush the system with DI water at 1.0 mL/min for 20 minutes.

- 4. Flush the system with methanol at 1.0 mL/min for 20 minutes. For very sensitive applications, continue flushing for several hours.
- 5. Flush the system with DI water at 1.0 mL/min for 15 to 20 minutes.
- 6. Reinstall the column (and the suppressor, if removed) and equilibrate the system before resuming operation.

# IMPORTANT If there is a heavy salt build-up on the pistons, remove the pistons and clean them with DI water. Refer to the pump user's manual for instructions. (Cleaning the pistons removes salt crystals that can abrade the piston, thereby causing the main seal to wear out prematurely and allow leaks.)

# IMPORTANT Replace the piston seals if there is a heavy salt build-up on the pistons, if the seals leak, or if the pistons are replaced. Refer to the pump user's manual for instructions.

The PDA-100 Photodiode Array Detector is designed for use with IC (ion chromatography), BioLC<sup>®</sup> (biocompatible liquid chromatography), and HPLC (high-performance liquid chromatography) systems and should not be used for any other purpose. Operation of the PDA-100 in a manner not specified by Dionex may result in personal injury.

If you have a question regarding appropriate usage, contact Dionex before proceeding. In the U.S., call 1-800-346-6390 and select the Technical Support option. Outside the U.S., call the nearest Dionex office.

# 3.1 Getting Started

Before beginning operation of the PDA-100, follow these steps:

- Press the **Power** switch on the front panel to turn on the detector (see Figure 2-1).
- Verify that the **Power** LED is lighted.
- Verify that the **Deuterium** and/or **Tungsten** LEDs are lighted.
- Wait 20 to 30 minutes for the lamps to stabilize. (For sensitive applications work or for detector validation, allow at least 2 hours. After installing a new lamp, allow 8 to 24 hours.)

# 3.2 Routine Operation

#### 3.2.1 Direct Control

In the direct control mode, PDA-100 commands and operating parameters are selected from the Chromeleon menu bar, toolbars, and Control panels. Direct control commands are executed as soon as they are entered. Parameters can be modified at any time during manual data acquisition.

#### NOTE This section provides a brief overview of detector control. For complete operating instructions, refer to the Chromeleon online Help or user's guide.

- 1. Start the Chromeleon Server by double-clicking the Server Monitor icon on the taskbar.
- 2. Start Chromeleon by selecting **Start > Programs > Chromeleon > Chromeleon**. The main window and the Browser will appear.
- In the Browser, expand the Dionex Templates\Panels\Dionex\_IC\ PDA-100(USB) folder. The folder contains two default Control panels:
  - DIONEX\_PUMP\_PDA\_AUTOSAMPLER.PAN, the 2D panel, monitors chromatograms of up to five signals (see Figure 3-1).
  - DIONEX\_PUMP\_PDA3D\_AUTOSAMPLER.PAN, the 3D panel, monitors chromatograms, contour plots, and spectra online (see Figure 3-2).

#### NOTE The 3D Control panel is not available unless Chromeleon includes the PDA Control option.

4. Open a Control panel by double-clicking its name in the list.



Figure 3-1. PDA-100 2D Control Panel Example

- 5. The Control panel is initially unconnected to the PDA-100 timebase. To connect the panel to the timebase, open the **Control** menu and click the name of the timebase from the list at the bottom of the menu.
- 6. Select the operating parameters.
- 7. Turn on the pump. Set the flow rate and mobile phase composition.
- 8. Click the **Acquisition On/Off** toolbar button to begin manual data acquisition.



Figure 3-2. PDA-100 3D Control Panel Example

- 9. Click the **Acquisition On/Off** toolbar button to stop manual data acquisition after the desired run time.
- 10. Data collected during the run may be saved to a sequence. To retrieve the data, select the assigned sample name within the sequence.

## 3.2.2 Automated Control

A PGM file is a list of time-precise control commands, created by the user, for automated operation of the PDA-100. PGM files can be created automatically (with the help of the PGM Wizard) or manually (by editing an existing PGM file).

#### NOTE This section provides a brief overview of detector control. For complete operating instructions, refer to the Chromeleon online Help or user's guide.

- 1. Start the Chromeleon Server by double-clicking the Server Monitor icon on the taskbar.
- 2. Start Chromeleon by selecting **Start > Programs > Chromeleon > Chromeleon**. The main window and the Browser will appear.
- In the Browser, expand the Dionex Templates\Panels\Dionex\_IC\ PDA-100(USB) folder. The folder contains two default Control panels:
  - DIONEX\_PUMP\_PDA\_AUTOSAMPLER.PAN, the 2D panel, monitors chromatograms of up to five signals (see Figure 3-1).
  - DIONEX\_PUMP\_PDA3D\_AUTOSAMPLER.PAN, the 3D panel, monitors chromatograms, contour plots, and spectra online (see Figure 3-2).

#### NOTE The 3D Control panel is not available unless Chromeleon includes the PDA Control option.

#### **Creating a New PGM File**

Before creating a PGM file, see <u>Section 3.3</u> for a description of detector operating parameters. You can optimize the performance of the PDA-100 by careful selection of each operating parameter.

1. Select **File > New**.

A dialog box appears.

2. Select **Program File** and click **OK**.

Program Wizard: Select Timebase Options					
The Program Wizard guides you t To start, select the timebase whe To skip the Program Wizard, pres	hrough the creation of program files. re the program will run. s Finish now.				
Timebase: System_1 Computer: MELAB_1 Protocol: My Computer	My Computer PDA_100 PDA_100 PDA_100B System 1 AD25 Favorites Network Neighborhood				
the list at right.	sk: <u>N</u> ext > Finish Cancel	Help			

The initial Program Wizard screen is displayed.

- 3. Select the **Timebase** where the program will run.
- 4. Click **Next** to continue.

If the selected timebase includes other devices (pump, autosampler, etc.), Program Wizard screens for these devices will be displayed before the PDA-100 screens.
If necessary, click the **Next** button on each screen for another device until the following screen is displayed.

Acquisition Time: From: 0000 min Io: 30.000 min Select the channels to acquire: Image: Select All   Image: Se	Program Wizard: Acq	uisition Optior	1\$			×
	Program Wizard: Acq Acquisition Time: En Select the channels t V3DFIELD VUV_VIS_1 VUV_VIS_2 VUV_VIS_3 VUV_VIS_4 VUV_VIS_5	uisition Optior om: 0.000 r to acquire:	nin I.o. 30.000	min		Select <u>A</u> ll
<u>ABack</u> <u>N</u> ext > Finish Cancel Help		< <u>B</u> ack	<u>N</u> ext >	Finish	Cancel	Help

- 5. Select the data Acquisition Time.
- 6. Check (or clear) the **3DFIELD** check box to determine whether 3D data will be collected.
- 7. Check (or clear) the check box for each 2D (single-wavelength) channel to be collected.
- 8. Click Next.

The following screen is displayed.

Program Wizard: UV Options	×
Data Collection Rate: 2.50 💌 Hz	
<u>R</u> ise Time: 0.5	
Lamps	
UV Lamp: Visible Lamp:	
Un 🔽 On 🔽	
-	
< <u>B</u> ack <u>N</u> ext >	Finish Cancel Help

- 9. Select the **Data Collection Rate** and the **Rise Time**.
  - NOTE You can optimize PDA-100 performance by careful selection of the data collection rate, rise time, and other operating parameters. For details, see <u>Section 3.3</u>.
- 10. Select the Lamp status.
- 11. Click Next.

Program Wizard: 3DFIELD Options	
Wavelength: 191 to 800 [190800 nm]	
Reference	
Wavejength: 750 190800 nm]	
Bandwidt <u>h</u> : 50 1610 nm]	
< <u>B</u> ack <u>N</u> ext > Finish Cancel	Help

The following screen is displayed.

- 12. Select a sample **Wavelength** within the range of the lamp(s) turned on in <u>Step 10</u>.
- 13. Select the Reference Wavelength and Reference Bandwidth.
  - NOTE The total of the Reference Wavelength plus one-half the Reference Bandwidth must be within the range of 190 to 800 nm; if it is not, the reference wavelength will default to the longest wavelength.

In the screen shot above, the selected parameters are acceptable because 750 nm plus 25 nm (one-half of 50 nm) is less than 800 nm.

14. Click Next.

Channel	Wavelength	Bandwidth	RefWavelength	RefBandwidth	Recorder_Range	
UV_VIS_1	210	10	Off	50 🍨	1.0000	
JV_VIS_2	254	10	Off	50	1.0000	
UV_VIS_3	272	10	Off	50	1.0000	
UV_VIS_4	520	10	Off	50	1.0000	
UV_VIS_5	520	10	750	50		

The following screen is displayed.

- 15. Program the single-wavelength channels by selecting the **Wavelength**, **Bandwidth**, **Reference Wavelength**, and **Reference Bandwidth** for each channel.
  - NOTE The settings selected for UV\_VIS\_1 through UV\_VIS\_4 apply to both analog outputs and digital outputs. (UV channel 1 = analog output 1, UV2 = analog output 2, etc.). Specify the Recorder Range (in mAU per volt) for each analog output that will be collected.
  - NOTE When the Reference Wavelength is Off, the Reference Bandwidth is not used. The reference value is the first data point of the sample wavelength and bandwidth after autozero.
- 16. When you have finished selecting PGM file parameters, click Finish.

# 3.3 Optimizing Detector Performance

The performance of the PDA-100 can be optimized by careful selection of key operating parameters. <u>Section 3.3</u> defines these parameters, describes how they interact with each other, offers guidelines for their selection, and includes example chromatograms.

Operating Parameter	Performance Characteristics Affected
Flow cell material	Chemical compatibility
Rise time	Peak resolution, sensitivity, baseline noise
Data collection rate	Peak resolution, disk space
Sample wavelength	Sensitivity, linearity
Bandwidth	Sensitivity vs. baseline noise
Reference wavelength	Baseline drift
Reference bandwidth	Baseline noise, baseline drift
Bunch width	Spectral resolution, peak match, disk space
Step*	Baseline noise, rise time, data collection rate, disk space
Average*	Rise time, data collection rate
Negative absorbance	Peak detection, baseline noise, linearity

The following table summarizes the topics discussed in this section.

\* Set automatically by Chromeleon.

#### **Flow Cell Material**

The flow cell material must be chemically compatible with the mobile phases and analytes of interest.

## IMPORTANT

Do not use the PEEK flow cell (P/N 056346) with normal phase or chlorinated solvents; these solvents will damage the cell.

# IMPORTANT

Do not use the stainless steel flow cell (P/N 056126) with high pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

#### **Rise Time**

Rise time is a measure of how quickly the PDA-100 responds to a change in signal. The rise time settings are 0.1, 0.2, 0.5, 1.0, 2.0 (default), or 5.0 seconds. The selected rise time is applied to all data collected, and to both analog and digital output.

Select a rise time that is approximately 25% of the peak width at half-height of the narrowest peak of interest. A longer rise time allows more averaging of the signal and results in less short-term noise (see Figure 3-3). However, a rise time that is too long may result in reduced peak heights and asymmetric peak shapes. When set correctly, the rise time significantly reduces baseline noise, but reduces peak height only slightly.



Figure 3-3. Effect of Rise Time on Baseline Noise

## **Data Collection Rate**

The data collection rate (or *sample rate*) is the number of data points per second (Hz) at which the computer stores data from the PDA-100. (The detector electronics always collect data at 10.0 Hz.)

The data collection rate settings are 0.25, 0.5, 1.0, 2.0, 2.5 (default), 5.0, or 10.0 Hz. The maximum number of data points is stored at 10.0 Hz. Slower data collection rates store the average of the 10.0 Hz points; for example, a 2.5 Hz rate stores the average of four 10.0 Hz points.

- In general, each peak should be defined by at least 20 data points. For chromatograms with co-eluting peaks or low signal-to-noise ratios, 40 data points per peak is recommended.
- If all peaks are relatively wide, select a slower data collection rate (1.0 Hz, for example).
- If any peaks of interest are less than a few seconds, select a faster data collection rate (5.0 Hz, for example).
- If the data collection rate is too slow, the start and end points of peaks are not accurately determined. If the collection rate is too fast, data files may occupy excessive disk space and post-run analyses may require more processing time.
- A slow data collection rate with a fast rise time may result in a longer system response than indicated by the rise time. For example, a data collection rate of 0.5 Hz and a rise time of 0.5 second results in a system response longer than 2.0 seconds.
- The table below lists the recommended pairings for the rise time and data collection rate.

Rise Time (seconds)	Data Collection Rate (Hz)
0.1	10.0
0.2	10.0
0.5	5.0
1.0	2.5
2.0	2.0
5.0	0.5

#### Sample Wavelength

The PDA-100 measures absorbance over all wavelengths from 190 to 800 nm. The deuterium lamp optimizes the UV range (190 to 380 nm), while the tungsten lamp optimizes the visible range (380 to 800 nm). Set the sample wavelength at the wavelength with the absorbance maxima for the analytes of interest.

When the analyte wavelength maxima are known, the PDA-100 can run five separate wavelengths without being required to collect spectra. Collecting individual wavelengths instead of the spectra offers two advantages: it uses less disk space and it eliminates the need to perform extractions for analyses that do not require spectral data.

If little is known about the analytes in the sample, collect spectra over the full wavelength range (using both lamps). Although this requires more disk space, it provides complete information about the analytes and the spectra can be used to determine peak identity, purity, and wavelength maxima.

# Bandwidth

The bandwidth defines a wavelength range around the wavelength at which the chromatogram is measured. Increasing the bandwidth increases the number of different wavelength signals averaged at each point in time. As the bandwidth increases, baseline noise decreases; however, this may decrease the signal, especially of those peaks that exhibit spectral peak maxima.

Select a bandwidth that provides the resolution required to achieve the desired sensitivity. Selecting a wider bandwidth reduces baseline noise, but may also reduce peak height. For narrow spectral peaks, select narrow bandwidths.

# Example Chromatogram: Selecting the Bandwidth

<u>Figure 3-4</u> illustrates how selecting a wide bandwidth for both the sample and reference wavelengths reduces noise by averaging over a wavelength range. The operating conditions are:

Sample wavelength = 254 nm

Reference wavelength = Off

Flowing water at 1.0 mL/min, 10.35 MPa (1500 psi)



Figure 3-4. Effect of Bandwidth on Baseline Noise

#### **Reference Wavelength**

The reference wavelength is the center wavelength of the reference intensity determination. Absorbance is measured by comparing the reference intensity to the sample intensity. Selecting a reference wavelength implies a reference mode for each of the single wavelengths being collected. There are two types of reference mode: "active" or "off."

• To *select* the active reference mode, enter a wavelength within the selected sample scanning range of the PDA-100. The selected wavelength will be used as the reference wavelength. The reference intensity will vary over time, as the signal intensity changes.

For maximum compensation, select a reference wavelength from the same lamp as the sample wavelength. For example:

If the sample wavelength is 272 nm, you might select a reference wavelength of 325 nm because both wavelengths are within the UV spectrum (190 to 380 nm) of the deuterium lamp.

If the sample wavelength is 372 nm, you might select a reference wavelength of 425 nm because both wavelengths are within the visible spectrum (380 to 800 nm) of the tungsten lamp.

The active reference mode minimizes baseline drift and is ideal for gradient analyses, where the baseline may shift. This mode also helps compensate for fluctuations in ambient temperature caused by air conditioning.

- NOTE When the active reference mode is selected, select a reference wavelength and reference bandwidth in a quiet area of the spectrum where little absorption occurs. Remember that the reference wavelength plus one-half the reference bandwidth must always be within the range of 190 to 800 nm (see page 3-9).
- To *cancel* the active reference mode, enter "Off" as the reference wavelength. The intensity reading at the time of autozero will be used as the reference intensity.

When running an isocratic analysis in a laboratory with stable ambient temperature, operating the detector with the active reference mode turned off ensures the lowest baseline noise. It also ensures that fluctuations in signal intensity in the reference wavelength do not cause additional noise.

#### Example Chromatogram: Selecting the Reference Wavelength

Figure 3-5 shows the results obtained from two different channels within the same run. When the reference wavelength mode is "Off" (i.e., the active reference mode is cancelled), the baseline drift is -120  $\mu$ AU/hour. When the active reference mode is selected, the baseline drift is reduced to -40  $\mu$ AU/hour.

The operating conditions are:

Sample wavelength = 520 nm

Bandwidth = 10 nm

Reference bandwidth = 50 nm (active reference mode only)

Flowing water at 1.0 mL/min, 10.35 MPa (1500 psi)



Figure 3-5. Effect of Reference Wavelength on Baseline Drift

#### **Reference Bandwidth**

The reference bandwidth is analogous to the conventional bandwidth of a channel. The purpose of the reference bandwidth is to average several photodiode signals of the reference wavelength.

It is not necessary to select a reference bandwidth unless you enter a reference wavelength (i.e., you select the active reference mode). If you enter "Off" as the reference wavelength, the reference bandwidth will be the same as the bandwidth.

If you specify a reference bandwidth, it should meet the following criteria:

- Select a reference bandwidth in an area of the spectrum where the sample does not absorb.
- Select a reference bandwidth that is narrow enough not to interfere with nearby compounds. A wider bandwidth will reduce baseline noise, but may increase the chance that the sample absorbs in the reference bandwidth; this will reduce sensitivity.

# Example Chromatogram: Selecting the Sample Wavelength, Reference Wavelength, and Reference Bandwidth

Figure 3-6 shows the spectrum of caffeine. The operating conditions are:

Sample wavelength = 272 nm

Bandwidth = 5 nm

Reference wavelength = 325 nm

Reference bandwidth = 51 nm

These settings calculate the absorbance by the following equation:

 $AU = \log \frac{\text{avg} \left[I_K(300 \text{ to } 350)\right]}{\text{avg} \left[I_K(270 \text{ to } 274)\right]} - \text{offset in AU (at autozero at 272 nm)}$ 

Where:

 $I_{K} = Light intensity$ 

Offset = Absorbance offset at autozero



Figure 3-6. Caffeine Spectrum

## **Bunch Width**

The bunch width setting determines how many nanometers are averaged when collecting 3D data. The default is 1 nm. Selecting a bunch width above 1 nm will reduce the required data storage by approximately the reciprocal. Do not select a bunch width when the spectrum of the compound has a fine structure; the bunch width will reduce the spectral resolution.

#### Step

#### IMPORTANT Chromeleon automatically selects the appropriate step setting. Therefore, it is not necessary for users to select a step setting.

A step is the time interval between two successively stored data points. The smaller the step, the more data points that are recorded and, in general, the more precise the analytical results. However, because collecting more data points requires more storage capacity, the step setting sometimes represents a compromise between the amount of information collected and the file size.

Chromeleon automatically selects the step value that is the inverse of the data collection rate selected by the user. For example, if the data collection rate is 5.0 Hz, the software sets the step to 0.2 second. The following table indicates the step setting automatically selected for each data collection rate.

Data Collection Rate (Hz)	Step (seconds)
0.25	4.0
0.5	2.0
1.0	1.0
2.0	0.5
2.5	0.4
5.0	0.2
10.0	0.1

Step Override

#### IMPORTANT

This section explains how to override the step setting automatically selected by Chromeleon. This information is provided for reference by advanced users only. Be aware that selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems.

In almost all cases, the step setting selected by Chromeleon is the preferred setting. However, it is possible to override this setting by manually changing the step in the PGM file or on the Control panel.

For example, you may want to increase the step time for the 3D field or any of the five absorbance channels. For the 3D field, the step determines the sampling rate at which the detector stores spectra. Ideally, the step is no smaller than the interval suggested for the rise time (see the table in the "Data Collection Rate" section on page 3-13). The advantage of a larger step size is that it reduces the amount of data stored; for example, selecting a step twice as large as that listed in the table reduces the data file size for the 3D field by 50%.

If you select a larger step size when the average parameter is on (see the "Average" section on page 3-21), the system response is a combination of the selected rise time and the average of the data points between steps. If you select a larger step size when the average parameter is off, the selected rise time is valid, but some data points will be skipped.

#### Average

The Average parameter operates in conjunction with the Step parameter (see the "Step" section on page 3-20).

When Average is On, Chromeleon averages the data points between Step intervals, reports that value, and noise is reduced. This setting is recommended for most applications.

When Average is Off, Chromeleon reports the data points at the step interval. The data points between steps are skipped and noise is not reduced.

# IMPORTANT

If average is On and the step size is more than double the rise time, the system response is dominated by the step size. The system response time is approximately the larger of (a) the rise time *or* (b) two times the step size (when average is On).

#### **Negative Absorbance**

Negative absorbance is the result of decreased baseline absorption of the mobile phase after the autozero routine (which usually occurs at the beginning of a run). At the default setting of 1, the PDA-100 becomes saturated when the light transmission is 20% above the autozero level. This results in a flat, truncated baseline.

If desired, increase the negative absorbance level in order to continue seeing the actual baseline and peaks. This is especially helpful when running a gradient application in which the absorbance decreases more than 100 mAU. As the following table indicates, the extent of negative absorbance depends on the wavelength. (The values in this table are intended as guidelines only.)

Negative Absorbance Level	Approximate Negative Absorbance at 210 nm	Approximate Negative Absorbance at 240 nm	Approximate Negative Absorbance at 254 nm
0	-2200 mAU	-50 mAU	-300 mAU
1 (default)	-2300 mAU	-100 mAU	-350 mAU
2	-2600 mAU	-200 mAU	-450 mAU
3	-2600 mAU	-300 mAU	-550 mAU
4	-2700 mAU*	-400 mAU	-630 mAU
5	-2700 mAU*	-500 mAU	-660 mAU
6	-2700 mAU*	-600 mAU	-760 mAU

\* These signals are saturated and will not report a further negative value.

- 1. In Chromeleon, press F8 to display the Commands dialog box.
- 2. Select **UV** and then select **NegativeAbsorptionLevel**. Enter a new setting and click **Execute**.
- 3. Select **Autozero** in the Commands dialog box and click **Execute** to run the autozero routine.
- 4. Select **LampIntensity** in the Commands dialog box to check the intensity of the deuterium lamp. The reading should be above 10 million counts.

If the reading is below 10 million counts, select **UV\_Calibration Intensity** and click **Execute** to calibrate the intensity of the deuterium lamp. Afterward, check the lamp intensity again. If the reading is still too low, clean the flow cell (see Section 5.2) or replace the lamp (see Section 5.7).

5. You may continue increasing the negative absorbance level if the range is required; however, be aware that baseline noise may increase as the negative absorbance increases.

# 3.4 Stand-Alone Analog Operation

<u>Section 3.4</u> describes limited, stand-alone operation of the PDA-100 when the detector is not interfaced to a computer running Chromeleon. In stand-alone operation, TTL inputs on the PDA-100 rear panel can be used to trigger autozero or to turn the lamps on and off. All other detector parameters will remain at their factory default settings and cannot be changed.

## **Factory Default Settings**

The PDA-100 outputs four analog signals at all times, even when the detector is not interfaced to a computer. Data is presented at the analog output channels whenever the detector power is on. A chart recorder or other voltage-measuring device can be used to monitor the channels.

When the PDA-100 power is first turned on, the analog output settings are at the factory defaults listed in the table below.

Channel	Single Wavelength	Bandwidth	Reference Wavelength	Full-Scale Output	% Offset
1	210	10	Off	1.0 AU	10%
2	254	10	Off	1.0 AU	10%
3	272	10	Off	1.0 AU	10%
4	520	10	Off	1.0 AU	10%

The following default settings are also in effect when the power is first turned on:

- Rise time: 2.0 seconds.
- Lamp status: The deuterium and tungsten lamps are on.

## **Stand-Alone Operation**

The table below indicates which PDA-100 parameter each TTL input controls during stand-alone operation.

TTL Input	PDA-100 Parameter
TTL 1 in	Autozero
TTL 2 in	Reserved
TTL 3 in	Deuterium lamp on/off
TTL 4 in	Tungsten lamp on/off

# 3.5 Routine Maintenance

The following maintenance procedures may be performed by users. All other maintenance procedures must be performed by Dionex Service personnel.

- Before shutting down the detector for more than 24 hours, flush the system with deionized (DI) water or methanol for at least 15 minutes at 1.0 mL/min (or maintain a continuous flow at 0.2 mL/min). This rinses corrosive acids, salts, or bases from the flow paths.
- Periodically check the drain tube connected to the leak tray at the bottom of the optical bench. Verify that the tubing is unclogged and that it is routed below the leak tray.
- Periodically check all tubing and fittings and replace as necessary (see <u>Section 5.1</u>). Tubing and fitting components may eventually become loose and leak, or pinched off and plugged. Look for these symptoms:

Decreasing pressure; this may indicate a leak.

A sudden increase in backpressure; this may indicate plugged tubing. (A slow increase in backpressure is normal as the separator column ages.) To troubleshoot, bypass the column and then start the pump; the backpressure should decrease substantially.

Without the column, backpressure is usually less than 0.34 MPa (50 psi), depending on the flow rate and tubing ID. If the backpressure remains high, remove the fittings and tubing from part of the flow path, section by section; starting at the waste line, work back "upstream" until you identify the section causing the substantial backpressure. Replace tubing and fittings as necessary.

- Periodically check the flow cell tubing connections for leaks or restrictions. Replace tubing and fittings as necessary.
- Periodically repassivate stainless steel systems. This is especially important when operating with extreme pH levels (which will shorten the life of the passivation finish) or after a major disassembly. For instructions, see *Passivation of Stainless Steel Systems and Components* (Document No. 031152).

• Monitor baseline noise. If the noise increases above an acceptable level, especially after a lamp(s) has been in service for an extended length of time, replace the lamp.

To check the lamp lifetime:

- 1. In Chromeleon, press F8 to display the Commands dialog box and select UV.
- 2. Select **UVLampAge**. If the deuterium lamp has been in service for more than 1000 hours, replace it (see Section 5.7).
- 3. Select **VisLampAge**. If the tungsten lamp has been in service for more than 2000 hours, replace it (see <u>Section 5.8</u>).
- Check the deuterium lamp intensity approximately every 6 months:
  - 1. In Chromeleon, press F8 to display the Commands dialog box and select UV.
  - 2. Select LampIntensity. The reading should be above 10 million counts.
  - 3. If the lamp intensity reading is too low, calibrate the intensity: In the Commands dialog box, select **UV\_Calibration**, select **Intensity**, and click **Execute**.
  - 4. After calibrating the lamp, check the intensity again. If the reading is still too low, follow the steps in <u>Section 4.7</u> to resolve the problem.
- Run the wavelength verification test for both the deuterium and tungsten lamps approximately every 6 months (see <u>Section 4.15.2</u>). If a lamp fails the test, calibrate the wavelength (see <u>Section 4.15.2</u>) and then run the wavelength verification test again.

If a lamp fails the test again, replace it. For lamp replacement instructions, see <u>Section 5.7</u> or <u>Section 5.8</u> for the deuterium lamp or tungsten lamp, respectively.

This chapter is a guide to troubleshooting problems that may occur while operating the PDA-100 Photodiode Array Detector:

- <u>Section 4.1</u> describes error messages and how to troubleshoot them.
- <u>Section 4.2</u> through <u>Section 4.14</u> describe routine operating problems and how to resolve them.
- <u>Section 4.15</u> describes some of the diagnostic features available in the PDA-100 Moduleware and Chromeleon software.

If you are unable to eliminate a problem, contact Dionex. In the U.S., call 1-800-346-6390 and select the Technical Support option. Outside the U.S., call the nearest Dionex office.

# 4.1 Alarms and Error Conditions

The PDA-100 Moduleware periodically checks the status of certain system parameters. If a problem is detected, it is reported to the Chromeleon PC and displayed in the Chromeleon Audit Trail. Each error message is preceded by an icon that identifies the seriousness of the underlying problem (see the table below). You can change the severity level assigned to a problem whenever appropriate.

lcon	Severity Level	Description
()	Warning	A message is displayed in the Audit Trail, but the current run is not interrupted.
▲	Error	A message is displayed in the Audit Trail, and the system attempts to correct the problem (sometimes by using an alternative parameter).
Stop	Abort	A message is displayed in the Audit Trail, and the running batch is aborted.

The table below lists the PDA-100 error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

Alarms and Error Conditions	Default Severity Level	See
3DFIELD RefWavelength and/or RefBandwidth is out of range.	Error	page 4-4
Acquisition On for 3DFIELD rejectedno PDA Control license found.	Abort	<u>page 4-5</u>
Autozero is already in progress.	Error	page 4-5
Calibration is running.	Abort	page 4-5
Cannot change 3DFIELD parameters while data acquisition is running.	Warning	page 4-5
Cannot set manual parameters for the required operation because the DSP is not ready.	Abort	page 4-6
Can't execute calibration command during data acquisition.	Error	<u>page 4-6</u>
Can't execute diagnostic command during data acquisition.	Error	<u>page 4-6</u>
Can't execute X command during data acquisition.	Error	page 4-6
Command not executed because an Autozero command is already running.	Warning	page 4-6
Data is unavailable.	Abort	page 4-7
Diagnostic is running.	Abort	page 4-7
DSP communication failure.	Abort	page 4-7
Filter wheel sensor could not detect blocked position.	Warning	page 4-8
Filter wheel sensor could not detect open position.	Warning	page 4-8
Invalid step for the 3DFIELD. Valid steps are 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.	Error	<u>page 4-9</u>
Leak sensor error.	Warning	page 4-9
Not enough light to perform operation.	Warning	page 4-9
Over temperature error.	Abort	page 4-10
PDA is running a calibration or diagnostic function. Not ready to accept this command.	Error	<u>page 4-10</u>
The minimum wavelength must be less than the maximum wavelength. Min wavelength = X; max wavelength = X.	Error	<u>page 4-10</u>
The range of wavelength bunching is 1 to 25 nm.	Error	page 4-10

Alarms and Error Conditions	Default Severity Level	See
The range of wavelengths is outside the 190 to 800 nm limit. Wavelength = X; Bandwidth = X.	Error	page 4-11
UV lamp control circuitry error while lamp is off.	Abort	page 4-13
UV lamp error or control circuitry error while lamp is on.	Abort	page 4-11
UV lamp error.	Abort	page 4-11
UV lamp error while attempting to turn off.	Abort	page 4-11
UV lamp error while attempting to turn on.	Abort	page 4-11
UV lamp failed to turn on for the required operation.	Abort	page 4-11
Visible lamp control circuitry error while lamp is off.	Abort	page 4-13
Visible lamp error or control circuitry error while lamp is on.	Abort	page 4-11
Visible lamp error.	Abort	page 4-11
Visible lamp error while attempting to turn off.	Abort	page 4-13
Visible lamp error while attempting to turn on.	Abort	page 4-11

# 3DFIELD RefWavelength and/or RefBandwidth is out of range.

#### To troubleshoot:

Select parameters that meet the following criteria:

$$190 \le \lambda \pm \frac{\mathsf{BW}}{2} \le 800$$

#### Where:

 $\lambda = Reference wavelength$ 

**BW** = Reference bandwidth

#### For example, the following parameters are VALID:

Wavelength = 190 to 800 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are valid because 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is less than 800 nm.

#### The following parameters are NOT VALID:

Wavelength = 190 to 600 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are invalid because 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is greater than 600 nm.

# Stop

# Acquisition On for 3DFIELD rejected--no PDA Control license found.

#### To troubleshoot:

Chromeleon cannot perform digital data acquisition of a 3D data field unless the software includes the PDA Control option. Contact Dionex if you want to order this option.

# Δ

## Autozero is already in progress.

#### To troubleshoot:

This message appears if you select **Autozero** (in the Commands dialog box) when the autozero routine is already in progress. Allow the autozero routine to continue running until completion.

## Stop

## Calibration is running.

#### To troubleshoot:

This message appears if you attempt to issue a command or select a parameter while a calibration procedure is in progress. Wait until the procedure finishes running before making any changes.

# •

# Cannot change 3DFIELD parameters while data acquisition is running.

#### To troubleshoot:

This message appears if you attempt to select a parameter for the 3D field while data acquisition is in progress. Wait until data acquisition is complete, or stop the run and then change the parameters.

# Cannot set manual parameters for the required operation because the DSP is not ready.

#### To troubleshoot:

This message appears if certain commands are issued before the DSP (digital signal processor) is ready to accept them. Turn off the PDA-100 power for 30 seconds, and then turn it on again. If the error message appears again, contact Dionex for assistance.

(1) Can't execute calibration command during data acquisition.

A Can't execute diagnostic command during data acquisition.

## A Can't execute X command during data acquisition.

#### To troubleshoot:

During data acquisition, no other commands can be executed. Wait until data acquisition is completed before attempting to issue any other command.

# Command not executed because an Autozero command is already running.

#### To troubleshoot:

Wait until the autozero routine is completed before attempting to issue any other command.

# Data is unavailable.

#### To troubleshoot:

This message appears if the PDA-100 fails to respond when the computer attempts to communicate with the detector. Shut down the Chromeleon server and turn off the PDA-100 power; wait 30 seconds, and then power up both the server and the detector. If the error message appears again, contact Dionex for assistance.



## Diagnostic is running.

#### To troubleshoot:

This message appears if you attempt to issue a command or select a parameter while a diagnostic routine is running. Wait until the diagnostic routine is completed before making any changes.



## DSP communication failure.

#### To troubleshoot:

This message appears if there is a communication failure between the DSP (digital signal processor) and the CPU. Turn off the PDA-100 power for 30 seconds, and then turn it on again. If the error message appears again, contact Dionex for assistance.

# Filter wheel sensor could not detect blocked position.

## Filter wheel sensor could not detect open position.

A backup paddle alignment routine ensures that the PDA-100 remains functional even if the sensor cannot detect the filter wheel position. Thus, if this is the only error reported, you may continue operation and defer troubleshooting, if desired.

#### To troubleshoot:

- 1. Turn off the PDA-100 power for 30 seconds, and then turn it on again.
- 2. If the error message appears again, check the deuterium lamp intensity:
  - a. In Chromeleon, press F8 to display the Commands dialog box.
  - b. Select **UV**, and then select **LampIntensity**. The intensity reading should be above 10 million counts. If the reading is acceptable, resume normal operation. If the reading is below 10 million counts, see <u>Section 4.7</u>.
- 3. If the error message appears again, there may be a faulty connection to the filter wheel sensor. Contact Dionex for assistance.
  - NOTE The PDA-100 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

# Δ

# Invalid step for the 3DFIELD. Valid steps are 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.

## To troubleshoot:

This message appears if you enter an invalid step setting. Chromeleon automatically selects the step value that is the inverse of the data collection rate. In future, accept the value selected by the software.

# IMPORTANT

Only advanced users should override the step setting selected by Chromeleon. Selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems.

# Leak sensor error.

## To troubleshoot:

Locate the source of the leak. First, tighten (or replace) all liquid line connections. If the connections are made with Dionex ferrule fittings, refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

If the cell is leaking, check that the backpressure on the cell does not exceed the specification (see Section A.5).

Wipe up all liquid and dry the leak sensor before resuming operation.

If the cell leaks after you resume operation, replace it (see Section 5.6).

Not enough light to perform operation.

## To troubleshoot:

Follow the troubleshooting steps in Section 4.7.

## Over temperature error.

#### To troubleshoot:

The fan that cools the optical bench may have malfunctioned or a component on the lamp supply board may have failed. Turn off the PDA-100 power and contact Dionex for assistance.

NOTE Do not attempt to service the PDA-100 electronics components. All repairs of the electronics components must be performed by a Dionex Service Representative.

# PDA is running a calibration or diagnostic function. Not ready to accept this command.

#### To troubleshoot:

Wait until the calibration procedure or diagnostic routine finishes running before attempting to issue any command.

# The minimum wavelength must be less than the maximum wavelength. Min wavelength = X; max wavelength = X.

#### To troubleshoot:

Select a minimum wavelength that is less than the maximum wavelength. Both wavelengths must be within the 190 to 800 nm range.

## A The range of wavelength bunching is 1 to 25 nm.

#### To troubleshoot:

This message appears if you enter an invalid setting for the bunch width. Select a bunch width setting from 1 to 25 nm.

## A The range of wavelengths is outside the 190 to 800 nm limit.

#### To troubleshoot:

The PDA-100 can measure the absorbance spectrum from 190 to 800 nm. Make sure both the minimum and maximum wavelengths are within this range.



Stop

UV lamp error.

UV lamp error or control circuitry error while lamp is on.

- UV lamp failed to turn on for the required operation.
- UV lamp error while attempting to turn on.
- Visible lamp error.



Visible lamp error or control circuitry error while lamp is on.

Visible lamp error while attempting to turn on.



Visible lamp failed to turn on for the required operation.

# To troubleshoot:

 Check that the lamp referred to in the error message is turned on. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted. If necessary, turn on the lamp from either Chromeleon or the TTL input.

- 2. If you recently installed a new deuterium lamp, check that the UV lamp connector wires are routed through the wire guide in the optical bench (see Figure 5-3) and are not pinched under the lamp cover.
- 3. Check the number of hours the lamp has been in service:

In Chromeleon, press F8 to display the Commands dialog box and select **UV**.

If the error message relates to the deuterium lamp, select the **UVLampAge** command.

If the deuterium lamp has been in operation for *more* than 1000 hours, replace it (see Section 5.7). If the error message appears again, contact Dionex for assistance.

If the deuterium lamp has been in operation for *less* than 1000 hours, turn off the lamp for at least 3 minutes and then turn it on again. If the error message reappears, replace the lamp (see Section 5.7). If the error message does *not* appear again, run a manual data acquisition for about 30 minutes. If you notice a shift in the absorbance signal during this time, replace the lamp.

If the error message relates to the tungsten lamp, select the **VisLampAge** command.

If the tungsten lamp has been in operation for *more* than 2000 hours, replace it (see Section 5.8). If the error message appears again, contact Dionex for assistance.

If the tungsten lamp has been in operation for *less* than 2000 hours, turn off the lamp for at least 30 seconds and then turn it on again. If the error message reappears, replace the lamp (see <u>Section 5.8</u>). If the error message does *not* appear again, run a manual data acquisition for about 30 minutes. If you notice a shift in the absorbance signal during this time, replace the lamp.

UV lamp control circuitry error while lamp is off.

- UV lamp error while attempting to turn off.
- Stop

Visible lamp control circuitry error while lamp is off.



Visible lamp error while attempting to turn off.

#### To troubleshoot:

The lamp power supply may have failed or the CPU board may have failed. Contact Dionex for assistance.

NOTE The PDA-100 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

# 4.2 Flashing LED

## • Run LED is flashing

The leak sensor in the drip tray may have been triggered. Check the Chromeleon Audit Trail for a leak-related error message. Find and eliminate the source of the leak.

#### • Deuterium LED is flashing

The lamp did not power on properly. Check the Chromeleon Audit Trail for a lamp-related error message. If necessary, replace the lamp (see Section 5.7).

If the LED flashes again, there may be a bad connection between the lamp power supply and the lamp. Contact Dionex for assistance.

> NOTE The PDA-100 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

## • Tungsten LED is flashing

The lamp did not power on properly. Check the Chromeleon Audit Trail for a lamp-related error message. If necessary, replace the lamp (see <u>Section 5.8</u>).

If the LED flashes again, there may be a bad connection between the lamp power supply and the lamp. Contact Dionex for assistance.

#### NOTE The PDA-100 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

## • Power LED is flashing

If the **Power** LED starts flashing the first time the deuterium lamp is turned on, the detector failed the start-up routine. Follow the steps below to resolve the problem.

- 1. Remove the cell from the optical bench (see <u>Section 5.4</u>).
- 2. In Chromeleon, press F8 to display the Commands dialog box and select UV.
- 3. Select **Autozero** and click **Execute** to run the autozero routine. If this corrects the problem, the **Power** LED will stop flashing and you may replace the cell in the optical bench and resume normal operation. If the **Power** LED continues flashing, go on to <u>Step 4</u>.
- 4. Check the deuterium lamp intensity:
  - a. In Chromeleon, press F8 to display the Commands dialog box.
  - b. Select UV, and then select LampIntensity. The intensity reading should be above 10 million counts. If the reading is acceptable *and* the Power LED is no longer flashing, resume normal operation. If the reading is acceptable but the Power LED is still flashing, contact Dionex for assistance. If the reading is below 10 million counts, follow the troubleshooting steps in Section 4.7.

# 4.3 Lamp Does Not Light or Lamp LED Is Flashing

## • Lamp default setting is not in effect

When the factory default settings are in effect, the deuterium and tungsten lamps turn on automatically each time the detector power is turned on. If the required lamp does not turn on automatically, you can turn it on from Chromeleon or from the TTL input.

To restore the default setting for a lamp:

- 1. Press F8 in Chromeleon to display the Commands dialog box and select UV.
- 2. Select UVLampPowerOnSetting (for the deuterium lamp) and click Execute.
- 3. Select **VisLampPowerOnSetting** (for the tungsten lamp) and click **Execute**.

## • Lamp is old or burned out

Replace the lamp. See <u>Section 5.7</u> or <u>Section 5.8</u> for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

# 4.4 No Detector Response

#### • Detector power not on

Check that the power cord is connected from the PDA-100 rear panel to a power source.

Check that the main **Power** switch is turned on.

Check the fuses and replace them, if necessary (see Section 5.8).

## • Lamp not turned on

Turn on the lamp from either Chromeleon or the TTL input. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.

## • Lamp LED flashing

The corresponding lamp failed to power on properly. In Chromeleon, press F8 to display the Commands dialog box and select **UV**. Check that the command (**UVLampPowerOnSetting** or **VisLampPowerOnSetting**) is **On**.

If the LED continues flashing, replace the lamp. See Section 5.7 or Section 5.8 for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

#### • Detector and/or Control panel not connected to timebase

In the Chromeleon Server Configuration program, add the PDA-100 Control panel to a timebase. For details, see "How to...: Actions in the Server Configuration Program" in the Chromeleon Help.

#### • Windows operating system does not recognize PDA-100

When the computer is turned on, Chromeleon scans the network for all installed instruments. If the PDA-100 power is off, the detector cannot be identified.

If you connect the PDA-100 to the computer before installing Chromeleon, a Windows message box requesting a USB configuration file (cmwdmusb.inf) will appear when you turn on the computer. See <u>Section 4.14</u> for instructions on how to resolve this.

# 4.5 Noisy Baseline

## • Mobile phase or post-column reagent contains light-absorbing impurities

Prepare all mobile phases and reagents with spectro-grade solvents, reagentgrade chemicals, and ASTM Type I (or better) filtered, DI water.

## • Leaking fittings

Locate the source of the leak. Tighten (or replace) all liquid line connections. If the connections are made with Dionex ferrule fittings, refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

# • Insufficient time for system equilibration after turning on lamp(s) or after changing mobile phase strength or composition

The lamps usually require 20 to 30 minutes to stabilize. For sensitive applications work or for detector validation, allow at least 2 hours for the
lamps to stabilize. After installing a new lamp, allow from 8 to 24 hours for the lamp to stabilize.

After changing mobile phases, allow 20 to 30 column volumes for the system to stabilize.

#### • Strongly-retained components from previous analysis are slowly eluting

To elute strongly-retained species, use a stronger mobile phase. Re-equilibrate with the standard mobile phase before resuming routine operation.

Clean the column as instructed in the column manual. If the problem persists, refer to the column manual for troubleshooting guidance.

#### • Plot scale exaggerates baseline noise

If autoscale is selected, even a plot with low noise may fill the screen. Check the Y-axis scale values.

#### • Air bubbles in flow cell

Remove the trapped air (see <u>Section 5.3</u>).

To prevent air from becoming trapped in the cell again, follow these steps:

- Degas mobile phases and post-column reagents by vacuum degassing or sparging with helium.
- Connect a backpressure line to the cell (see <u>Section B.3.5</u>). This increases the backpressure on the cell, thereby shrinking bubbles and allowing them to pass more easily through the cell.

#### • Contaminants in flow cell

Clean the cell (see <u>Section 5.2</u>).

#### • Incorrect lamp selected

If a UV wavelength is selected, make sure the deuterium lamp is on. If a visible wavelength is selected, make sure the tungsten lamp is on. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.

#### • Lamp needs replacement

The light output of the lamp decreases over time. The lower light output may increase noise above an acceptable level, especially if the lamp has been in service for more than 1000 hours (deuterium lamp) or 2000 hours (tungsten

lamp). If this occurs, replace the lamp. See <u>Section 5.7</u> or <u>Section 5.8</u> for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

#### • Lamp incorrectly installed

Make sure that both lamps are fully seated in the optical bench. See <u>Section 5.7</u> or <u>Section 5.8</u> for installation instructions for the deuterium lamp or tungsten lamp, respectively.

#### • Inappropriate reference wavelength

Select a reference wavelength in a quiet area of the spectrum where little absorption occurs. Dionex recommends selecting a reference wavelength below 380 nm for UV applications and above 600 nm for visible applications. For more information, see page 3-16.

#### • Bandwidth is too narrow

Select a wider bandwidth and reference bandwidth, if appropriate for the application. For guidelines when selecting the sample and reference bandwidths, see <u>page 3-14</u> and <u>page 3-16</u>.

#### • Front panel, lamp cover, or flow cell cover is missing

Optical components are sensitive to temperature and light. Do not operate the PDA-100 unless the front panel, lamp cover, and flow cell cover are in place.

#### • Crimped or plugged tubing

Over time, tubing may become pinched off and plugged. Periodically check all tubing and replace as needed (see Section 5.1).

#### • Flow cell installed incorrectly

Check that the cell is inserted straight into the optical bench and is fully seated.

#### • Inappropriate rise time

In general, the rise time should be approximately 25% of the peak width at half-height of the narrowest peak of interest. See <u>page 3-12</u> for more information.

#### • Detector exposed to high vibration

Optical detectors are sensitive to vibrations. Make sure the PDA-100 installation site is vibration-free.

#### • Filter paddle has malfunctioned

If you have recently autozeroed the detector, check the Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see <u>page 4-8</u> for instructions.

#### • Pump needs priming and/or maintenance

If the baseline noise is synchronized with the pump stroke, reprime the pump and clean the piston seals and/or check valves. Refer to the pump user's manual for instructions.

#### • Negative absorbance level is too high

Baseline noise may increase as the negative absorbance increases. If the noise is unacceptable, reduce the negative absorbance level (see <u>page 3-21</u>).

## 4.6 Drifting Baseline

#### • Fluctuations in ambient temperature

Make sure the PDA-100 installation site temperature remains consistent. Allow at least 6 cm (2.4 in) of clearance behind the PDA-100 for ventilation.

Check that the flow cell inlet tubing is routed through the heat exchanger (see Figure 2-4) and is correctly connected to the inside front panel (see Figure 5-2). Check that solvent flow is directed to the cell inlet and out the cell outlet.

Select the "active" reference mode by entering a reference wavelength (see page 3-16.)

#### • Front panel, lamp cover, or flow cell cover is missing

Optical components are sensitive to temperature and light. Do not operate the PDA-100 unless the front panel, lamp cover, and flow cell cover are in place.

#### • Flow cell heat exchanger not in place

Check that the flow cell inlet tubing is threaded through the tubing guide on the lamp cover and secured in place with the tubing bracket (see Figure 5-1).

#### • Lamp not stabilized or burned in

The lamps usually require 20 to 30 minutes to stabilize. For sensitive applications work or for detector validation, allow at least 2 hours for the

lamps to stabilize. After installing a new lamp, allow from 8 to 24 hours for the lamp to stabilize.

After changing mobile phases, allow 20 to 30 column volumes for the system to stabilize.

#### • Leaking flow cell

Tighten fittings. Also, check that the backpressure on the cell does not exceed the specification (see Section A.5).

If the cell continues to leak, replace it (see Section 5.6).

#### • Absorbance in the reference wavelength

Sample absorbance in the selected reference wavelength may cause excessive baseline drift. Select a reference bandwidth in an area of the spectrum where little absorption occurs (see page 3-18).

Dionex recommends selecting a reference wavelength (i.e., active reference mode) for gradient analyses and turning off the reference wavelength for isocratic analyses in a laboratory with a stable ambient temperature (see page 3-16).

## 4.7 Deuterium Lamp Intensity Reading Too Low

#### • Light signal is not reaching the data processor

Check that at least one lamp is turned on. (When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.) If necessary, turn on one of the lamps from either Chromeleon or the TTL input.

#### • Low light throughput

- 1. Remove the cell from the optical bench (see <u>Section 5.4</u>).
- 2. Check the deuterium lamp intensity:
  - a. In Chromeleon, press F8 to display the Commands dialog box.
  - b. Select UV, and then select LampIntensity.

If the lamp intensity reading is above 10 million counts, follow these steps:

- a. In Chromeleon, press F8 to display the Commands dialog box.
- b. Select UV and UV\_Calibration.

- c. Select Intensity and click Execute to calibrate the lamp intensity.
- d. Select **Autozero** (in the Commands dialog box) and click **Execute** to run the autozero routine.
- e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in <u>Section 5.2</u>.
- f. Reinstall the cell in the optical bench.
- g. Autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Dionex for assistance.

If the lamp intensity reading is below 10 million counts, follow these steps:

- a. Check that the flange of the deuterium lamp is aligned with the two locating pins in the optical bench (see Figure 5-3). If the flange is aligned, the lamp is defective and should be replaced (see Section 5.7).
- b. If the error message appears again, contact Dionex for assistance.

#### • Filter paddle has malfunctioned

Check the Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see <u>page 4-8</u> for instructions.

#### • Failure of an electronics component

Turn off the PDA-100 power and contact Dionex for assistance.

NOTE Do not attempt to service the PDA-100 electronics components. All repairs of the electronics components must be performed by a Dionex Service Representative.

## 4.8 Wavelength Calibration Fails

#### • Deuterium lamp may be defective

If the **Deuterium** LED is flashing—or if the LED fails to light—replace the deuterium lamp (see Section 5.6).

If the **Deuterium** LED is lighted but is not flashing, open the PDA-100 Wellness panel and run the wavelength calibration and the wavelength verification test (see <u>Section 4.15.2</u>). If the PDA-100 fails these procedures, remove the flow cell from the detector and repeat both procedures. If the detector fails the wavelength calibration again, contact Dionex for assistance.

#### • Deuterium lamp intensity out of calibration

- 1. Remove the cell from the optical bench (see <u>Section 5.4</u>).
- 2. Check the deuterium lamp intensity:
  - a. In Chromeleon, press F8 to display the Commands dialog box.
  - b. Select UV, and then select LampIntensity.

If the lamp intensity reading is above 10 million counts, follow these steps:

- a. In Chromeleon, press F8 to display the Commands dialog box.
- b. Select UV and UV\_Calibration.
- c. Select Intensity and click Execute to calibrate the lamp intensity.
- d. Select **Autozero** (in the Commands dialog box) and click **Execute** to run the autozero routine.
- e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in <u>Section 5.2</u>.
- f. Reinstall the cell in the optical bench.
- g. Autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Dionex for assistance.

If the lamp intensity reading is below 10 million counts, follow these steps:

- a. Check that the flange of the deuterium lamp is aligned with the two locating pins in the optical bench (see Figure 5-3). If the flange is aligned, the lamp is defective and should be replaced (see Section 5.7).
- b. If the error message appears again, contact Dionex for assistance.

#### • Flow cell contains air bubble or old mobile phase

Flush the cell with the mobile phase for the application.

#### Cell not clear

Clean the cell (see Section 5.2).

If the detector fails the wavelength calibration again, replace the flow cell windows (see <u>Section 5.5</u>). Contaminated flow cell windows can significantly increase baseline noise and cause wavelength calibration to fail.

## 4.9 Wavelength Verification Fails

#### • Detector wavelength requires recalibration

Open the PDA-100 Wellness panel and run the wavelength calibration procedure (see Section 4.15.2).

#### • No solvent flow through cell

If the **Power** LED is flashing (see <u>Figure 2-1</u>), flush the cell with DI water or methanol and then turn off the detector power for 30 seconds. Turn on the power again and verify that the **Power** LED is no longer flashing.

Check the Audit Trail. If the "Not enough light to perform operation." message is displayed, flush the cell with DI water or methanol until the autozero routine can be performed without error.

#### • Lamp(s) needs replacement

Check the number of hours the lamps have been in service:

1. In Chromeleon, press F8 to display the Commands dialog box and select UV.

- 2. Select **UVLampAge**. If the deuterium lamp has been in service for more than 1000 hours, replace it (see Section 5.7).
- 3. Select **VisLampAge**. If the tungsten lamp has been in service for more than 2000 hours, replace it (see <u>Section 5.8</u>).

#### • Solvent absorbs at the wavelength reported for verification

Pump DI water or methanol through the system during the wavelength verification test.

#### • Filter paddle position error

Check the Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see <u>page 4-8</u> for instructions.

## 4.10 No Spectra Collected

#### • Spectral range was not programmed

In Chromeleon, select a spectral range in the PGM file or on the PDA-100 Control panel.

# • Detector and PGM file (or Control panel) connected to different timebases

In Chromeleon, add the detector to the same timebase as the PGM file or the PDA-100 Control panel.

## 4.11 Low Spectral Resolution

#### • Bunch width too wide

Select a bunch width setting of 1 nm, and then check the resolution again.

#### • Bandwidth too wide

Select a bandwidth that includes only the absorbing wavelength.

#### • Incorrect reference wavelength

Select a reference wavelength where minimum absorbance occurs. Make sure the reference wavelength is included in the wavelength range.

#### • Wavelength range too narrow

Make sure the spectrum wavelength range includes both the reference wavelength and reference bandwidth. Make sure the peak absorbance wavelength is not included in the reference bandwidth.

#### • Reference bandwidth too wide

Select a narrower reference bandwidth. The reference bandwidth should not overlap the bandwidth.

## 4.12 Peaks Too Large or Small

#### • Analog output-related problem

Check the recorder and integrator input voltage. The PDA-100 provides a 1 volt full-scale output.

Verify that the selected output range (AU full-scale) is appropriate.

#### • System-level problem

Verify that the sample volume or concentration is appropriate.

Verify that the rise time (see <u>page 3-12</u>) and data collection rate (see <u>page 3-13</u>) are appropriate to capture the narrowest peak.

Prepare fresh post-column reagent.

Verify that the reagent flow rate is correct.

#### • Reference bandwidth too wide

Select a narrower reference bandwidth. The reference bandwidth should not overlap the sample bandwidth.

## 4.13 Poor Peak Shape

#### • Concentration of standard is too high; column is overloaded

Dilute the standard.

Install a sample loop with a smaller volume.

#### • Concentration of standard is too low

Increase the standard concentration.

#### • Inappropriate mobile phase

Do not use a mobile phase that absorbs strongly at the peak absorbance wavelength.

#### • Incorrect sample wavelength selected

If little is known about the analytes in the sample, collect spectra over the full wavelength range (using both lamps). For guidelines to follow when selecting the sample wavelength, see page 3-14.

#### • Incorrect reference wavelength selected

Select a different reference wavelength. For guidelines to follow when selecting the reference wavelength, see <u>page 3-16</u>.

#### • Bandwidth too wide

Select a bandwidth that provides the resolution required to achieve the desired sensitivity. Narrow spectra peaks require narrow bandwidths. A wider bandwidth reduces baseline noise, but may reduce peak height.

#### • Reference bandwidth too wide

Select a bandwidth that provides the resolution required to achieve the desired sensitivity. A wider bandwidth reduces baseline noise, but may increase the chance that the sample absorbs in the reference bandwidth, thus causing reduced sensitivity and peak height.

#### • Cell flow is reversed

Check that the flow cell inlet tubing is routed through the heat exchanger (see Figure 2-4) and is correctly connected to the inside front panel (see Figure 5-2). Check that solvent flow is directed to the cell inlet and out the cell outlet.

## 4.14 Faulty USB Communication

#### • PDA-100 not recognized by Windows operating system

Dionex strongly recommends installing Chromeleon *before* connecting the PDA-100 to the computer. When the software is installed first, USB driver information is loaded automatically.

If you connect the PDA-100 to the computer before installing Chromeleon, a Windows message box requesting a USB configuration file (cmwdmusb.inf) will appear when you turn on the computer. Follow these steps to resolve the problem:

- 1. Click **Cancel** in the Windows message box.
- 2. Turn off the power to the PDA-100 and unplug the USB cable from the computer.
- 3. Install Chromeleon.
- 4. Reconnect the USB cable to the computer and turn on the power to the PDA-100. Windows will now automatically recognize the detector.

## 4.15 Diagnostics

### 4.15.1 Moduleware Run-Time Diagnostics

The PDA-100 Moduleware periodically checks the status of certain system parameters. All problems are reported to the computer and logged in the Chromeleon Audit Trail. The Audit Trail includes the date, time, and severity level for each problem the PDA-100 reports. There are two ways to review this information:

- Check the Audit Trail displayed in the PDA-100 Control panel, or
- Retrieve the Audit Trail from a sequence by selecting the Audit Trail folder in the timebase and the file for that date.

### 4.15.2 Software Diagnostics and Calibration

Chromeleon includes comprehensive diagnostics for the PDA-100. This section describes some of the most frequently used diagnostic procedures. For more information, refer to the Chromeleon Help or user's guide.

#### • Lamp age

**Purpose:** Indicates the total number of hours that each lamp has been turned on in its lifetime.

To reset the lamp age:

- a. After installing a new lamp, press F8 to display the Commands dialog box.
- b. Select UV.
- c. Select the **UVLampAge** command (for the deuterium lamp) or **VisLampAge** command (for the tungsten lamp).
- d. Reset the number to zero.
- e. Click Execute.
  - NOTE When the detector power is initially turned on, the lamp age already indicates some elapsed time; this is the time that was required for factory calibration and test procedures.

#### • Wavelength calibration

**Purpose:** Improves wavelength accuracy in the UV range by reassigning new wavelengths to each pixel, based on the emission spectrum from the deuterium lamp. The results of both the initial calibration and the last calibration are stored in the calibration log. You can view the results at any time.

- a. Verify the following conditions: no solvent is flowing through the cell, the background absorbance is low, and there are no bubbles in the light path.
- b. In the Browser, navigate to the following folder: **Dionex Templates/Panels/Dionex\_IC\PDA-100(USB)\Wellness**.
- c. Open the PDA-100 Wellness panel.
- d. Click the **wavelength** script button under **Calibration**. The detector will perform the wavelength calibration routine and upload the result (pass or fail) to Chromeleon.

#### Wavelength verification

**Purpose:** Tests the accuracy of the selected wavelength. The result of the last wavelength verification test is stored in the calibration log and can be retrieved at any time.

- a. In the Browser, navigate to the following folder: **Dionex Templates/Panels/Dionex\_IC\PDA-100(USB)\Wellness**.
- b. Open the PDA-100 Wellness panel.
- c. Click the **wavelength verification** script button under **Diagnostics**. The detector will perform the wavelength verification test and upload the result (pass or fail) to Chromeleon.

This chapter describes routine service procedures that users may perform. All procedures not described here must be performed by Dionex Service personnel. To contact Dionex in the U.S., call 1-800-346-6390 and select the Technical Support option. Outside the U.S., call the nearest Dionex office.

NOTE All repairs of the PDA-100 electronics must be performed by Dionex Service personnel.



The Data Processing board contains a lithium battery. If the board is replaced, dispose of the used battery according to local regulations.

Before replacing any parts, review the troubleshooting information in <u>Chapter 4</u> to isolate the cause of the problem.

IMPORTANT

Substituting non-Dionex parts may impair the performance of the PDA-100, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

# 5.1 Liquid Leaks

The PDA-100 is plumbed with the components listed below. For tightening requirements, refer to *Installation of Dionex Ferrule Fittings*.

Note that the tubing connected to the flow cell heat exchangers (see <u>Section 2.3.1</u>) cannot be replaced. If a leak (at the flow cell inlet, for example) cannot be eliminated by tightening the fitting, replace the entire cell.

Component	PDA-100 with PEEK Flow Cell	PDA-100 with Stainless Steel Flow Cell
Tubing, 0.25-mm (0.010-in) ID	P/N 042690	P/N 019245
Reducing Union Fitting	P/N 055903	P/N 055902
Union Fitting	P/N 042627	P/N 042627
Ferrules	P/N 043276	P/N 010262
Nuts	P/N 043275	P/N 010261

# 5.2 Cleaning the Flow Cell

# Film deposits on the flow cell windows may cause excessive baseline noise or high absorbance offset.

- 1. Pump methanol through the cell for 20 to 30 minutes at 1.0 mL/min.
- 2. Pump DI (deionized) water through the cell for 20 to 30 minutes at 1.0 mL/min.

#### If the procedure above does not clean the cell, follow these steps:

- 1. Disconnect the liquid lines from the cell in and out connections.
- 2. Connect a luer adapter to the cell in connection.
- 3. Using a syringe, flush the cell with a succession of at least 10  $\mu$ L each of DI water, acetone, and 3 M HNO<sub>3</sub>.
- 4. Reconnect the cell liquid lines.
- 5. Flush the cell with DI water for 20 to 30 minutes at 1.0 mL/min.

## 5.3 Removing Trapped Air from the Flow Cell

# Trapped air bubbles in the flow cell may cause pulsations of the baseline or random noise and low readings.

Flush the cell with methanol for 20 minutes. If this does not remove the trapped air, follow these steps:

- 1. Disconnect the waste line from the cell. Also disconnect the backpressure device.
- 2. Using a small syringe, push 3 to 5 mL of methanol through the cell. Or, bypass the column and pump methanol through the system at 1.0 mL/min.
- 3. Reconnect the waste line and backpressure device.

## 5.4 Removing the Flow Cell from the Optical Bench

Remove the flow cell from the optical bench before replacing the cell windows (see <u>Section 5.5</u>), installing a new cell (see <u>Section 5.6</u>), or inspecting the cell for leaks or obstructions.

- 1. Grasp the detector front panel by the sides and pull straight off to remove.
- 2. Disconnect the tubing connected to the flow cell inlet and outlet (see Figure 5-1).
- 3. Remove the flow cell cover from the inside front panel (see Figure 2-2).
- 4. Remove the tubing bracket from the lamp cover (see <u>Figure 5-1</u>).
- 5. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (see <u>Figure 5-1</u>).





Figure 5-1. Removing the Flow Cell from the Optical Bench (Flow cell cover not shown)

# 5.5 Replacing the Flow Cell Windows

Contaminated flow cell windows can significantly increase baseline noise and cause wavelength calibration to fail. If cleaning the cell (see <u>Section 5.2</u>) does not eliminate these problems, replace both silica windows.

The flow cell windows replacement kit (P/N 060498) contains all of the parts required for this procedure. The kit is optional and must be ordered separately from the PDA-100.

- 1. Remove the flow cell from the optical bench (see Figure 5-1) and place the cell on a clean work surface.
- 2. Put on a pair of latex gloves.
- 3. Using the windows replacement tool (P/N 060497), remove one of the window nuts from the flow cell (see Figure 5-2). (It does not matter which window nut is removed first.)
- 4. Remove and discard the old window.



Figure 5-2. Replacing the Flow Cell Windows (Light inlet side shown)

5. Use tweezers to carefully remove the O-ring from the groove (see Figure 5-2). **Do not scratch the groove.** 

# IMPORTANT Scratches in the groove will prevent the new O-ring from sealing properly and will cause leaks.

- 6. Install a new O-ring (P/N 054136) in the groove.
- 7. Place a new window (P/N 054135) into the counterbore in the window nut. Screw the window into place.
- 8. Using the windows replacement tool, tighten the window nut until snug and then turn an additional one-quarter turn.
- 9. Repeat <u>Step 3</u> through <u>Step 8</u> to install the second window.
- 10. Connect the flow cell to the pump and pressurize to 2.07 MPa (300 psi). Check for leaks around the windows. If there is any leakage, tighten the window nut(s) with the windows replacement tool until the leak stops. Dry the cell thoroughly with a lint-free, optical-grade tissue.
- 11. Reinstall the flow cell in the optical bench.

## 5.6 Replacing the Flow Cell

Replace the flow cell if a colored substance stains the cell interior and cleaning the cell (see <u>Section 5.2</u>) does not remove the stain, or if excessive backpressure cracks the silica windows and you do not want to replace them (see <u>Section 5.5</u>).

- 1. Remove the flow cell from the optical bench (see Section 5.4).
- 2. Squeeze the handle of the new flow cell (PEEK, P/N 056346; stainless steel, P/N 056126) and insert it straight into the optical bench. Release the handle, and then pull it forward slightly. If there is a click, the cell is properly mounted. If there is no click, rotate the handle slightly just until the cell clicks into place.
- 3. Thread the flow cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket (see Figure 5-1).
- 4. Thread the flow cell outlet tubing through the slot on the side of the flow cell cover. Replace the cover and reconnect the cell inlet and outlet tubing.
- 5. Push the detector front panel into place.

# 5.7 Replacing the Deuterium Lamp

- 1. Stop the Chromeleon server.
- 2. Turn off the PDA-100 power switch.



The deuterium lamp emits UV radiation that is harmful to the eyes. Always turn off the detector power before removing the lamp cover.



La lampe deuterium emettet de rayons ultraviolets, qui sont dangeroux pour les yeux. Voulez vous etteindre le detecteur avant d'enlever le couvercle de la lampe.



Die Deuteriumlampe gibt UV-Strahlung ab; diese ist schädlich für die Augen. Schalten Sie den Detektor immer aus, ehe Sie die Abdeckung von der Lampe entfernen.



The lamp housing and base may be hot to the touch, especially after the lamp has been in operation for a long time. Wait until the lamp has cooled down before continuing.



La lampe et la base de la lampe peuvent être chaudes au toucher, particulièrement après que la lampe a été allumée pendant longtemps. Attendez que la lampe ait refroidi avant de continuer.



Das Lampengehäuse und die Lampenbefestigung können sehr heiß werden, besonders wenn die Lampe längere Zeit in Betrieb war. Warten Sie, bis die Lampe abgekühlt ist, bevor Sie diese berühren.

- 3. Grasp the detector front panel by the sides and pull straight off to remove.
- 4. Remove the tubing bracket from the lamp cover (see Figure 5-1).
- 5. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (see Figure 5-1).
- 6. Remove the flow cell cover from the inside front panel (see Figure 5-1).
- 7. Move the flow cell tubing out of the way. Remove the four screws holding the lamp cover in place, and then remove the cover and set it aside.
- 8. Squeeze the clip on the UV lamp connector and disconnect it from the 6-pin bulkhead connector on the side wall (see Figure 5-3).



Figure 5-3. Lamp Connections

9. Loosen the three screws in the deuterium lamp flange (see <u>Figure 5-3</u>) and pull the lamp out of the optical bench.

# NOTE The screws are captive in the lamp base and do not need to be removed.

- 10. Inspect the new deuterium lamp (P/N 939016T) for fingerprints and dust. If necessary, clean the lamp with IPA (isopropyl alcohol).
- 11. Line up the flange of the new lamp with the two locating pins and three threaded holes in the optical bench (see <u>Figure 5-3</u>). When the flange is aligned, gently push the lamp into the optical bench.
- 12. When the lamp is fully seated, tighten the screws in the flange with a screwdriver.

## IMPORTANT To ensure proper performance, the lamp must be fully seated.

- 13. Squeeze the clip on the UV lamp connector and insert it into the 6-pin bulkhead connector on the side wall. Route the wires through the wire guide in the optical bench (see Figure 5-3).
- 14. Replace the lamp cover.

#### IMPORTANT

Do not pinch the UV lamp connector wires under the lamp cover.

- 15. Thread the flow cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket.
- 16. Replace the flow cell cover and reconnect the inlet and outlet tubing.

## IMPORTANT Do not pinch the tubing under the flow cell cover.

- 17. Replace the detector front panel.
- 18. Turn on the detector power.
- 19. Start the Chromeleon server.
- 20. Reset the lamp lifetime:
  - a. Press F8 to display the Commands dialog box.
  - b. Select UV.
  - c. Select UVLampAge.
  - d. Reset the lamp age to zero.
  - e. Click Execute.

# NOTE It is normal for the baseline to drift for several hours after a new deuterium lamp is installed.

21. Run the wavelength calibration and wavelength verification test for the new lamp (see Section 4.15.2).

## 5.8 Replacing the Tungsten Lamp

- 1. Stop the Chromeleon server.
- 2. Turn off the PDA-100 power switch.



The lamp housing and base may be hot to the touch, especially after the lamp has been in operation for a long time. Wait until the lamp has cooled down before continuing.



La lampe et la base de la lampe peuvent être chaudes au toucher, particulièrement après que la lampe a été allumée pendant longtemps. Attendez que la lampe ait refroidi avant de continuer.



Das Lampengehäuse und die Lampenbefestigung können sehr heiß werden, besonders wenn die Lampe längere Zeit in Betrieb war. Warten Sie, bis die Lampe abgekühlt ist, bevor Sie diese berühren.

- 3. Grasp the detector front panel by the sides and pull straight off to remove.
- 4. Remove the tubing bracket from the lamp cover (see Figure 5-1).
- 5. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (see Figure 5-1).
- 6. Remove the flow cell cover from the inside front panel (see Figure 5-1).
- 7. Move the flow cell tubing out of the way. Remove the four screws holding the lamp cover in place, and then remove the cover and set it aside.
- 8. Squeeze the clip on the visible lamp connector and disconnect it from the 4pin bulkhead connector on the side wall (see Figure 5-3).
- 9. Use a screwdriver to loosen the tungsten lamp screws, and then pull the mounting assembly away from the optical bench.

# NOTE The screws are captive in the lamp base and do not need to be removed.

10. Inspect the new tungsten lamp (P/N 056123T) for fingerprints and dust. If necessary, clean the lamp with IPA (isopropyl alcohol).

11. Carefully insert the new lamp into the optical bench and tighten the three screws that secure the lamp to the optical bench. (The replacement lamp is pre-aligned in the mount.)

## IMPORTANT To ensure proper performance, the lamp must be fully seated.

- 12. Squeeze the clip on the Vis lamp connector and insert it into the 4-pin bulkhead connector on the side wall. Route the wires through the wire guide in the optical bench (see Figure 5-3).
- 13. Replace the lamp cover.

## IMPORTANT Do not pinch the Vis lamp connector wires under the lamp cover.

- 14. Thread the cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket
- 15. Replace the flow cell cover and reconnect the inlet and outlet tubing.

### IMPORTANT Do not pinch the tubing under the flow cell cover.

- 16. Replace the detector front panel.
- 17. Turn on the detector power.
- 18. Start the Chromeleon server.
- 19. Reset the lamp lifetime:
  - a. Press F8 to display the Commands dialog box.
  - b. Select UV and select VisLampAge.
  - c. Reset the lamp age to zero.
  - d. Click Execute.
    - NOTE Before resuming routine operation, allow 20 to 30 minutes for the lamp to stabilize.

## 5.9 Replacing the Main Power Fuses



HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the PDA-100.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du PDA-100.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der Rückseite des PDA-100.

- 1. Turn off the PDA-100 power switch.
- 2. Disconnect the power cord from the PDA-100 rear panel.
- 3. The fuse holder is part of the main power receptacle (see Figure 5-4). Note the recessed lock on each side of the fuse holder.

Using a small screwdriver (or your fingernails), push one of the locks toward the center to release it. (The fuse holder will pop out slightly when the lock is released.) Repeat for the other lock. Pull the fuse holder straight out of the compartment.



Figure 5-4. Main Power Fuse Holder

- 4. Replace the two fuses in the holder with new 4.0 amp fast-blow IEC 127 fuses (P/N 954763). Dionex recommends always replacing both fuses.
- 5. Reinsert the fuse holder into the compartment. (The fuse holder is keyed to fit only in the correct orientation.) Apply pressure evenly against the holder until the locks are engaged. When correctly installed, the fuse holder is flush against the rear panel.
- 6. Reconnect the power cord and turn on the power.

# A.1 Electrical

Main Power	85 to 265 VAC, 47 to 63 Hz (auto-sensing; no adjustment
	necessary)
	Typical input power: 100 W
	Maximum line draw: 3.5 A at 110 VAC at power-up
Fuses	Two 4.0 amp fast-blow IEC 127 fuses (P/N 954763)

# A.2 Physical

Dimensions	Height: 17.5 cm (6.9 in)
	Width: 38.6 cm (15.2 in)
	Depth: 49.0 cm (19.3 in)
	Clearance required behind detector: 6 cm (2.4 in)
Weight	16 kg (35 lbs)
Decibel Level	<50 db (at "A WEIGHTING" setting) during routine operation 56 db (at "A WEIGHTING" setting) at power-up

# A.3 Environmental

Operating Temperature	4 to 40 °C (40 to 104 °F) constant temperature
Operating Humidity	5% to 95% relative humidity, noncondensing

## A.4 Detector

Optical System	Single-beam, reverse-optics design with concave holographic grating	
Light Sources	Deuterium lamp (30 W) for ultraviolet spectrum analysis Tungsten lamp (15 W) for visible spectrum analysis	
Photodiode Array	1024-element photodiode array bench	
Wavelength Range	190 to 800 nm	
<b>Pixel Resolution</b>	0.7 nm	
Wavelength Accuracy	$\pm 1$ nm, self-calibration with deuterium lines, verification with built-in holmium oxide filter	
Noise	$<\pm$ 10 $\mu AU$ at 254 nm, 2-second rise time, and flowing water $<\pm$ 15 $\mu AU$ at 520 nm, 2-second rise time, and flowing water	
Drift	<1000 µAU/hour (after warm-up)	
Linearity	Deuterium lamp: >2 AU Tungsten lamp: >2 AU	
Analog Outputs	Four; 0 to 3 AU, selectable, 1000 mV range	
Voltage Out	1 V output	
Analog Output Range	0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale	
Control and Data Evaluation	Provided by Chromeleon software; the Chromeleon PC and the detector are connected through USB (Universal Serial Bus)	

## A.5 Flow Cells

Cell Body	PEEK or stainless steel		
Volume and Optical Path Length	PEEK cell: 13 $\mu$ L volume; 10 mm (0.394 in) path length Stainless steel cell: 13 $\mu$ L volume; 10 mm (0.394 in) path length		
Maximum Operating Pressure	PEEK cell: 2 MPa (300 psi) Stainless steel cell: 3 MPa (500 psi)		
Chemical Compatibility	• Do not use bases stronger than 0.1 M; they will etch the silica windows of the flow cells.		
	• Do not use normal phase or chlorinated solvents with the PEEK flow cell; they will damage the cell.		
	• Do not use high pH solutions with the stainless steel flow cell; they may cause corrosion, contamination, and metal		

# A.6 Heat Exchangers

NOTE Each flow cell assembly contains two built-in heat exchangers (see Section 2.3.1).

Maximum	PEEK cell: 2 MPa (300 psi)		
Operating	Stainless steel cell: 3 MPa (500 psi)		
Pressure			
Volume	PEEK cell: 20 μL Stainless steel cell: 9 μL		

leaching.

## **B.1 Facilities Required**

- Make sure the PDA-100 installation site meets the power and environmental specifications listed in Appendix A.
- Optical detectors are sensitive to vibrations. Install the PDA-100 on a sturdy workbench in an environment that is vibration-free.
- Allow at least 6 cm (2.4 in) behind the PDA-100 for power connections and ventilation. Do not block air flow to the detector; during operation, room air must be inducted into the detector to cool the electronics and dissipate the heat generated by the lamps.
- Optical detectors are sensitive to changes in temperature and light. Protect the PDA-100, the columns, and all tubing connections from drafts. Do not operate the PDA-100 unless the front panel, lamp cover, and flow cell cover are in place.

## B.2 Unpacking the PDA-100

• Carefully remove the PDA-100 from the shipping container.

#### IMPORTANT

Lift the PDA-100 by the side or bottom only; the front panel of the detector snaps into place and must not be used for lifting.

- Do not turn on the PDA-100 unless it is at room temperature (4 to 40 °C; 40 to 104 °F). If the detector is moved from a cold environment to a warm environment, wait at least 1 hour for condensation to evaporate before turning on the mains power.
- The PDA-100 Ship Kit contains the parts required to install the detector:

Product Line	PDA-100	Flow Cell Material	Ship Kit
ICS-2500	P/N 060973	PEEK	P/N 060977
BioLC	P/N 060975	PEEK	P/N 060977
SUMMIT <sup>TM</sup>	P/N 060926	Stainless Steel	P/N 060708

## **B.3** Installation

## B.3.1 Installing Chromeleon Software

NOTE Dionex strongly recommends installing Chromeleon before connecting the PDA-100 to the computer. When Chromeleon is installed first, USB driver information is loaded automatically.

Before going on to <u>Section B.3.2</u> or <u>Section B.3.3</u>, verify that the following tasks have been completed:

- Chromeleon is installed on the computer.
- The Chromeleon license is installed.

Detailed installation instructions are available in two formats. A printed manual is shipped with Chromeleon and an Adobe PDF file is provided on the Chromeleon CD-ROM.

## B.3.2 Connecting the PDA-100 to the Chromeleon PC

The PDA-100 is usually connected directly to a USB port on the computer running Chromeleon. If a USB port is not available on the PC, connect the PDA-100 to an external USB hub (see <u>Section B.3.3</u>).

- 1. Plug the "A" connector of a USB cable (P/N 960777) into the USB port on the computer (see Figure B-1).
- 2. Plug the "B" connector of the USB cable into a USB receptacle on the PDA-100 rear panel.



Figure B-1. PDA-100 Connected Directly to the Chromeleon PC

### B.3.3 Connecting the PDA-100 to an External USB Hub

IMPORTANT The USB standard limits the USB cable length to 5 meters (5.5 yds). Each USB device can be separated from the PC by no more than five hubs. Thus, if five hubs are installed, each USB device can be located no more than 30 meters (32 yds) from the PC.

# **IMPORTANT** Carefully secure all USB cables, the USB hub, and the hub power cable so that they cannot be accidentally disconnected.

If the PDA-100 cannot be connected directly to the Chromeleon PC (see <u>Section B.3.2</u>), connect it to an external USB hub (P/N 060392) and then connect the hub to the computer. Note that the hub is an option that must be ordered separately from the PDA-100.

- 1. Plug the "A" connector of a USB cable (P/N 960777) into a port on the external USB hub (P/N 060392). Plug the "B" connector of the cable into the USB receptacle on the PDA-100 rear panel (see Figure B-2).
- 2. Plug the "A" connector of a USB cable (P/N 960777) into a USB port on the computer. Plug the "B" connector of the cable into a port on the USB hub (see Figure B-2).



Figure B-2. Three USB Devices Connected to a USB Hub

### **B.3.4** Connecting the Power Cord



SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the PDA-100 or connect it to AC power mains without an earthed ground connection.



The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the PDA-100 and is easily accessible.



Operation at AC input levels outside of the specified operating voltage range may damage the PDA-100.



DANGER D'ÉLECTROCUTION—Pour éviter toute électrocution, il faut utiliser une prise de courant avec prise de terre. Ne l'utilisez pas et ne le branchez pas au secteur C.A. sans utiliser de branchement mis à la terre.



Le cordon d'alimentation principal est utilisé comme dispositif principal de débranchement. Veillez à ce que la prise de base soit située/installée près du module et facilement accessible.



STROMSCHLAGGEFAHR—Zur Vermeidung von elektrischen Schlägen ist eine geerdete Steckdose zu verwenden. Das Gerät darf nicht ohne Erdung betrieben bzw. an Wechselstrom angeschlossen werden.



Das Netzkabel ist das wichtigste Mittel zur Stromunterbrechung. Stellen Sie sicher, daß sich die Steckdose nahe am Gerät befindet und leicht zugänglich ist.

Connect a modular power cord (IEC 320 C13) from the PDA-100 main power receptacle (see <u>Figure B-3</u>) to a grounded, single-phase power source. Use the front panel **Power** switch to turn the detector on and off.



Figure B-3. PDA-100 Rear Panel

## B.3.5 Connecting the Flow Cell

- IMPORTANT Do not touch the cell windows. If you touch a window, clean it with denatured alcohol and a clean lens tissue.
- IMPORTANT Do not use the PEEK flow cell with normal phase or chlorinated solvents; these solvents will damage the cell.
- IMPORTANT Do not use the stainless steel flow cell with high pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

The flow cell is installed in the PDA-100 at the factory. Follow the steps below to complete the cell connections.

- 1. Grasp the PDA-100 front panel by the sides and pull straight off to remove.
- 2. Connect the cell inlet tubing to the reducing union fitting (P/N 055903 for PEEK cell; P/N 055902 for stainless steel cell) on the heat exchanger tubing (see Figure B-4).

- Connect a backpressure line to the cell outlet connection (see <u>Figure B-4</u>). The backpressure line will provide enough restriction to generate the small amount of backpressure (0.14 to 0.34 MPa; 20 to 50 psi) required to help prevent bubbles from forming in the cell.
  - a. Locate the following items in the PDA-100 Ship Kit: 0.25-mm (0.010-in) ID tubing (P/N 052306), two ferrule fittings (P/N 043276), and two 10-32 bolts (P/N 043275).
  - b. The backpressure line should be 1 meter long for a flow rate of 1.0 mL/min, 2 meters for a flow rate of 0.5 mL/min, and so on. Cut the tubing to the required length.
  - c. Install a ferrule fitting and 10-32 bolt on each end of the tubing.
  - d. Install the backpressure line after the cell outlet. Use a union (P/N 042627) to connect the backpressure line to the waste line.
- 4. Thread the cell outlet tubing through the slot near the bottom of the front panel, thread the cell inlet tubing through the slot on the right side, and push the front panel into place.
- 5. Connect the cell inlet tubing to the separator column outlet.

IMPORTANT The backpressure on the cell must not exceed 2 MPa (300 psi) for a PEEK cell or 3 MPa (500 psi) for a stainless steel cell. Backpressure above this level will damage the heat exchanger.



Figure B-4. Flow Cell Connections
#### B.3.6 Connecting the Waste Line

Connect the waste line (P./N 054779) to the PDA-100 rear panel (see <u>Figure B-3</u>). Place the free end of the waste line in a waste container below the level of the workbench on which the PDA-100 is installed.

IMPORTANT The drain tube must remain below the drain port. If the drain tube is above the drain port, the drip tray may overflow inside the detector.

#### **B.3.7** Analog Output Connection

Connect a recorder, an integrator, or an A/D data acquisition device to the **ANALOG OUT** connector on the PDA-100 rear panel (see Figure B-3). The four PDA-100 analog outputs provide a 1 volt output maximum.

The outputs are controlled by absorbance wavelengths 1 through 4 in Chromeleon. The software controls the wavelength, sample bandwidth, reference wavelength, full-scale absorbance output, and offset within the 0 to 1 volt range.

#### B.3.8 Relay/TTL Connections



Connection of relay loads and their power sources to the TTL outputs will damage the TTL output stage. If the relay load can source more than 200 mA at 5V or higher, it may damage the Data Processing board.



SHOCK AND FIRE HAZARD—Inputs and outputs are not rated for connection to an AC power main. DO NOT CONNECT TO 100, 115, 220, 230, OR 240 VAC POWER SOURCES; FIRE OR ELECTROCUTION MAY RESULT.



DANGER D'ÉLECTROCUTION ET D'INCENDIE—Le module d'alimentation électrique fonctionne aux potentiels du secteur. Faites effectuer toutes les réparations par un personnel qualifié.



STROMSCHLAG UND BRANDGEFAHR—Das Modul zur Stromversorgung wird mit Netzspannung betrieben. Der Service darf nur von qualifiziertem Personal durchgeführt werden.

The PDA-100 Ship Kit includes a 12-pin connector for Relay and TTL connections (see Figure B-5). To attach individual wires and twisted pairs to the connector, strip the end of the wire(s), insert into the connector, and tighten the locking screw(s) with a screwdriver.



Figure B-5. Relay and TTL Connector

The table below describes the pin assignments for the Relay and TTL connectors. Relays 1 and 2 can be configured in Chromeleon to switch any low-voltage control. The switched current must be less than 200 mA and 42 V peak.

Pin	Assignment		Input Pin Function
1	Relay 1 output	Active	
2	Relay 1 output	Ground	
3	Relay 2 output	Active	
4	Relay 2 output	Ground	
5	TTL 1 output	Active	
6	TTL 2 output	Active	
7	TTL 1 input	Active	Autozero
8	TTL 2 input	Active	Reserved
9	TTL 3 input	Active	UV lamp on/off
10	TTL 4 input	Active	Visible lamp on/off
11	TTL input or output	Ground 1	
12	TTL input or output	Ground 2	

# **C** • Reordering Information

Part Number	Item	Quantity	
939016T	Deuterium lamp	1	
056123T	Tungsten lamp	1	
954763	Fuse, 4.0 amps (fast-blow IEC 127)	1	
923686	12-pin relay/TTL connector	1	
043598	Twisted wire assembly	2 m (8 ft)	
960777	USB cable, 1.8 m (6 ft)	1	
	PDA-100 with PEEK Flow Cell		
056346	PEEK flow cell (with heat exchangers)	1	
060037	PEEK flow cell outlet tubing assembly	1	
043276	Ferrule	1	
043275	Nut	1	
060498	Flow cell windows replacement kit	1	
PDA-100 with Stainless Steel Flow Cell			
056126	Stainless steel flow cell (with heat exchangers)	1	
060038	Stainless steel flow cell outlet tubing assembly	1	
055902	Reducing union fitting	1	
010262	Ferrule	1	
010261	Nut	1	
060498	Flow cell windows replacement kit	1	

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