



# PDA-100 PHOTODIODE ARRAY DETECTOR OPERATOR'S MANUAL

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## 1.1 Product Overview

The PDA-100 Photodiode Array Detector is an optical detector capable of measuring the absorbance spectrum from 190 nm to 800 nm. A deuterium lamp optimizes the UV range (190 nm to 380 nm) and a tungsten lamp optimizes the visible range (380 nm 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 runs that do not require spectral data and it conserves disk space.

Key PDA-100 features include:

- Deuterium and tungsten lamps ensure low baseline noise, high signal intensity, and applications flexibility in the wavelength range from 190 nm to 800 nm.
- A 1024-element photodiode array optimizes spectral resolution.
- A built-in holmium oxide filter verifies wavelength calibration accuracy.
- 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 host computer running PeakNet<sup>®</sup> 6 or Chromeleon<sup>®</sup> software (Release 6.1 or later). The detector communicates with the host computer via the Dionex DX-LAN<sup>™</sup> interface.

To take full advantage of the capabilities of the PDA-100, order the 3D option for the software. The 3D option is required in order to perform the following:

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

## **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), whereas 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 run. 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 run, 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 run.



- **Determination of the correct wavelengths in one run**

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 and 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**

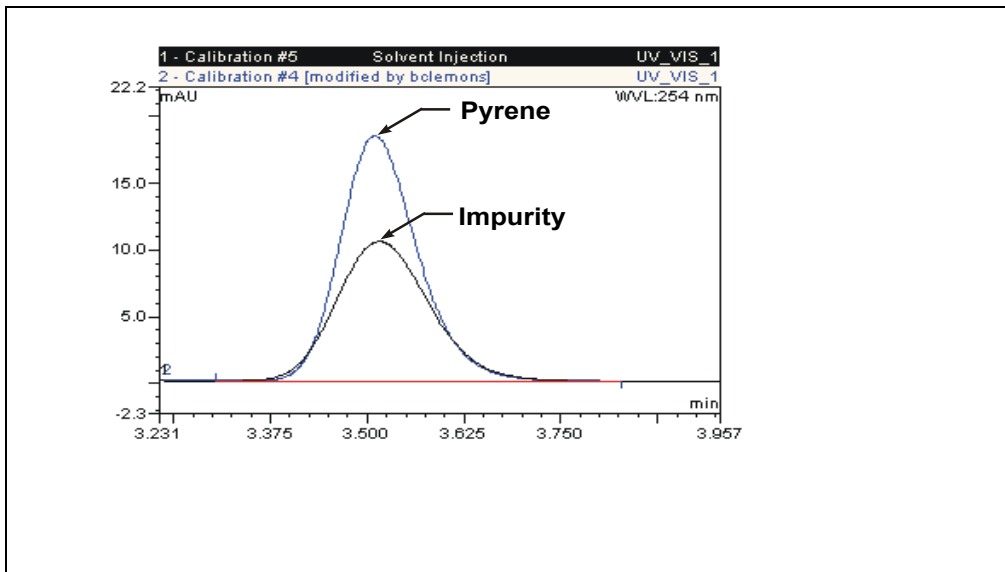
It is difficult to determine component purity from a chromatogram. However, a PDA detector can analyze peak purity by comparing spectra within a peak. A pure peak has matching spectra throughout the peak (at all wavelengths).

- **Positive peak identification**

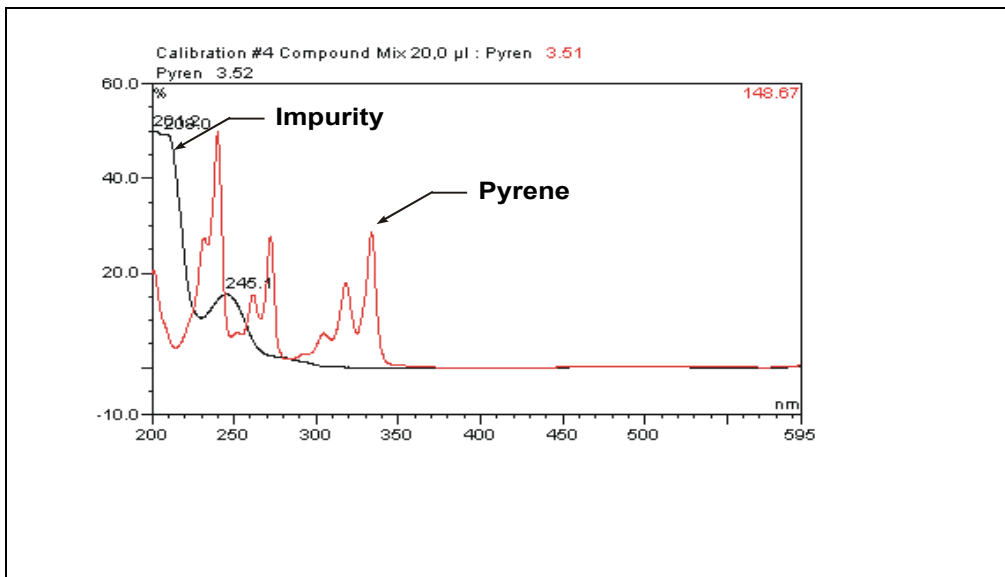
In liquid chromatography, peak identification is usually based on relative retention times. When a PDA detector is used, spectra are automatically collected as each peak elutes. The PDA software compares the spectra with those stored in a library to determine the best fit matches; this method increases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

For example, Figure 1-1 shows an overlay of pyrene and an impurity (obtained from two consecutive chromatographic runs). Based on 2D data, or if the run had been performed with a conventional variable wavelength detector, these peaks would have been misidentified as the same component.

A PDA-100, under the control of Dionex software with the 3D option, correctly identified and differentiated the components. The spectra of pyrene and the impurity (see Figure 1-2) 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 About This Manual

- Chapter 1 Introduction** Provides a brief overview of the PDA-100 and the theory of photodiode array detection. Summarizes the organization of the user's manual. Explains the meaning of safety messages and icons in the manual and the safety labels on the detector.
- Chapter 2 Description** Describes the PDA-100 front panel LED indicators, inside front panel, optical system, and rear panel connections.
- Chapter 3 Operation and Maintenance** Presents an overview of PDA-100 operation, as well as guidelines for optimizing detector performance. Lists routine preventive maintenance procedures.
- Chapter 4 Troubleshooting** Lists problems that may occur, along with step-by-step instructions for resolving them.
- Chapter 5 Service** Contains step-by-step instructions for routine service and parts replacement procedures.
- Appendix A Specifications** Lists the PDA-100 specifications and installation site requirements.
- Appendix B Installation** Describes how to install the PDA-100, including the DX-LAN connections to the host computer running Dionex chromatography software.
- Appendix C Reordering Information** Lists spare parts for the detector.

### 1.3.1 Safety Messages and Notes

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.



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 the user to an unexpected result of an action, suggest how to optimize instrument performance, etc.

## **1.4 Safety 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 61010-1:1993 (safety), CAN/CSA-C22.2 No. 1010.1-92 (safety), UL 3101-1/10.93 (safety), EN 50082-1:1992 (susceptibility), and EN 55011:1991 (emissions).

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

### 2.1 Front Panel

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

The **Power** switch provides on/off control of the detector power. LED indicators identify the status of four functions: the detector power, the deuterium and tungsten lamps, and the run. A solid lighted LED indicates that the corresponding function is in progress. A flashing LED indicates that an error occurred; for an explanation, check the Audit Trail in PeakNet 6 or Chromeleon.

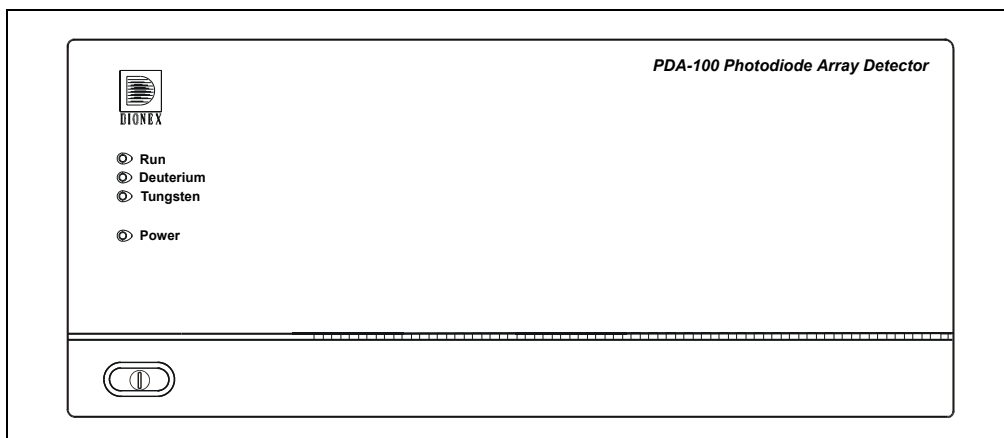


Figure 2-1. PDA-100 Front Panel

LED	LED Status	Function
<b>Run</b>	Lighted	Data is being acquired by the host computer.
	Flashing	An alarm was activated; for example, the leak sensor in the drip tray may have been triggered.
	Off	Data is not being acquired by the host computer.
<b>Deuterium</b>	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 4.5).
	Off	The deuterium lamp is off.

## **PDA-100 Photodiode Array Detector**

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<b>LED</b>	<b>LED Status</b>	<b>Function</b>
<b>Tungsten</b>	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 Section 4.5).
	Off	The tungsten lamp is off.
<b>Power</b>	Lighted	The detector power is on. Press the <b>Power</b> switch to turn the detector on and off.
	Flashing	The detector failed one of the electrical checks or optical checks performed during the power-up sequence (see Section 4.15.1).
	Off	The detector power is off. Press the <b>Power</b> switch to turn the detector on and off.



## 2.2 Inside Front Panel

**NOTE** For an illustration of the inside front panel with the flow cell cover and lamp cover removed, see Figure 5-1.

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

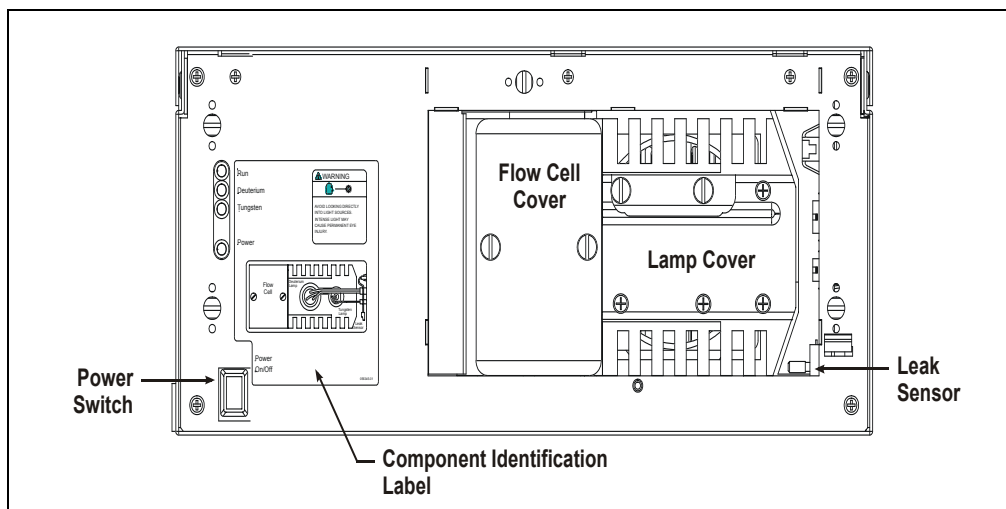


Figure 2-2. PDA-100 Inside Front Panel

- 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.

If the leak sensor is activated, it triggers an alarm in the detector and the front panel **Run** LED begins flashing (see Figure 2-1). In addition, the leak is reported to the host computer and PeakNet 6 or Chromeleon displays an error message.

## 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 separated into its component wavelengths and diffracted to the photodiode array. Each diode measures a narrow portion of the spectrum.

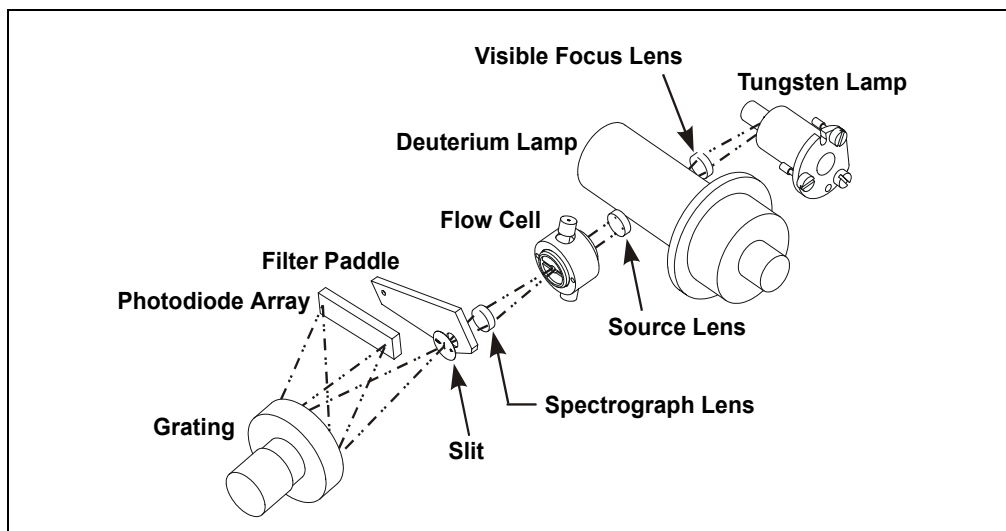


Figure 2-3. PDA-100 Optical System

Optical System Component	Function
Tungsten Lamp	The light source for visible and near-infrared wavelengths (380 nm 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 nm to 380 nm); also used to check the wavelength calibration.
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: <ul style="list-style-type: none"><li>• <i>Open</i> = Light passes, unobstructed, along the light path when the holmium oxide filter is moved out of the light path.</li><li>• <i>Blocked</i> = No light passes along the light path; used to measure the dark signal without turning off the lamps.</li><li>• <i>Holmium</i> = Places the holmium oxide filter in the light path; used to verify wavelength accuracy.</li></ul>
Slit	The width of the slit (equivalent to 1 nm) optimizes the optical resolution.
Grating	Diffraction 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 range of results.

## 2.3.1 Flow Cell

The PDA-100 flow cell assembly (see Figure 2-4) 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.

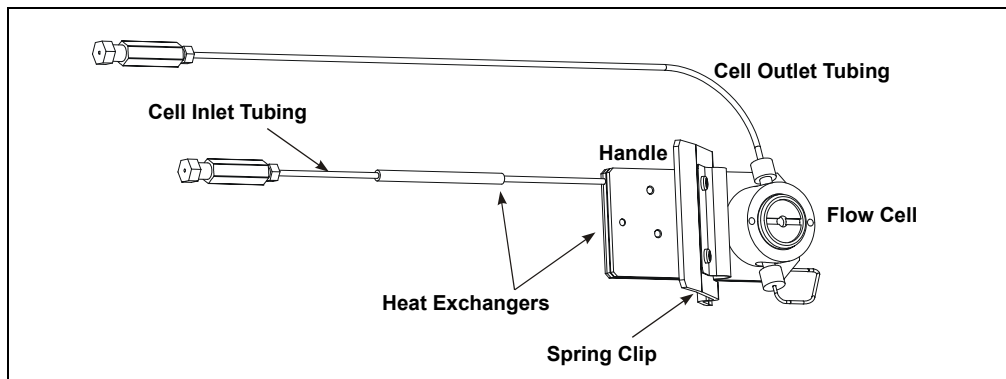


Figure 2-4. Flow Cell Assembly

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

**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 salts; the salts can cause corrosion, contamination, and metal leaching.

## 2.4 Electronics



Do not attempt to access the PDA-100 electronics. The components on the cards 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. The power supply is rated at 130 W. 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

The lamp power supply controls the deuterium and tungsten lamps; it also 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 Card

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 card. Preamp output is directed to the Data Processing card.

### **Data Processing Card**

The PDA-100 Moduleware resides on the Data Processing card. Four BNC connectors on the card provide the analog (recorder) outputs. Select the output scale, polarity, baseline offset, and filter rise time from PeakNet 6 or Chromeleon.

In addition, the Data Processing card provides the digital signal processing and conversion to log ratios when absorbance is measured.

## 2.5 Rear Panel

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

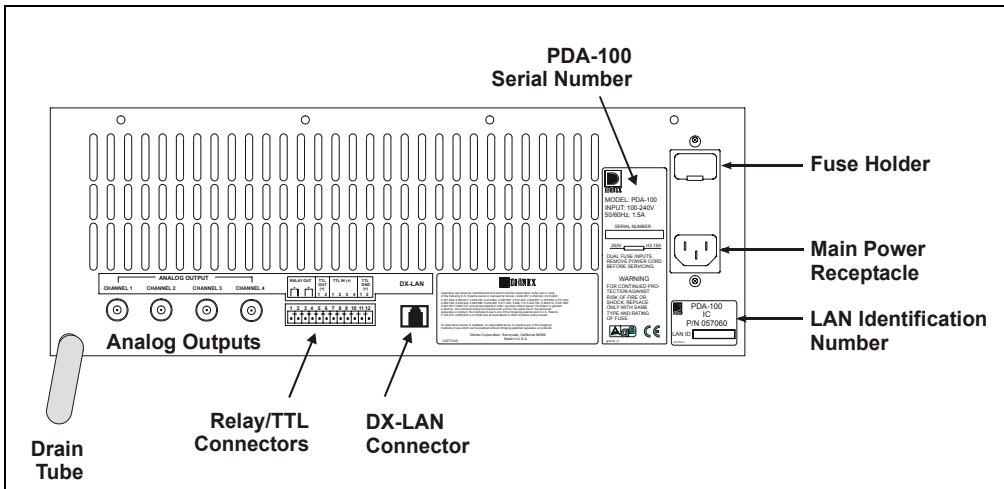


Figure 2-5. PDA-100 Rear Panel

### Power Entry

The power entry is socketed for a modular power cord (IEC 320 C13). The PDA-100 operates from input voltages over a range from 85 Vac to 264 Vac, 47 Hz to 63 Hz power and requires a grounded, single-phase power source. The input power is 162 W, maximum and the output power is 130 W, maximum. 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.





AVERTISSEMENT

**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.



MISE EN GARDE

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.



WARNUNG

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



VORSICHT

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 PDA-100 uses two 4.0 amp fast-blow fuses (IEC 127 type 1, P/N 954763). See Section 5.4 for fuse replacement instructions.



WARNING

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



AVERTISSEMENT

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



WARNUNG

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 (selected from the software) includes 0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, and 3.0 AU full-scale. For details about programming the analog outputs, see Section 3.3.2.

The analog outputs continue to output data even after the host computer is turned off or is disconnected from the PDA-100. When the detector power is turned on again, the analog output settings are either the factory defaults (see Section 3.5) or the settings selected the last time the detector was controlled by the host 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 Section 3.3.2) 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, refer to Section B.3.5.

### **DX-LAN Connector**

The 10BASE-T DX-LAN (RJ-45) connector on the rear panel allows communication between the PDA-100 and the host computer running PeakNet 6 or Chromeleon software. The PDA-100 is usually connected directly to the computer, using a 10BASE-T DX-LAN cable (P/N 960281). For installation instructions, refer to Section B.3.6.

### **Waste Line**

Waste exits the detector via the drain tube connected to 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 Section B.3.3.

#### **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.**

## 3 • Operation and Maintenance

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### 3.1 Operating Guidelines

**NOTE** The PDA-100 Photodiode Array Detector is designed for use with IC (ion chromatography), BioLC, and HPLC (high-performance liquid chromatography) systems and should not be used for any other purpose. If there is a question regarding appropriate usage, 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.

#### 3.1.1 Mobile Phases

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

**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 deionized water for 5 minutes at 1.0 mL/min immediately after the analysis.**

- Prepare all mobile phases with spectroscopy-grade solvents, reagent-grade chemicals, and ASTM Type I (or better) filtered, 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 deionized water immediately after the analysis; do not let buffers remain in the cell for extended periods.

### **3.1.2 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 extremes of pH or with high salt concentration. 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 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 suppressor (if installed).
2. Connect the line exiting the injection valve directly to the flow cell.
3. Flush the system with deionized 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 deionized 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 deionized 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.**

### 3.2 Getting Started

Before beginning operation of the PDA-100, check the following:

- Press the **Power** switch on the front panel to turn on the detector (see Figure 2-1).
- Make sure the **Power** LED is lighted, but is not flashing. If the **Power** LED is flashing, the detector failed one of the electrical checks or optical checks performed during the power-up sequence.
- Turn on the appropriate lamp(s) in PeakNet 6 or Chromeleon, if necessary.

The first time the detector power is turned on, both lamps are on (the factory default settings). To change the default settings, press F8 to display the Commands dialog box, select **UV** (to display UV-related commands), and then select **UVLampPowerOnSetting** and/or **VisLampPowerOnSetting**.

The first time the deuterium lamp is turned on, the detector checks the wavelength-to-pixel assignment; if a problem is detected, the **Power** LED will flash. If this occurs, flush the flow cell with deionized water or methanol and then turn off the detector power briefly. Turn on the power again and verify that the **Power** LED is lighted, but is not flashing.

**NOTE** If solvent remains in the flow cell for an extended period, it may form a bubble or decrease the intensity of the light passing through the cell. This will cause the detector to fail the wavelength calibration check (performed the first time the deuterium lamp is turned on after power-up) and/or the autozero.

- Make sure the **Deuterium** and/or **Tungsten** LEDs on the detector front panel are lighted (see Figure 2-1).

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.

## 3.3 Routine Operation

During routine operation, the PDA-100 is controlled from a host computer running either PeakNet 6 or Chromeleon software. The following sections describe the two modes of software control: panel control (in which commands are executed directly from a control panel) and automated control (control from a PGM file).

**NOTE** The 3D software option is required in order to perform the following: 3D data presentation and spectral analysis, spectral library search for positive peak identification, and peak purity analysis with selectable criteria.

### 3.3.1 Panel Control

In this mode, the user selects PDA-100 operating parameters and commands from a control panel in the software. Commands are executed as soon as they are entered and 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 PeakNet 6 or Chromeleon online Help or user's manual.

#### Summary of control panel operation

1. Start PeakNet 6 or Chromeleon.
2. Open the Browser.
3. Select one of the PDA-100 default control panels:
  - DIONEX\_PUMP\_PDA\_AUTOSAMPLER.PAN, the 2D control panel, monitors chromatograms of up to five signals (see Figure 3-1).
  - DIONEX\_PUMP\_PDA3D\_AUTOSAMPLER.PAN, the 3D control panel, monitors chromatograms, contour plots, and spectra on-line (see Figure 3-2). The 3D control panel is available only when PeakNet 6 or Chromeleon includes the 3D option.

## PDA-100 Photodiode Array Detector

4. Connect the control panel to the timebase.
5. Select the operating parameters.
6. Turn on the pump. Set the flow rate and mobile phase composition.
7. Click the **Acquisition On/Off** toolbar button to begin manual data acquisition.
8. Click the **Acquisition On/Off** toolbar button to stop manual data acquisition after the desired run time.
9. Data collected during the run may be saved to a sequence. To retrieve the data, select the assigned sample name within the sequence.

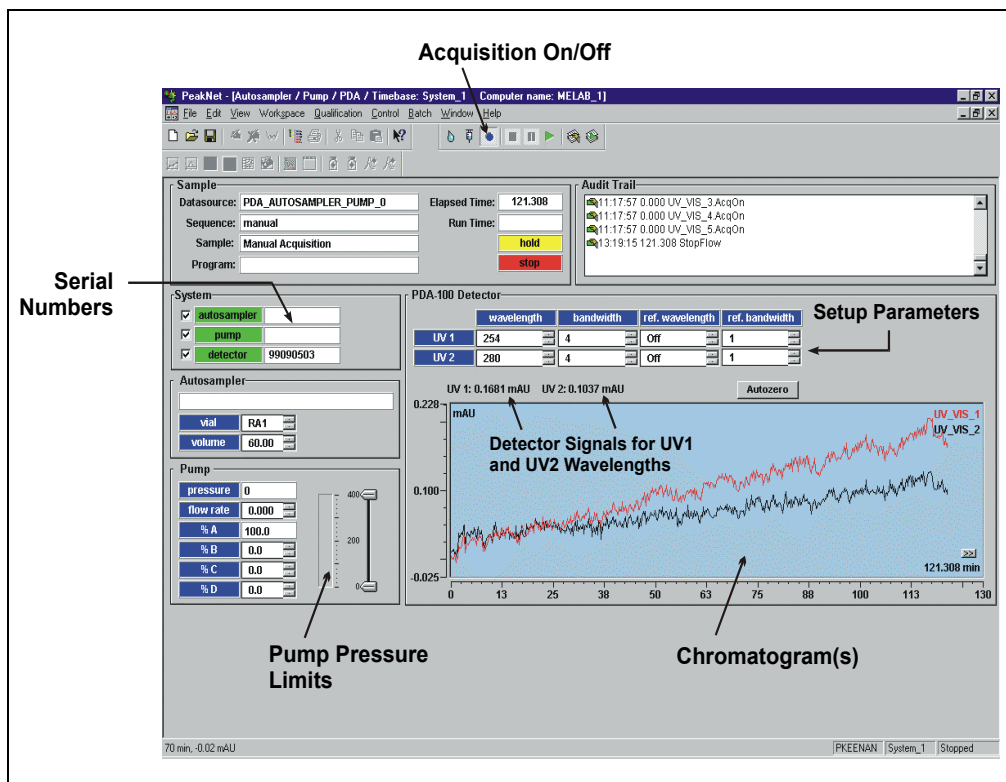


Figure 3-1. PDA-100 2D Control Panel Example



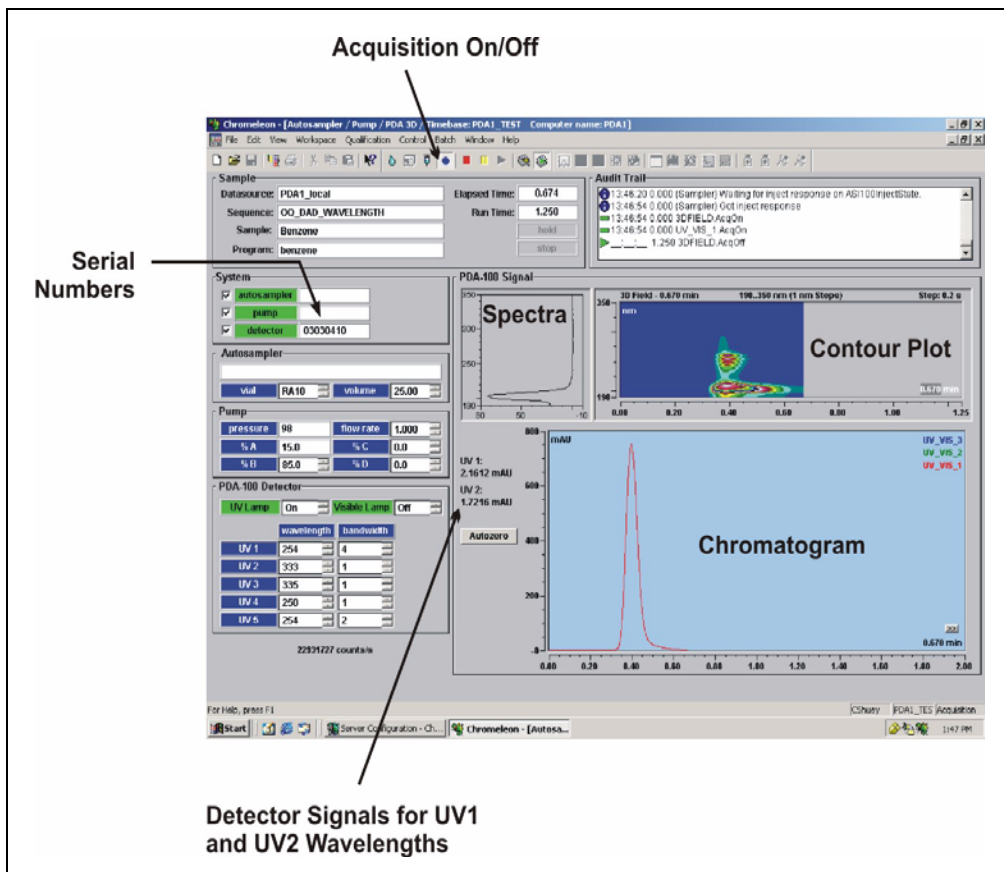


Figure 3-2. PDA-100 3D Control Panel Example

## PDA-100 Photodiode Array Detector

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PeakNet 6 and Chromeleon include several detector commands. These commands are selected from the default control panel or the Commands dialog box (selected from the **Control** menu or by pressing F8).

The table below lists several of the most frequently used detector commands. For more information, refer to the online Help.

PDA-100 Command	Function
Connect	Indicates whether the PDA-100 is connected to the server and the selected timebase.
UV Lamp On/Off	Turns the deuterium lamp on and off.
Vis Lamp On/Off	Turns the tungsten lamp on and off.
UVLampPowerOnSetting	Selects the power-up setting for the deuterium lamp.
VisLampPowerOnSetting	Selects the power-up setting for the tungsten lamp.
Autozero	Adjusts the output to zero.
Recorder_Range	Sets the full-scale recorder output range.
Offset_Level	Sets the offset applied to the recorder output.
Reference_Mode	Reports the reference mode.
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.
RefBandwidth	Sets the bandwidth of the reference wavelength, if one is selected. If RefWavelength is <b>Off</b> , the RefBandwidth setting has no effect.
Wavelength	Sets the sample wavelength (up to five sample wavelengths can be programmed).
Bandwidth	Sets the bandwidth of the sample wavelength.
BunchWidth	Bunching (or averaging) the signals of adjacent wavelengths reduces the size of the 3D data stored.
NegativeAbsorptionLevel	Adjusts the negative absorbance; this permits peaks to be seen even when the eluent decreases absorbance.
WavelengthCal	Runs the wavelength calibration procedure.

### 3.3.2 Programmed Control

A PGM file is a list of time-precise control commands created by the user for automated operation of the PDA-100. The user can create PGM files 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 PeakNet 6 or Chromeleon online Help or user's manual.

#### Summary of programmed control

1. Start PeakNet 6 or Chromeleon.
2. Create a new PGM file (see the following section) or modify an existing program.
3. Create a new sequence or modify an existing sequence.
4. Select one of the PDA-100 default control panels:
  - DIONEX\_PUMP\_PDA\_AUTOSAMPLER.PAN, the 2D control panel, monitors chromatograms of up to five signals (see Figure 3-1).
  - DIONEX\_PUMP\_PDA3D\_AUTOSAMPLER.PAN, the 3D control panel, monitors chromatograms, contour plots, and spectra on-line (see Figure 3-2). The 3D control panel is available only when PeakNet 6 or Chromeleon includes the 3D software option.

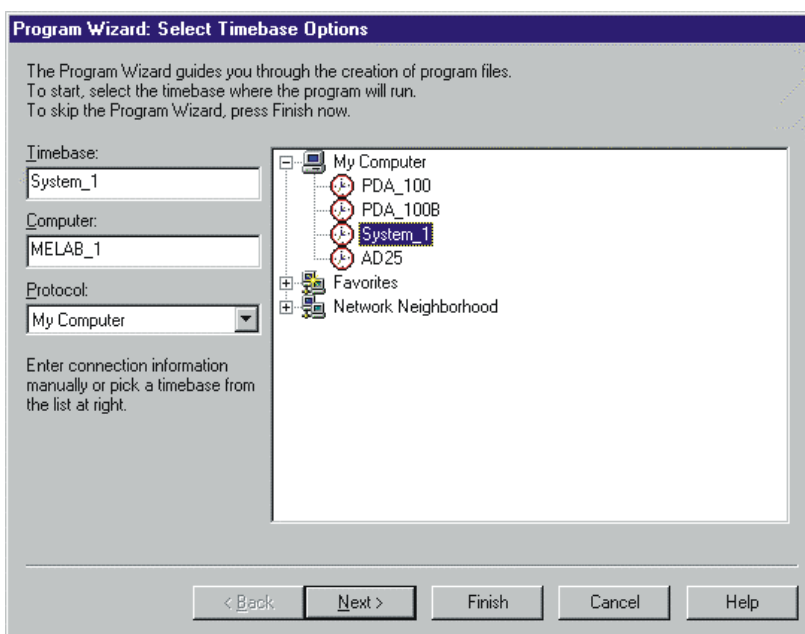
### Creating a New PGM File

1. PeakNet 6 and Chromeleon contain a Program Wizard to facilitate the creation of new PGM files. To activate the Wizard, go to the **File** menu and select the **New** command.

A dialog box appears.

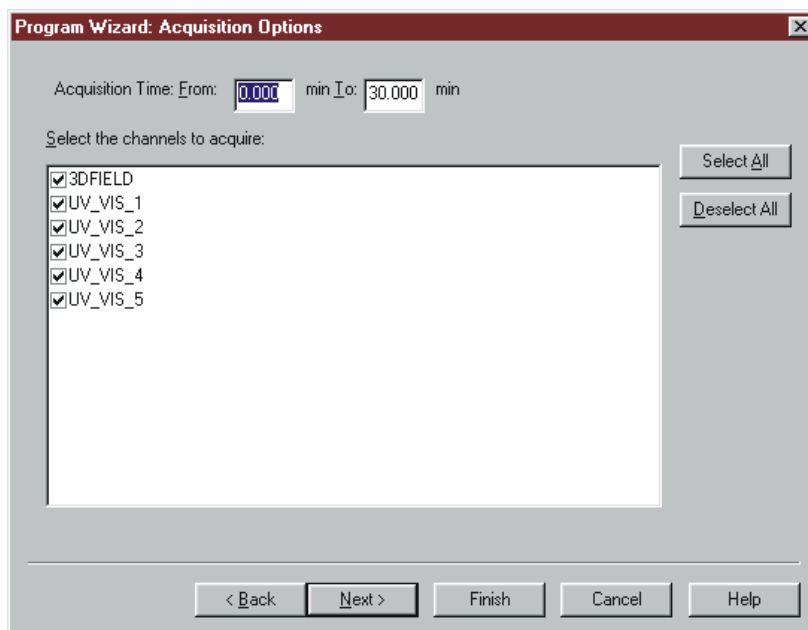
2. Select **Program File** and click **OK**.

The initial Program Wizard screen is displayed.



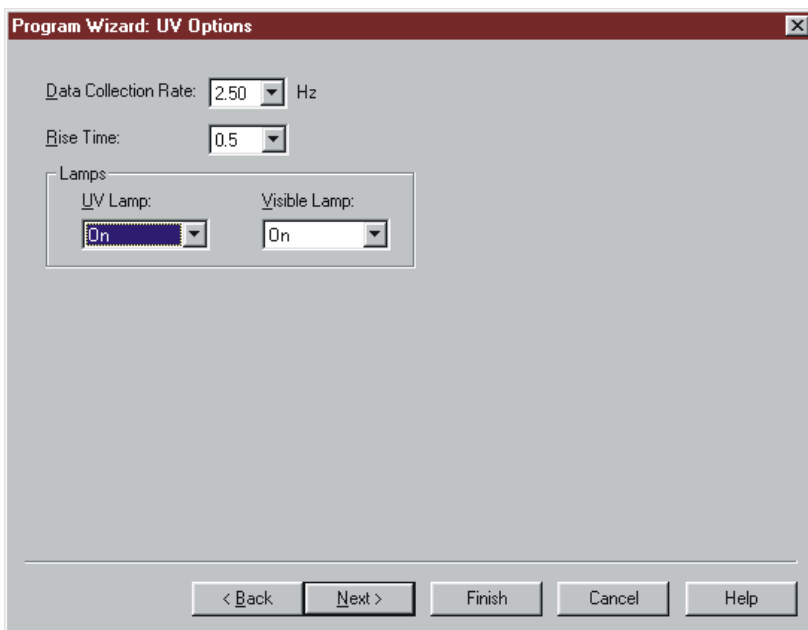
3. Select the **Timebase** where the program will run.
4. Click the **Next** button to continue.

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



5. Select the data **Acquisition Time**.
6. Check (or clear) the **3DFIELD** check box to indicate 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.

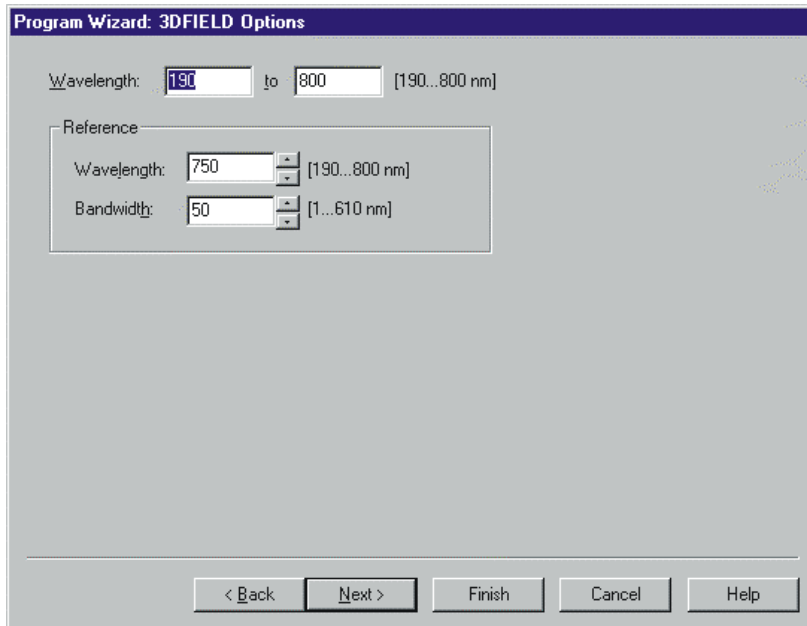


9. Select the **Data Collection Rate** and the **Rise Time**.

**NOTE** The PDA-100 performance can be optimized by careful selection of the data collection rate, rise time, and other operating parameters. For details, refer to Section 3.4.

10. Select the **Lamp** status.
11. Click **Next**.

The following screen is displayed.



12. Select the sample **Wavelength**, **Reference Wavelength**, and **Reference Bandwidth**.

**NOTE** The Reference Wavelength plus one-half the Reference Bandwidth cannot be outside the 3D Wavelength range. If the values are outside this range, the Reference Wavelength setting will default to the longest wavelength possible given the bandwidth. 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.

13. Click **Next**.

The following screen is displayed.

Channel	Wavelength	Bandwidth	RefWavelength	RefBandwidth	Recorder_Range
UV_VIS_1	210	10	Off	50	1.0000
UV_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	

14. 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 and the sample wavelength and sample bandwidth are used as reference parameters.

15. After selecting all PGM file parameters, click **Finish**.



## 3.4 Optimizing Detector Performance

The performance of the PDA-100 can be optimized by careful selection of the operating parameters. Section 3.4 defines several operating parameters, describes how they interact with each other, offers guidelines for selecting them, and includes example chromatograms.

The following table summarizes the topics discussed in this section.

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
Sample bandwidth	Sensitivity vs. baseline noise
Reference wavelength	Baseline drift
Reference bandwidth	Baseline noise, baseline drift
Step*	Baseline noise, rise time, data collection rate, disk space
Bunch width	Spectral resolution, peak match, disk space
Average*	Rise time, data collection rate
Negative absorbance	Peak detection, baseline noise, linearity

\* Set automatically by PeakNet 6 or 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 salts; this may result in 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 can be set to 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.

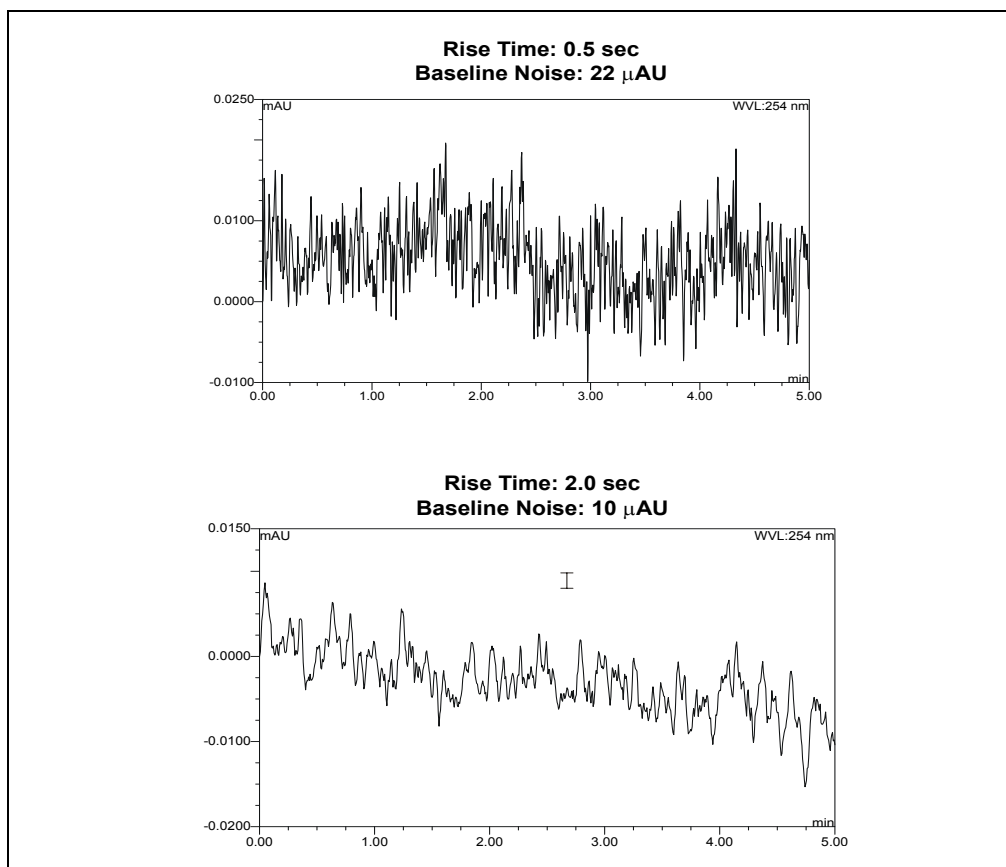


Figure 3-3. Effect of Rise Time on Baseline Noise

### Data Collection Rate

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

The data collection rate can be set to 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. However, 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 nm to 800 nm. The deuterium lamp optimizes the UV range (190 nm to 380 nm). The tungsten lamp optimizes the visible range (380 nm to 800 nm). Set the sample wavelength at the wavelength with the absorbance maxima.

When the analyte wavelength maximas are known, the detector can run five separate wavelengths without being required to collect spectra. Collecting individual wavelengths instead of the spectra offers two advantages: it saves disk space when 3D data is not required and it eliminates the need to perform extractions for runs 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.

### Sample Bandwidth

The sample bandwidth is the wavelength range at which the chromatogram is measured. As the bandwidth increases, baseline noise decreases. Increasing the bandwidth increases the number of wavelength signals overlaid at each point in time. Increasing the bandwidth may also decrease the signal, especially of those peaks with narrow peak maximas.

Select a sample 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.

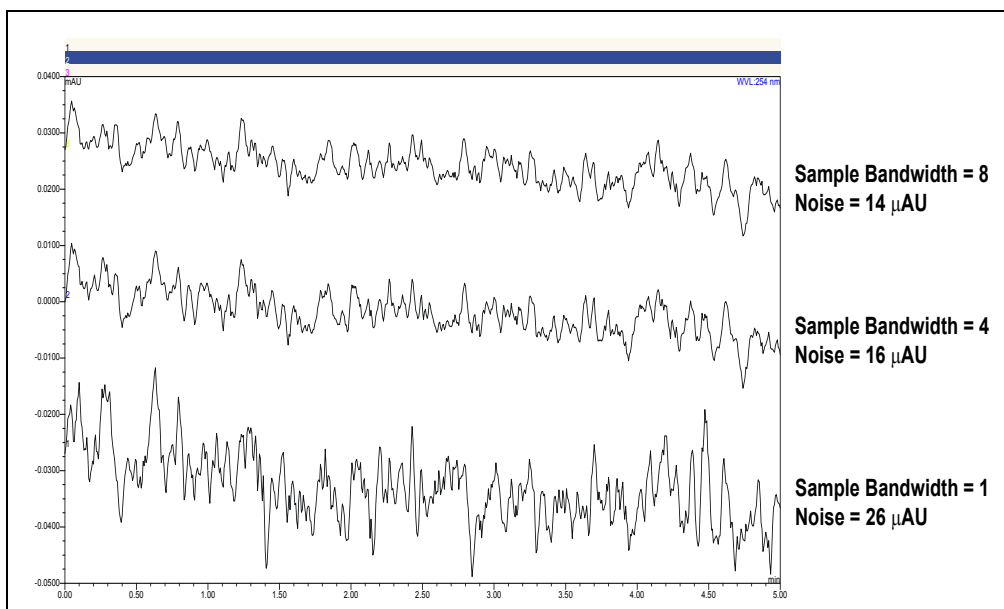
#### **Example Chromatogram: Selecting the Bandwidth**

Figure 3-4 illustrates how selecting a wide bandwidth for both the sample and reference 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 Sample 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” and “off.”

- To select the active reference mode, enter a wavelength within the scanning ability of the PDA-100. The selected wavelength will be used as the reference wavelength. The reference intensity will vary over time, as 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; both wavelengths are within the UV spectrum of the deuterium lamp (190 nm to 380 nm).

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

**NOTE** When active reference mode is selected, be sure to select a reference wavelength and reference bandwidth in a quiet area of the spectrum where little absorption occurs.

- To deselect the active reference mode, enter “Off” as the reference wavelength. The intensity reading at the time of autozero will be used as the reference wavelength.

Operating when the reference wavelength is off ensures the lowest baseline noise when running an isocratic analysis in a laboratory with stable ambient temperature. It also ensures that fluctuations in the reference intensity do not cause additional noise.

**Example Chromatogram: Selecting the Reference Wavelength**

Figure 3-5 shows results obtained from two different channels within the same run. When the reference wavelength is turned off, baseline drift is -120  $\mu$ AU/hour; when a reference wavelength (active mode) is selected, baseline drift is reduced to -40  $\mu$ AU/hour.

The operating conditions are:

Sample wavelength = 520 nm

Sample bandwidth = 10 nm

Reference bandwidth = 50 nm (active reference mode only)

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

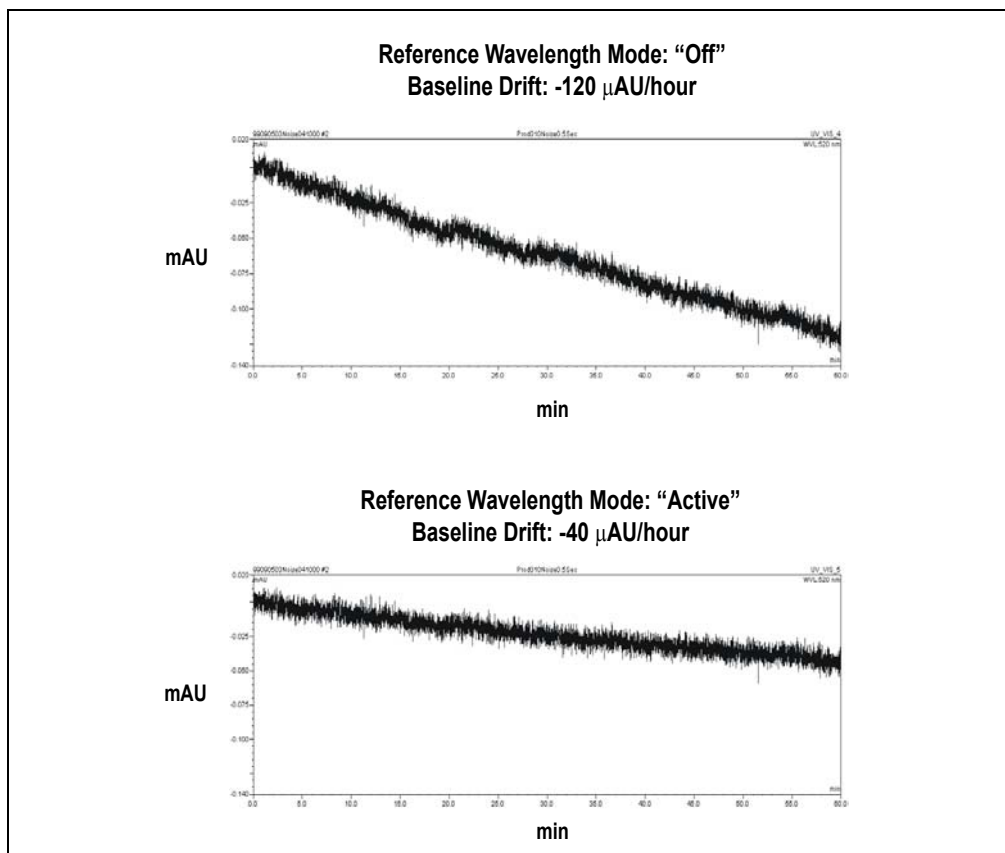


Figure 3-5. Effect of Reference Wavelength on Baseline Drift

### **Reference Bandwidth**

The reference bandwidth, which is analogous to the conventional bandwidth of a channel, serves to average several diode signals of the reference wavelength. A reference bandwidth is required only when a reference wavelength (active mode) is selected. When the reference wavelength is off, the reference bandwidth is the same as the sample bandwidth.

Select a reference bandwidth in an area of the spectrum where the sample does not absorb. The reference bandwidth should be 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

Sample bandwidth = 5 nm

Reference wavelength = 325 nm

Reference bandwidth = 51 nm

These settings calculate the absorbance by the following equation:

$$\text{AU} = \log \frac{\text{avg} [I_K(300 \text{ to } 350)]}{\text{avg} [I_K(210 \text{ to } 274)]} - \text{offset (at autozero)}$$

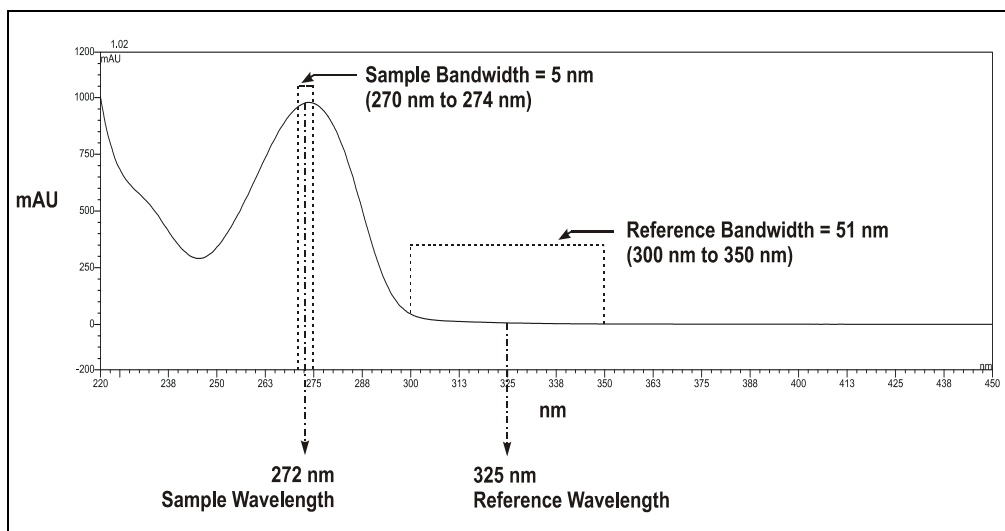


Figure 3-6. Caffeine Spectrum

### 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 the reciprocal. For example, a bunch width of 5 nm requires one-fifth the amount of storage as a bunch width of 1 nm. This parameter reduces the spectral resolution, so do not select a bunch width when the spectrum of the compound has a fine structure.

### Step

**IMPORTANT** The appropriate PDA-100 step setting is automatically selected by the software.

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.

The user does not need to select a step setting; PeakNet 6 or Chromeleon automatically selects the step value that is the inverse of the data collection rate. For example, if you set the data collection rate to 5.0 Hz, the software sets the step to 0.2 second.

The following table indicates the automatically selected step setting 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**

Selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems. Guidelines for overriding the automatic selected step setting are provided for reference by advanced users only.

In almost all cases, the step setting selected by PeakNet 6 or Chromeleon is preferred. However, it is possible to override this setting by manually changing the step in the PGM file or the control panel.

For example, it may be desirable 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 “Step”). 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 “Average”), 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 “Step”).

When average is On, PeakNet 6 or Chromeleon averages the data points between step intervals, reports that value, and noise is reduced.

**NOTE** The default setting for average (On) is recommended for most applications.

When average is Off, PeakNet 6 or 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 will be 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

**NOTE** The `NegativeAbsorptionLevel` command is available only if you are running PeakNet 6.40 SP3 (or later) or Chromeleon 6.50 SP1 (or later) *and* PDA-100 Moduleware version 2.0 (or later).

Negative absorbance is the result of decreased absorption of the eluent after autozero (which usually occurs at the beginning of a run). Depending on certain buffer conditions, this shift can result in flat baselines or truncated peaks; if this occurs, increase the `NegativeAbsorptionLevel` setting to shift the detection level. This is especially helpful when running a gradient application in which the absorbance decreases more than several hundred mAU.

For example, in Figure 3-7, the `NegativeAbsorptionLevel` setting is -100 initially and the baseline is flat when the gradient is  $\leq 50\%$ . The baseline is not flat when the gradient is  $>50\%$  and the `NegativeAbsorptionLevel` is set to -600.

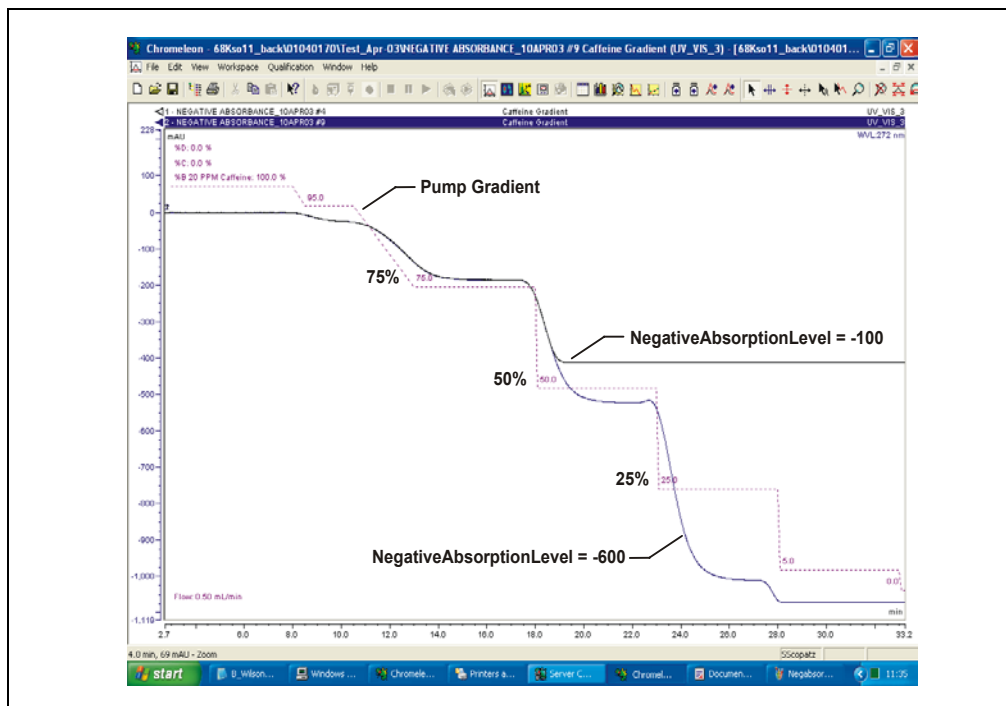


Figure 3-7. Gradient Baseline with `NegativeAbsorptionLevel` Settings of -100 and -600

In PeakNet 6 or Chromeleon, press F8 to display the Commands dialog box. Select **UV**, select the **NegativeAbsorptionLevel** command, and select a higher setting. You may continue increasing the setting if the range is required. **Baseline noise may increase as the NegativeAbsorptionLevel setting increases.**

At the default setting, the detector will become saturated when the light transmission is 20% above the autozero level. The extent of negative absorbance is wavelength-dependent, as indicated in the table below (these values are guidelines only).

After changing the negative absorbance setting, run autozero and then check the intensity of the deuterium lamp. Press F8 to display the Commands dialog box, select **UV**, and select **LampIntensity**. The reading should be above 10 million counts; if it is not, select the **UV\_Calibration Intensity** command to calibrate the lamp. After calibration, 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).

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	-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.

### 3.5 Stand-Alone Analog Operation

This section describes limited, stand-alone detector operation when the PDA-100 is not interfaced to a host computer running either PeakNet 6 or Chromeleon software. In stand-alone operation, TTL inputs can be used to trigger autozero or turn the deuterium and tungsten lamps on and off. All other detector parameters remain at their factory default settings and cannot be changed.

#### Factory Defaults

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

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

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

These factory default settings are also in effect:

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

#### Stand-Alone Operation

The table below lists the TTL inputs and the PDA-100 parameters they control during stand-alone operation.

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

## 3.6 Routine Maintenance

The following maintenance procedures may be performed by the user. Other maintenance procedures must be performed by trained Dionex Service personnel.

- Before shutting down the detector for more than 24 hours, flush the system with deionized 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 in the bottom of the optical bench; verify that the tubing is unclogged and remains routed below the leak tray.
- Periodically check the flow cell tubing connections for leaks or restrictions. Replace tubing and fittings as necessary.
- Periodically check all tubing and fittings, and replace as necessary (see Section 5.1). 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. However, if the backpressure remains high, remove the fittings and tubing from part of the flow path, section by section; start at the waste line and work back “upstream” until the section causing the substantial backpressure is identified. Replace tubing and fittings as necessary.

- Periodically repassivate stainless steel systems. This is especially important when operating with extremes of pH (which shortens the life of the passivation finish) or after a major disassembly. For instructions, see *Passivation of Stainless Steel Systems and Components* (Document No. 031152).

- Replace the deuterium and tungsten lamps when required (see below). The average lamp lifetime is 1000 hours. However, lamps often last longer than this with only minimal deterioration in performance.

Monitor baseline noise. If the noise increases above an acceptable level, especially after a lamp(s) has been in service for more than 1000 hours, replace the lamp. See Section 5.7 or Section 5.8 for replacement instructions for the deuterium or tungsten lamp, respectively.

Approximately every 6 months, check the intensity of the deuterium lamp as follows: Press F8 in PeakNet 6 or Chromeleon to display the Commands dialog box, select **UV** (to display UV-related commands), and select **LampIntensity**. The reading should be above 10 million counts; if it is not, select the **UV\_Calibration Intensity** command to calibrate the lamp. After calibration, 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).

- Approximately every 6 months, run the wavelength calibration and verification procedures for the deuterium and tungsten lamps from the Wellness panel (see Section 4.15.2).



## 4 • Troubleshooting

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This chapter is a guide to troubleshooting routine problems that may occur while operating the PDA-100 Photodiode Array Detector. Turn to the section that best describes the operating problem; there, possible causes of the problem are listed in order of probability, along with the recommended courses of action.

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 Flashing LED

- **Run LED is flashing**

An alarm was activated. Check the software Audit Trail for an error message.

- **Deuterium LED is flashing**

The lamp did not power on properly. Check the software Audit Trail for an 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 Technical Support for assistance.

- **Tungsten LED is flashing**

The lamp did not power on properly. Check the software Audit Trail for an error message. If necessary, replace the lamp (see Section 5.8).

- **Power LED is flashing**

The detector failed one of the checks performed during the power-up sequence (see Section 4.15.1) or the wavelength calibration check that is performed the first time the deuterium lamp is turned on after power-up. Check the software Audit Trail for an error message.

### **4.2 No Detector Response**

- **Detector power not turned on**

Check that the main power switch is turned on.

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

Check the fuses and replace them, if necessary (see Section 5.9).

- **Power-up sequence failed**

If the **Power** LED is flashing, turn off the detector power briefly. Turn on the power again and check that the **Power** LED is lighted, but is not flashing.

- **Lamp not turned on**

If a lamp is turned off, the corresponding front panel LED (**Deuterium** or **Tungsten**) is off, also. Turn on the lamp from the software or the TTL input.

- **Lamp LED flashing**

The lamp did not power on properly. Press F8 in the software to display the Commands dialog box, select **UV** (to display UV-related commands), and check that **UVLampPowerOnSetting** and **VisLampPowerOnSetting** are **Off**.

If the LED continues flashing, replace the lamp(s). See Section 5.7 or Section 5.8 for replacement instructions for the deuterium lamp or tungsten lamp, respectively.

- **PDA-100 not detected on the DX-LAN**

When the host computer is powered up, the software scans the DX-LAN for all installed instruments. If the PDA-100 power is off, the detector cannot be identified. Before turning on the computer, make sure the detector power is already turned on.

- **DX-LAN interface incorrectly installed**

Check the following (for installation instructions, see Section B.3.6):

- A 10BASE-T DX-LAN cable (P/N 960281) must be connected between the **DX-LAN** connector on the PDA-100 rear panel and the Ethernet hub (P/N 056909).

- A 10BASE-T DX-LAN cable (P/N 960281) must be connected between the Ethernet hub (P/N 056909) and the **DX-LAN** connector on the host computer's internal DX-LAN interface card.

**IMPORTANT**

When using 10BASE-T cabling, always install an Ethernet hub. If you simply plug the 10BASE-T cable from the PDA-100 into the connector on the host computer's DX-LAN card, the connection will not work.

- **Detector and/or control panel not connected to timebase**

Add the PDA-100 control panel to a software timebase.

### 4.3 Noisy Baseline

- **Mobile phase or post-column reagent contains light-absorbing impurities**

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

If the noise is synchronized with the pump stroke, the piston seal or check valves may need cleaning. Refer to the pump user's manual for instructions.

- **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 values.

- **Air bubbles in flow cell**

Remove the trapped air (see Section 5.3).

To prevent air from becoming trapped in the cell, 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 Section B.3.2). This increases the backpressure, thereby shrinking bubbles and allowing them to pass more easily through the cell.

- **Contaminants in flow cell**

Clean the cell (see Section 5.2).

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.

- **Deuterium lamp is dimming**

The light output of the deuterium lamp decreases over time. The lower light output may increase noise above an acceptable level, especially after the lamp has been in service for more than 1000 hours. If this occurs, replace the lamp (see Section 5.7).

- **Tungsten lamp needs replacement**

Install a new tungsten lamp (see Section 5.8).

- **Lamp incorrectly installed**

Make sure that both lamps are fully seated in the optical bench. See Section 5.7 or Section 5.8 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 Section 3.4.

- **Bandwidth is too narrow**

Select a wider sample bandwidth and reference bandwidth, if appropriate for the application. For guidelines when selecting the sample and reference bandwidths, see Section 3.4.

- **Flow cell cover, lamp cover, or front panel is missing**

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

Be sure the cell is inserted straight into the optical bench and is fully seated (see Section B.4.2).

- **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 Section 3.4 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**

The filter paddle is tested during the autozero routine. If the software displays an error message that the filter paddle test failed, remove the flow cell, check that at least one lamp is on, and then autozero the detector. If the error message appears again, contact Dionex Technical Support for assistance.

### **4.4 Drifting Baseline**

- **Fluctuations in ambient temperature**

Make sure the PDA-100 installation site temperature remains consistent. Allow at least 6 cm (2.4 in) 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 5-3). Check that solvent flow is directed to the cell inlet and out the cell outlet.

Select a reference wavelength (see Section 3.4).

- **New lamp not burned in**

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

- **Lamp cover, flow cell cover, or front panel is missing**

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

- **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**

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; if the cell continues to leak, replace it. Also, check that the backpressure on the cell does not exceed the specification (see Section A.5).

- **Inappropriate reference mode selected**

Dionex recommends selecting a reference wavelength (active reference mode) for gradient analyses and turning off the reference wavelength for

isocratic analyses in a laboratory with a stable ambient temperature. For more information, see Section 3.4.

- **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 Section 3.4).

Dionex recommends selecting a reference wavelength (active reference mode) for gradient analyses and turning off the reference wavelength for isocratic analyses in a laboratory with a stable ambient temperature. For more information, see Section 3.4.

## 4.5 Lamp Does Not Light or Lamp LED Is Flashing

- **Lamp is old or burned out**

Replace the lamp (see Section 5.7 or Section 5.8 for instructions on replacing the deuterium lamp or tungsten lamp, respectively). The average lamp lifetime is 1000 hours, although lamps often last longer with only minimal deterioration in performance.

## 4.6 Deuterium Lamp Intensity Count Too Low

If the deuterium lamp intensity reading is low (see Section 3.6), follow these steps to resolve the problem.

- **Light signal is not reaching the data processor; reading is  $\leq 1000$  counts**

Make sure that at least one lamp is turned on.

- **Low light throughput; reading is  $< 10,000,000$  counts**

Remove the cell from the optical bench (see Section 5.4). Insert a business card into the cell mounting location and check that bright light is shining onto the card.

If you can see bright light shining onto the business card, remove the card from the optical bench, run the lamp intensity calibration (see Section 3.6), and then autozero the detector. If no error message is displayed, flush the cell with eluent and/or clean the cell as instructed in (see Section 5.2). Reinstall the cell in the optical bench and autozero the

detector again to verify that the problem has been resolved. If the error message appears again, contact Dionex Technical Support for assistance.

If you see a weak light (or no light at all) shining onto the business card, the lamp intensity is too low. Check that the flange of the 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. (For lamp replacement instructions, see Section 5.7 or Section 5.8 for the deuterium lamp or tungsten lamp, respectively.) If the error message appears again, contact Dionex Technical Support for assistance.

- **Blockage in cell**

Flush the cell with eluent.

### **4.7 Wavelength Calibration Fails**

- **Flow cell contains air bubble or old mobile phase**

Flush mobile phase through the cell.

- **Cell not clear or mobile phase has high background absorbance**

Clean the cell as instructed in Section 5.2. If the detector fails the wavelength calibration again, replace the flow cell windows (see Section 5.5).

- **Deuterium lamp may be defective**

If the **Deuterium** LED flashes or fails to light, replace the deuterium lamp (see Section 5.6).

If the **Deuterium** LED is lighted (but is not flashing), run both the wavelength calibration and the wavelength verification from the Wellness panel (see Section 4.15.2). If these fail, remove the flow cell and repeat the wavelength calibration and the wavelength verification.

If the wavelength calibration still fails, contact Dionex Technical Support for assistance.



## 4.8 Wavelength Verification Fails

- **Detector wavelength requires recalibration**

In PeakNet 6 or Chromeleon, select the **UV Calibration** command on the Wellness panel. Or, press F8 to display the Commands dialog box and select **UV** (to display UV-related commands). Select the **UV\_Calibration** command, and then select **WavelengthCal**.

- **No solvent flow through cell**

If the **Power** LED is flashing (see Figure 2-1), flush the cell with deionized water or methanol and then turn off the detector power briefly. Turn on the power again and verify that the **Power** LED is lighted, but is not flashing.

If solvent sits in the cell for an extended period, it decreases the intensity of the light passing through the cell. To ensure reliable data, maintain a constant flow through the cell. Flushing the cell with deionized water or methanol each time the detector is powered up is helpful.

- **Lamp is dimming**

Check the lamps as follows: In PeakNet 6 or Chromeleon, press F8 to display the Commands dialog box and select **UV** (to display UV-related commands). Select **UVLampAge** and then **VisLampAge**. If either lamp has been in operation for more than 1000 hours, replace it. See Section 5.7 or Section 5.8 for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

- **Solvent absorbs at the wavelength reported for verification**

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

### 4.9 Faulty DX-LAN Communication

- **PDA-100 not detected on DX-LAN**

When the host computer is powered up, PeakNet 6 or Chromeleon scans the DX-LAN for all installed instruments. If the PDA-100 power is off, the detector cannot be identified. Before turning on the computer power, make sure the detector power is turned on.

- **DX-LAN interface incorrectly installed**

Check the following (for installation instructions, see Section B.3.6):

- A 10BASE-T DX-LAN cable (P/N 960281) must be connected between the **DX-LAN** connector on the PDA-100 rear panel and the Ethernet hub (P/N 056909).
- A 10BASE-T DX-LAN cable (P/N 960281) must be connected between the Ethernet hub (P/N 056909) and the **DX-LAN** connector on the host computer's internal DX-LAN interface card.

#### **IMPORTANT**

When using 10BASE-T cabling, always install an Ethernet hub. If you simply plug the 10BASE-T cable from the PDA-100 into the connector on the host computer's DX-LAN card, the connection will not work.

### 4.10 No Spectra Collected

- **Spectral range was not programmed**

Select a spectral range in the PGM file or control panel.

- **Detector and PGM file (or control panel) connected to different timebases**

Add the PDA-100 to the same timebase as the PGM file or control panel.

### 4.11 Low Spectral Resolution

- **Bunch width too wide**

Select a bunch width setting of 1 nm and then recheck the resolution.

- **Sample bandwidth too wide**

Select a sample 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 sample 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 and data collection rate are appropriate to capture the narrowest peak. See Section 3.4 for more information.

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, refer to Section 3.4.

- **Incorrect reference wavelength selected**

Select a different reference wavelength. For guidelines to follow when selecting the reference wavelength, refer to Section 3.4.

- **Sample 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 also 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.

## 4.14 Error Message: Not Enough Light to Perform Operation

- **Light signal is not reaching the data processor**

Make sure that at least one lamp is turned on.

- **Low light throughput**

Remove the cell from the optical bench (see Section 5.4). Insert a business card into the cell mounting location and check that bright light is shining onto the card.

If you can see bright light shining onto the business card, remove the card from the optical bench, run the lamp intensity calibration (see Section 3.6), and then autozero the detector. If no error message is displayed, flush the cell with eluent and/or clean the cell as instructed in (see Section 5.2). Reinstall the cell in the optical bench and autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Dionex Technical Support for assistance.

If you see a weak light (or no light at all) shining onto the business card, the lamp intensity is too low. Check that the flange of the new 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. (For lamp replacement instructions, see Section 5.7 or Section 5.8 for the deuterium lamp or tungsten lamp, respectively.) If the error message appears again, contact Dionex Technical Support for assistance.

- **Blockage or low light transmission in cell**

Flush the cell with eluent.

### 4.15 Diagnostics

#### 4.15.1 Moduleware Run-Time Diagnostics

The PDA-100 Moduleware periodically checks the status of certain system parameters. Minor problems that do not justify stopping operation are reported to the host computer and recorded in the Audit Trail. The Audit Trail includes the date, time, and error code 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 and the file for that date.

#### 4.15.2 Software Diagnostics and Calibration

PeakNet 6 and Chromeleon contain comprehensive diagnostics for the PDA-100. This section briefly describes some of these diagnostics; for a full description, refer to the software Help or user's manual.

- **Lamp age**

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

To reset the lamp age after installing a new lamp:

- a. Press F8 to display the Commands dialog box.
- b. Select **UV** (to display UV-related commands).
- c. Select **UVLampAge** or **VisLampAge**.
- d. Reset the number to zero.
- e. Click the **Execute** button.

**NOTE** When the detector power is initially turned on, the lamp age already indicates some elapsed time; this is the time required for factory calibration and test procedures.

- **Wavelength calibration**

Reassigns new wavelengths to each pixel, based on the emission spectrum from the deuterium lamp. Both the initial calibration and previous calibration are stored in the calibration log and can be retrieved at any time.

To calibrate the wavelength:

- a. Press F8 to display the Commands dialog box.
- b. Select **UV** (to display UV-related commands).
- c. Select **UV\_Calibration** and then **WavelengthCal**.
- d. Click the **Execute** button.
- e. When the calibration procedure is complete, select the **WavelengthCalResult** command, or click the **Wavelength** button on the PDA-100 Wellness control panel.





This chapter describes routine service procedures that the user may perform. All procedures not described here must be performed by trained 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** The PDA-100 electronics cannot be serviced by the user; all repairs of these components must be performed by trained Dionex Service personnel.



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

Before replacing any parts, review the troubleshooting information in Chapter 4 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*.

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
Union Fittings	P/N 055903	P/N 055902
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 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 deionized water, acetone, and 3 M HNO<sub>3</sub>.
4. Flush the cell with deionized water for 20 to 30 minutes at 1.0 mL/min.
5. Reconnect the cell liquid lines.

## **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

Remove the flow cell from the optical bench prior to inspecting the cell for leaks or obstructions, replacing the cell windows (see Section 5.5), or installing a new cell (see Section 5.6).

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.
4. Remove the tubing bracket from the lamp cover.
5. Remove the flow cell inlet tubing from the tubing guide in the lamp cover.
6. Squeeze the handle of the flow cell and pull it out of the optical bench.

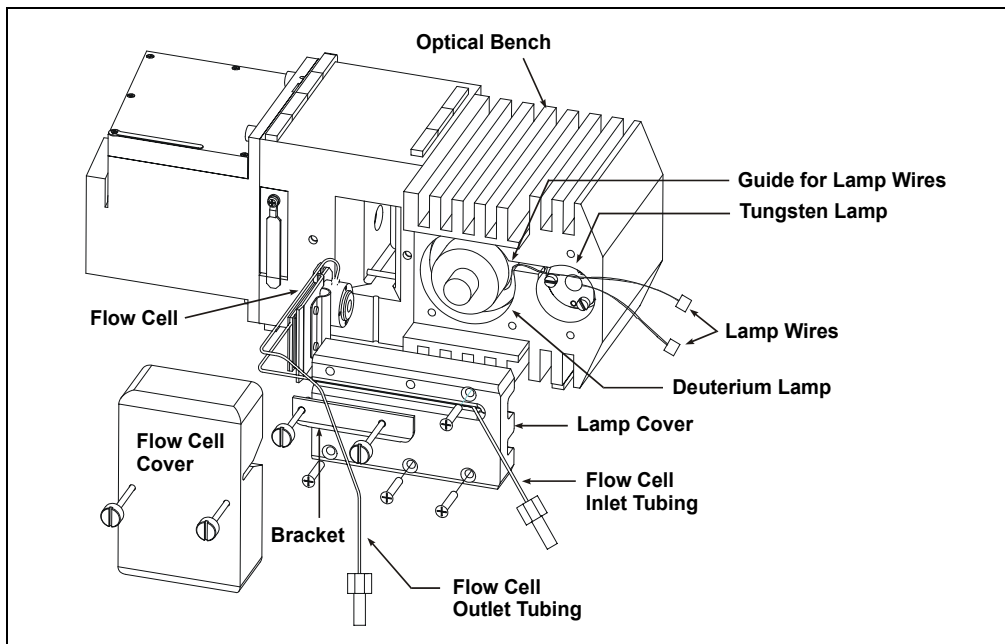


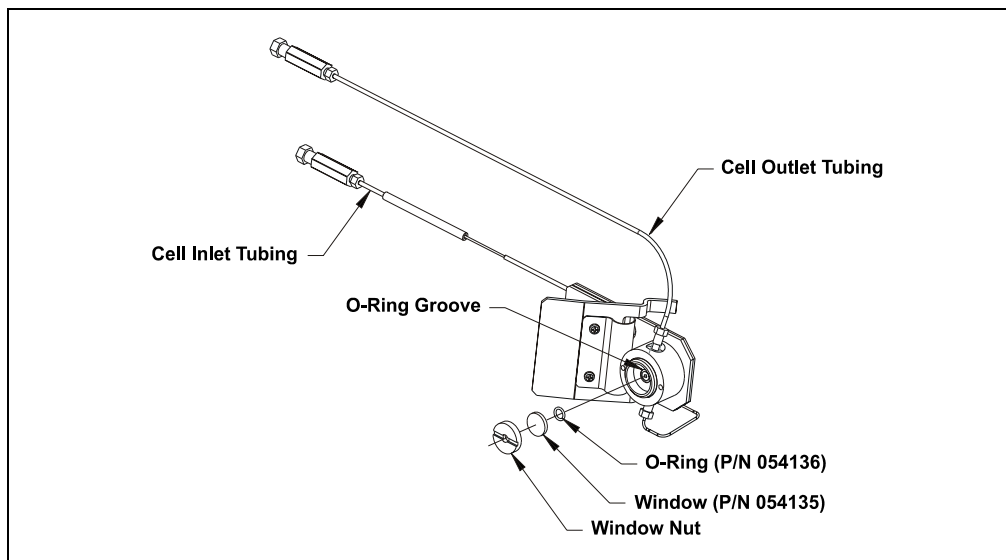
Figure 5-1. Removing the Flow Cell

### 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 Section 5.4) does not eliminate these problems, replace the two silica flow cell 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 Section 5.4) and place it 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 you remove 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 Step 3 through Step 8 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 Section 5.2) does not remove the stain, or if excessive backpressure cracks the silica windows and you do not want to replace them (see Section 5.5).**

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 PeakNet 6 or 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 UV lamp connector and disconnect it from the 6-pin bulkhead connector on the side wall (see Figure 5-3).
9. Loosen the three screws in the deuterium lamp flange (see Figure 5-3) 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 isopropyl alcohol (IPA).
11. Line up the flange of the new lamp with the two locating pins and three threaded holes in the optical bench (see Figure 5-3). When the flange is aligned, gently push the lamp into the optical bench.

- 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.

- 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).
- Replace the lamp cover.

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

- Thread the flow cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket.

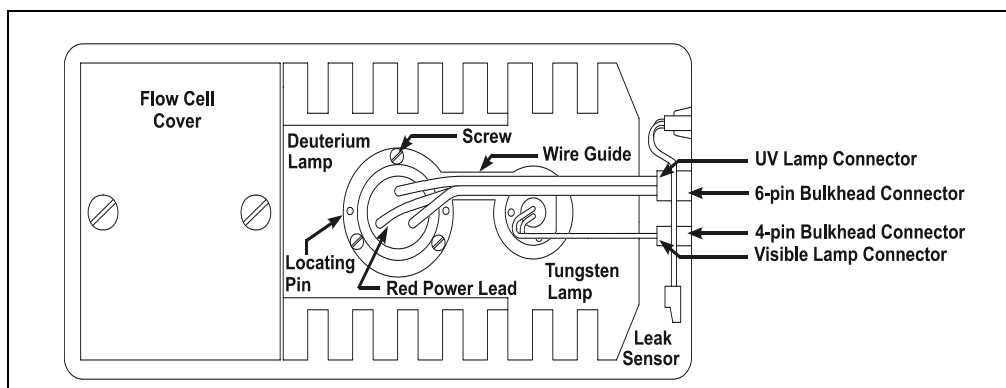


Figure 5-3. Lamp Connections

- Replace the flow cell cover and reconnect the inlet and outlet tubing.

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

- Replace the detector front panel.
- Turn on the detector power.
- Start the PeakNet 6 or Chromeleon server.

20. Reset the lamp lifetime as follows:
  - a. Press F8 to display the Commands dialog box.
  - b. Select **UV** (to display UV-related commands).
  - c. Select **UVLampAge**.
  - d. Reset the lamp age to zero.
  - e. Click the **Execute** button.

**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 verification for the new lamp from the Wellness panel (see Section 4.15.2).



## 5.8 Replacing the Tungsten Lamp

1. Stop the PeakNet 6 or 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 Vis lamp connector and disconnect it from the 4-pin 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 isopropyl alcohol (IPA).

## PDA-100 Photodiode Array Detector

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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 PeakNet 6 or Chromeleon server.
19. Reset the lamp lifetime as follows:
  - a. Press F8 to display the Commands dialog box.
  - b. Select **UV** (to display UV-related commands).
  - c. Select **VisLampAge**.
  - d. Reset the lamp age to zero.
  - e. Click the **Execute** button.

**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; now, using a small screwdriver (or your fingernails), push one of the locks toward the center to release it. The fuse holder pops out slightly when the lock is released. Repeat for the other lock.

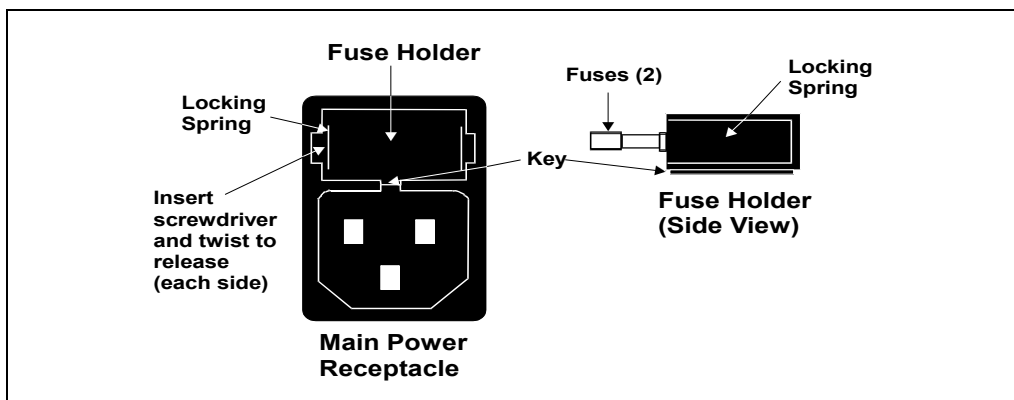


Figure 5-4. Main Power Fuse Holder

## ***PDA-100 Photodiode Array Detector***

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4. When both locks are released, pull the fuse holder straight out of the compartment and 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 panel.
6. Reconnect the power cord and turn on the power.

# A • Specifications

---

## A.1 Physical

**Dimensions** 17.5 cm H x 38.6 cm W x 49.0 cm D  
(6.9 x 15.2 x 19.3 in)  
6 cm (2.4 in) clearance required behind the detector

**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.2 Environmental

**Operating Temperature** 4 °C to 40 °C (40 °F to 104 °F)

**Operating Humidity** 5% to 95% relative humidity, noncondensing

## A.3 Electrical

**Main Power** 85 Vac to 264 Vac, 47/63 Hz. The input power is 162 W, maximum; the output power is 130 W, maximum. The maximum input current is 4 amps.  
The auto-sensing power supply requires no voltage or frequency adjustment.

**Fuses** Two 4.0 amp fast-blow IEC 127 fuses (P/N 954763)

### **A.4 Detector**

<b>Optical System</b>	Single-beam, reverse-optics design with concave holographic grating
<b>Light Sources</b>	Deuterium lamp (30 W) for ultraviolet spectrum analysis Tungsten lamp (15 W) for visible spectrum analysis
<b>Photodiode Array</b>	1024 elements
<b>Wavelength Range</b>	190 nm to 800 nm
<b>Pixel Resolution</b>	0.7 nm
<b>Wavelength Accuracy</b>	$\pm 1$ nm
<b>Noise</b>	$\pm 10$ $\mu$ AU with 2-second rise time, 254 nm, 4 nm bandwidth, and flowing deionized water $\pm 15$ $\mu$ AU with 2-second rise time, 520 nm, 10 nm bandwidth, and flowing deionized water
<b>Drift</b>	$< 500$ $\mu$ AU/hour (after warm-up)
<b>Linearity</b>	$> 2$ AU
<b>Analog Outputs</b>	Four built-in analog outputs
<b>Voltage Out</b>	1 V output
<b>Analog Output Range</b>	0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale
<b>Control and Data Evaluation</b>	Provided by PeakNet 6 or Chromeleon software; communication with the detector is via the DX-LAN interface

## A.5 Flow Cells

<b>Cell Body</b>	PEEK or stainless steel
<b>Volume and Optical Path Length</b>	PEEK cell: 13 $\mu\text{L}$ volume; 10 mm (0.394 in) path length Stainless steel cell: 13 $\mu\text{L}$ volume; 10 mm (0.394 in) path length
<b>Maximum Operating Pressure</b>	PEEK cell: 2 MPa (300 psi) Stainless steel cell: 3 MPa (500 psi)
<b>Chemical Compatibility</b>	<ul style="list-style-type: none"> <li>• Do not use bases stronger than 0.1 M; they will etch the silica windows of the flow cells.</li> <li>• Do not use normal phase or chlorinated solvents with the PEEK flow cell; they will damage the cell.</li> <li>• Do not use high pH salts with the stainless steel flow cell; they will cause corrosion, contamination, and metal leaching.</li> </ul>

## A.6 Heat Exchangers

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

<b>Maximum Operating Pressure</b>	PEEK cell: 2 MPa (300 psi) Stainless steel cell: 3 MPa (500 psi)
<b>Volume</b>	PEEK cell: 20 $\mu\text{L}$ Stainless steel cell: 9 $\mu\text{L}$





### B.1 Facilities Required

- Make sure the PDA-100 installation site meets the electrical and environmental specifications listed in Appendix A.
- Install the PDA-100 on a sturdy workbench. Allow at least 6 cm (2.4 in) of clearance behind the PDA-100 for power connections and ventilation. Do not block air flow to the detector; during operation, room air is 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, flow cell cover, and lamp cover are in place.
- Optical detectors are sensitive to vibrations. Make sure the PDA-100 installation site is vibration-free.

### B.2 Unpacking

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

**IMPORTANT**

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

- Do not turn on the PDA-100 unless it is at room temperature (10 °C to 40 °C; 50 °F 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
DX-600	P/N 057061	PEEK	P/N 056471
BioLC	P/N 054105	PEEK	P/N 056471
Summit®	P/N 054100	Stainless Steel	P/N 054103

## B.3 Installation

### B.3.1 Power



**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.

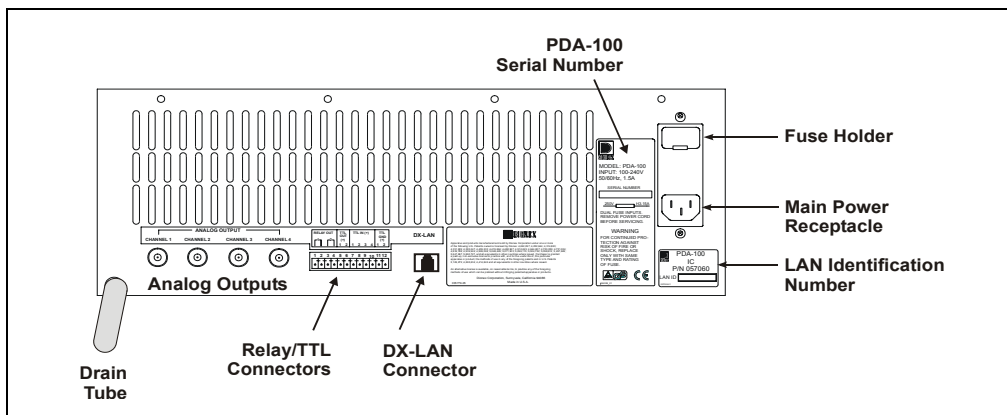


**STROMSCHLAGEFAHR**—Zur Vermeidung von elektrischen Schlägen ist eine geerdete Steckdose zu verwenden. Das Gerät darf nicht ohne Erdung betrieben bzw. an Wechselstrom angeschlossen werden.



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

The PDA-100 power supply is auto-sensing, so it is not necessary to manually select the line voltage or frequency. To control power to the detector, connect a modular power cord (IEC 320 C13) from the PDA-100 main power receptacle (see Figure B-1) to a grounded, single-phase power source. Use the PDA-100 power switch (see Figure 2-1) to turn the detector on and off.



*Figure B-1. PDA-100 Rear Panel*

### **B.3.2 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 salts; the salts can cause corrosion, contamination, and metal leaching.**

The flow cell is installed in the detector at the factory. Follow the steps below to complete the cell installation.

1. Grasp the PDA-100 front panel by the sides and pull straight off to remove.
2. Connect the cell inlet tubing to the union fitting on the heat exchanger tubing (see Figure B-2).

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3. Connect a backpressure line made from 0.25-mm (0.010-in) ID tubing to the cell outlet connection (see Figure B-2).

The backpressure line should provide enough restriction to generate the small amount of backpressure (0.14 MPa to 0.34 MPa; 20 psi 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. Cut the 0.25-mm (0.010-in) ID tubing to the required length. The backpressure line should be 1 meter long for a flow rate of 1.0 mL/min, 2 meters for 0.5 mL/min, and so on.
- 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.

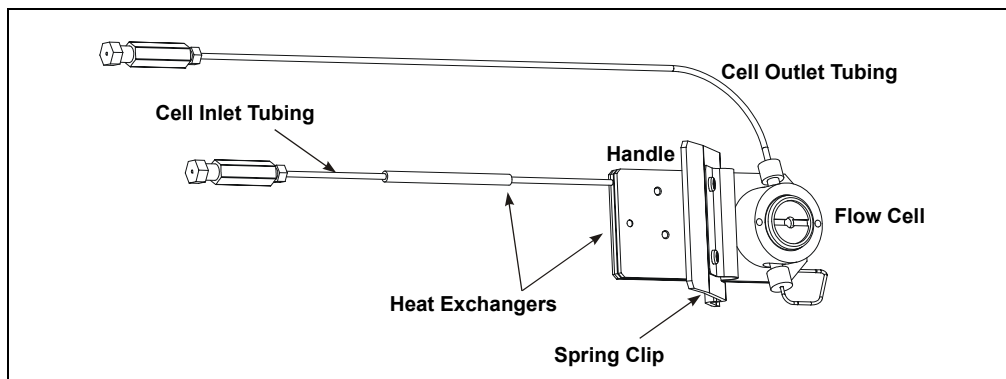


Figure B-2. Flow Cell Connections

4. The cell connections exit the detector through slots in the front panel. 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. Push the front panel into place.
5. Connect the cell outlet tubing to the separator column outlet.

**IMPORTANT**

Operating at backpressures greater than 2 MPa (300 psi) or 3 MPa (500 psi) will damage the PEEK or stainless steel cell heat exchanger, respectively.

### **B.3.3 Waste Line**

Connect the waste line (P/N 054779) to the PDA-100 rear panel (see Figure B-1). Place the free end of the line in a waste container below the level of the lab bench 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.3.4 Detector Output (Analog)**

The PDA-100 provides four analog outputs controlled by absorbance wavelengths 1 through 4 in PeakNet 6 or Chromeleon software. They provide a 1 volt output maximum. The software controls the full-scale absorbance output and offset within the 0 to 1 volt range. Connect a recorder or an integrator to the **ANALOG OUT** connector on the PDA-100 rear panel (see Figure B-1).

**B.3.5 Relay/TTL Control**



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 card.



**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.**

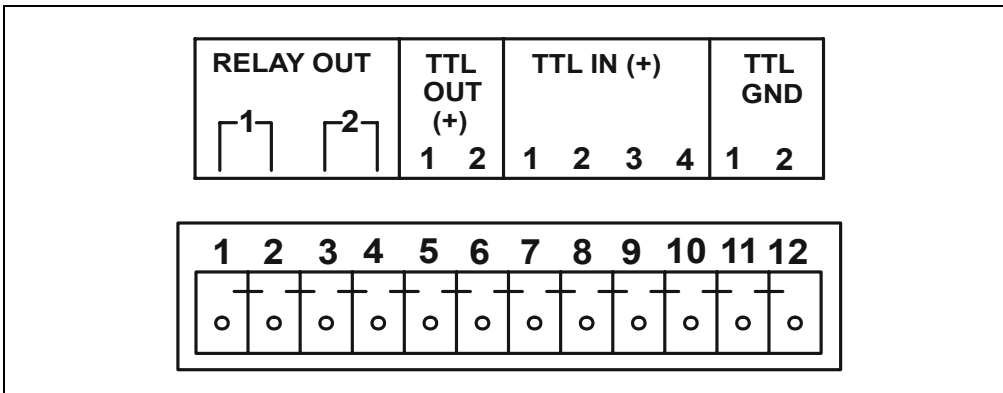


**DANGER D'ÉLECTROCUTION ET D'INCENDIE**—Le module d'alimentation électrique fonctionne aux potentiels du secteur. Faites effectuer toutes les réparations par un personnel qualifié.



**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-3). 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-3. Relay and TTL Connector*

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

<b>Pin</b>	<b>Assignment</b>	<b>Input Pin Function</b>	
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	Remote inject
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	

### B.3.6 DX-LAN Interface

In order to communicate with a host computer running Dionex software, the DX-LAN interface must be installed. Connect an unshielded twisted-pair DX-LAN cable (P/N 960281) from the RJ-45 (10BASE-T) connector on the PDA-100 rear panel to a “combo” 10BASE-T Ethernet hub (P/N 056909).

#### Installing the DX-LAN Cable

1. Install the “combo” 10BASE-T Ethernet hub (P/N 056909) on a workbench or on the wall. For installation instructions and site requirements, refer to the installation guide shipped with the hub.
2. Plug the 10BASE-T DX-LAN cable (P/N 960281) into a 10BASE-T port on the front panel of the hub.

#### IMPORTANT

The 10BASE-T DX-LAN cable is a Category 5 unshielded twisted-pair cable. Do not substitute a cable of an inferior grade. Failure to use the correct cable will cause the PDA-100 to lose communication with the host computer.

3. Connect the other end of the cable into the 10BASE-T DX-LAN connector on the PDA-100 rear panel (see Figure B-1).
4. Connect a 10BASE-T cable (P/N 960281) from a 10BASE-T port on the hub to the 10BASE-T port on the host computer’s internal DX-LAN card. If the connection is via port 8 on the hub, set the Normal/Uplink push button to Normal.

**NOTE** Installation instructions for the host computer’s internal DX-LAN card are in *Installing the Dionex PeakNet 6 System* (Document No. 031631).

#### IMPORTANT

When using 10BASE-T cabling, always install an Ethernet hub. If you simply plug the 10BASE-T cable from the PDA-100 into the connector on the host computer’s DX-LAN card, the connection will not work.

#### Cascading Hubs

Cascading, or connecting hubs together through their 10BASE-T ports, increases the number of ports or the number of users supported on the network. For instructions, refer to the installation guide for the hub.



## C • Reordering Information

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Part Number	Item	Quantity
939016T	Deuterium Lamp	1
056123T	Tungsten Lamp	1
954763	Fuse, 4 amps (Fast-Blow IEC 127)	1
923686	12-Pin Relay/TTL Connector	1
043598	Twisted Wire Assembly	2 m (8 ft)
<i>PDA-100 with PEEK Flow Cell</i>		
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
<i>PDA-100 with Stainless Steel Flow Cell</i>		
056126	Stainless Steel Flow Cell (with heat exchangers)	1
060038	Stainless Steel Flow Cell Outlet Tubing Assembly	1
055902	Union Fitting	1
010262	Ferrule	1
010261	Nut	1
060498	Flow Cell Windows Replacement Kit	1



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