

# Dionex ICS Series Photodiode Array Detector Operator's Manual

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# Dionex PDA Operator's Manual

### 1.1 Detector Overview



Figure 1-1. Thermo Scientific Dionex ICS Series Photodiode Array Detector

The Thermo Scientific Dionex<sup>™</sup> ICS Series Photodiode Array Detector (Dionex PDA) (P/N 074114) is an optical chromatography detector capable of measuring absorbance signals and absorbance spectra from 190 to 800 nm. The Dionex PDA can be used with the following Thermo Scientific Dionex systems: the ICS-5000 (analytical systems only), ICS-2100, ICS-1600, ICS-1100, and ICS-900 (as well as some legacy systems). The Dionex PDA can be used in numerous laboratory environments for routine analysis, as well as for sophisticated research tasks.

A deuterium lamp optimizes the UV range (190 to 380 nm) and a tungsten lamp optimizes the visible range (380 to 800 nm). When using the Dionex PDA, up to five single wavelengths (2D chromatograms) can be collected without the necessity for collecting 3D data. Collecting individual wavelengths instead of the spectra offers two advantages: it eliminates the need to perform signal extractions for analyses that do not require spectral data and it conserves disk space.

Key features of the Dionex PDA include:

- The deuterium and tungsten lamps ensure low baseline noise, high signal intensity, and applications flexibility in the wavelength range from 190 to 800 nm
- The 1024-element photodiode array optimizes spectral resolution.
- The built-in holmium oxide filter verifies wavelength calibration accuracy.
- Five flow cells are available for standard, semi-micro, and semi-preparative applications.
- LEDs on the front cover provide detector status at a glance.
- Four analog outputs support data collection on alternate data collection systems.
- The detector is controlled with a PC (personal computer) running Microsoft<sup>®</sup> Windows Vista<sup>®</sup> or Windows<sup>®</sup> XP and either the Chromeleon<sup>®</sup> 7 Chromatography Data System or the Chromeleon<sup>®</sup> 6.8 Chromatography Data System. Chromeleon 7 and Chromeleon 6.8 also provide data acquisition and data processing functions.

NOTE Thermo Scientific Dionex Chromeleon® Xpress software can provide real-time control and monitoring of the detector, but does not include data management capabilities.

# 1.2 Theory of Photodiode Array Detection

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

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

# 1.2.1 Advantages of Photodiode Array Detection

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

Photodiode array 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 photodiode array detector is used, a wavelength range can be programmed and all compounds that absorb within this range can be detected in a single analysis.

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

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

## Detection of multiple wavelengths

A photodiode array 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 photodiode array detector can analyze peak purity by comparing spectra within a peak. The better the spectra match, the higher the possibility that the peak is pure.

### • Positive peak identification

In liquid chromatography, peak identification is usually based on relative retention times. When a photodiode array detector is used,

spectra are automatically collected as each peak elutes. The detector 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 1-2 shows an overlay of pyrene and an impurity obtained from two consecutive chromatographic analyses. Based on 2D data, or if the analyses had been performed with a conventional variable wavelength detector, the peaks might have been misidentified as the same component.

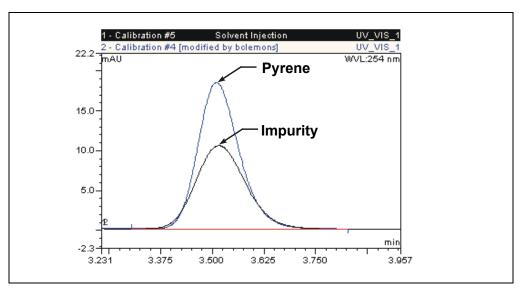


Figure 1-2. Overlay of Pyrene and an Impurity

The Dionex PDA, under the control of Chromeleon 6.8 with the 3D Data Acquisition license, correctly identified and differentiated the components. The spectra of pyrene and the impurity (see <u>Figure 1-3</u>) clearly indicate that the two peaks are different components.

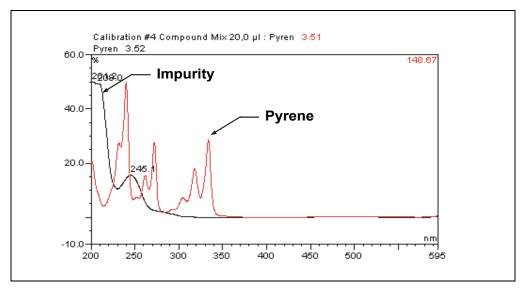


Figure 1-3. Spectra of Pyrene and an Impurity

# 1.3 Operator's Manual Overview

The electronic version (i.e., the PDF file) of this manual contains numerous hypertext links that can quickly take you to other locations within the file. These links include:

- Table of contents entries
- Index entries
- Cross-references (underlined in blue) to sections, figures, tables, etc.

If you are not familiar with how to navigate PDF files, refer to the Help system for Adobe<sup>®</sup> Acrobat<sup>®</sup> or Adobe Reader<sup>®</sup> for assistance.

Chapter 1	An introduction to the Dionex PDA and the theory of
Introduction	photodiode array detection; includes an explanation of
	conventions used in this manual.
Chapter 2	An overview of Dionex PDA operating features; includes
Description	an introduction to the chromatography software
•	(Chromeleon) required for detector control.
Chapter 3	Instructions for routine Dionex PDA operation with
Operation and	Chromeleon and guidelines for optimizing Dionex PDA
Maintenance	performance. Preventive maintenance procedures.
Chapter 4	Minor problems that may occur during operation, with
Troubleshooting	step-by-step procedures for how to isolate and eliminate
<u> </u>	the cause of each problem. A list of Chromeleon Audit
	Trail error messages, with an explanation of the possible
	cause of each message and the corrective action to take.
Chapter 5	Step-by-step instructions for routine service and parts
Service	replacement procedures the user can perform for the
	Dionex PDA.
Appendix A	Specifications and installation site requirements for the
Specifications	Dionex PDA.
Appendix B	Installation instructions for the Dionex PDA.
Installation	
Appendix C	Spare parts for the Dionex PDA.
Reordering	
Information	

# 1.4 Safety and Regulatory Information

The Dionex PDA was manufactured by Thermo Fisher Scientific at the following location: 527 Lakeside Drive, Sunnyvale, CA 94088-3603 U.S.A. The Dionex PDA is designed for use with IC (ion chromatography), BioLC<sup>®</sup> (biocompatible liquid chromatography), and HPLC (high-performance liquid chromatography) systems and should not be used for any other purpose. Operation of the Dionex PDA in a manner not specified by Thermo Fisher Scientific may result in personal injury.

If there is a question regarding appropriate usage, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

## 1.4.1 Safety Messages and Notes

This manual contains warnings and precautionary statements that can prevent personal injury and/or damage to the Dionex PDA 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.



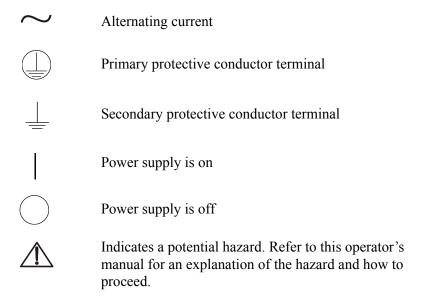
Bedeutet eine mögliche Gefährdung. Mißachtung kann zu kleineren oder mittelschweren Verletzungen führen. Wird auch verwendet, wenn eine Situation zu schweren Schäden am Gerät führen kann, jedoch keine Verletzungsgefahr besteht.

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

# 1.4.2 Safety Labels

These symbols appear on the Dionex PDA or on Dionex PDA labels.



# 1.4.3 Declaration of Conformity

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

# Dionex PDA Operator's Manual

# 2.1 Front Features

<u>Figure 2-1</u> illustrates the features on the front of the Thermo Scientific Dionex ICS Series Photodiode Array Detector (Dionex PDA).

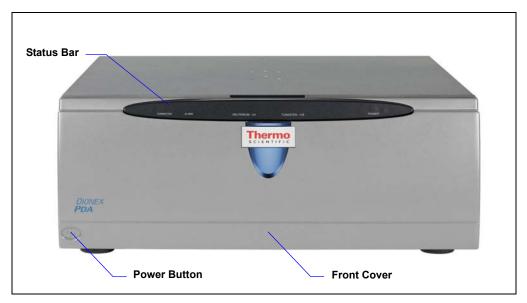


Figure 2-1. Dionex PDA Front Features

- The status bar provides LEDs (light emitting diodes) that indicate the status of several Dionex PDA components and functions (see Figure 2-2).
- Use the power button for routine on/off control of the Dionex PDA.
  - NOTE The main power switch is on the Dionex PDA rear panel (see <u>Figure 2-8</u>). Always leave the main power switch on unless instructed to turn it off (for example, before performing a service procedure).
- The front cover provides access to the components inside the Dionex PDA. To remove the cover, grasp the cover by the sides and pull it straight off.

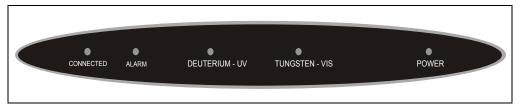


Figure 2-2. Dionex PDA Status Bar

LED Label	LED Status	Function
CONNECTED	Lighted	The Dionex PDA is connected to a Chromeleon 7 instrument or a Chromeleon 6.8 timebase.
	Off	The Dionex PDA is not connected to a Chromeleon 7 instrument or a Chromeleon 6.8 timebase.
ALARM	Lighted	A problem has occurred (a leak, for example). Check the Chromeleon Audit Trail for the cause.
	Off	No problems have been detected.
DEUTERIUM – UV	Lighted	The deuterium lamp is on.
	Flashing	The LED flashes once every second while the deuterium lamp is firing.  The LED flashes every 0.5 second if a command is issued to turn on the lamp, but the lamp fails to turn on (see Section 4.3). If a subsequent attempt to turn on the lamp is successful, the LED will automatically stop flashing.
	Off	The deuterium lamp is off.
TUNGSTEN – VIS	Lighted	The tungsten lamp is on.
	Flashing	The LED flashes once every second while the tungsten lamp is firing.  The LED flashes every 0.5 second if a command was issued to turn on the lamp, but the lamp failed to turn on (see Section 4.3). If a subsequent attempt to turn on the lamp is successful, the LED will automatically stop flashing.  The tungsten lamp is off.
POWER	Lighted	The Dionex PDA power is on.
	Off	The Dionex PDA power is off.

## 2.2 Inside Front Panel

Grasp the Dionex PDA front cover by the sides and pull it straight off to access the inside front panel (see Figure 2-3).

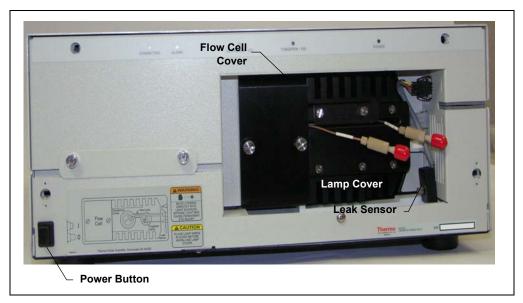


Figure 2-3. Dionex PDA Inside Front Panel

- 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 will not be apparent immediately. To prevent damage to detector components, the bottom of the optical bench contains a built-in leak tray. A sensor in the sump of the leak tray responds to changes in the index of refraction when wet.

When the leak sensor is activated, it triggers an alarm in the detector, the **ALARM** LED lights (see <u>Figure 2-1</u>), and an error message is logged in the Chromeleon Audit Trail. After fixing the leak and drying the sensor, clear the **ALARM** LED (see Section 4.2).

# 2.3 Optical System

Figure 2-4 is a schematic of the Dionex PDA optical system.

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

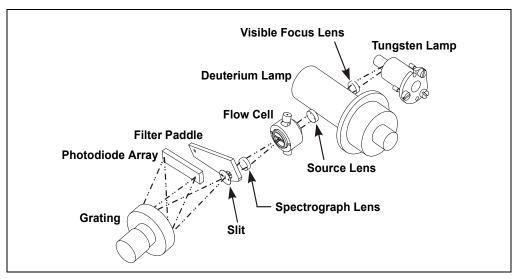


Figure 2-4. Dionex PDA Optical System

Optical System Component	Function		
Tungsten Lamp	The light source for visible and near-infrared wavelengths (380 to 800 nm). The tungsten lamp is focused through an opening in the internal structure of the deuterium lamp; this allows the lamps to share the optical axis to the source lens.		
Visible Focus Lens	Focuses the visible light from the tungsten lamp to the throughhole of the deuterium lamp.		
Deuterium Lamp	The light source for UV wavelengths (190 to 380 nm).		
Source Lens	Receives the light from the tungsten and deuterium lamps and focuses it so that the beam passes through the flow cell.		
Flow Cell	The measurement site for sample absorbance. The cell has a flat window on each side. Thermo Fisher Scientific offers five cells for use with the Dionex PDA: standard cells (in both PEEK and 316 stainless steel), semi-micro cells (in both PEEK and 316 stainless steel), and semi-preparative cells (in PEEK only).		
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:		
	• Open = Light passes, unobstructed, along the light path when the holmium oxide filter is moved out of the light path.		
	<ul> <li>Blocked = No light passes along the light path; used to measure the dark signal without turning off the lamps.</li> </ul>		
	• <i>Holmium</i> = Places the holmium oxide filter in the light path; used to verify wavelength accuracy.		
Slit	The width of the slit (equivalent to 1 nm) optimizes the optical resolution.		
Grating	Diffracts the light beam into its component wavelengths and directs the light onto the photodiode array.		
Photodiode Array	A series of 1024 photosensitive elements. Each element measures a narrow band of the spectrum. A spectrum is obtained by measuring the light intensity of each wavelength and reporting the results over the selected wavelength range.		

### 2.3.1 Flow Cells

Table 2-1 lists the flow cells available for use with the Dionex PDA.

STANDARD FLOW CELLS							
Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range			
PEEK (P/N 056346)	13 μL	10 mm	45 μL	0.5 to 5.0 mL/min			
316 stainless steel (P/N 056126)	13 μL	10 mm	20 μL	0.5 to 5.0 mL/min			
SEMI-MICRO FLOV	V CELLS						
Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range			
PEEK (P/N 064169)	3.1 μL	9 mm	5 μL	Up to 1.0 mL/min			
316 stainless steel (P/N 064168)	3.1 μL	9 mm	5 μL	Up to 1.0 mL/min			
SEMI-PREPARATIV	SEMI-PREPARATIVE FLOW CELL						
Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range			
PEEK (P/N 064167)	0.7 μL	0.4 mm	N/A	5.0 to 100.0 mL/min			

Table 2-1. Dionex PDA Flow Cells

IMPORTANT

Do not touch the cell windows. If you touch a window, clean it with isopropyl alcohol (IPA) and a clean lens tissue.

IMPORTANT

Strong bases can etch the fused 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. If strong base remains in the cell for 1 to 2 days, the cell windows may need to be replaced (see Section 5.5).

IMPORTANT

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

IMPORTANT

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

#### **Standard Flow Cell**

The standard flow cell assembly (see <u>Figure 2-5</u>) is available in PEEK and 316 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.

The standard cell is designed for applications with 3- to 4-mm ID separator columns and flow rates of 0.5 to 5.0 mL/min.

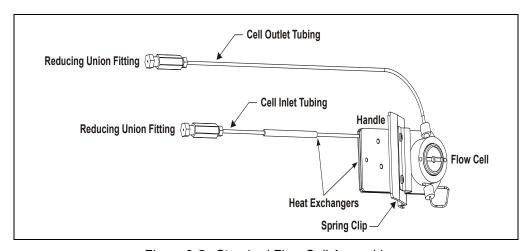


Figure 2-5. Standard Flow Cell Assembly

Standard Cell Component	PEEK Cell	SST Cell
Inlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A
Outlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A
Inlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 056124
Outlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 051650
Reducing union fitting	P/N 055903	P/N 055902

#### Semi-Micro Flow Cell

The semi-micro flow cell assembly (see <u>Figure 2-6</u>) is available in PEEK and 316 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.

The semi-micro flow cell is designed for applications with 2- to 3-mm ID separator columns and flow rates up to 1.0~mL/min.

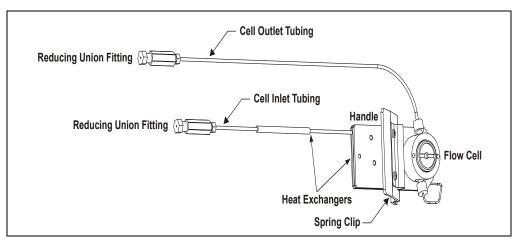


Figure 2-6. Semi-Micro Flow Cell Assembly

Semi-Micro Cell Component	PEEK Cell	SST Cell
Inlet tubing: 0.12-mm (0.005-in) ID	P/N 063897	N/A
Outlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A
Inlet tubing: 0.12-mm (0.005-in) ID	N/A	P/N 063903
Outlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 051650
Reducing union fitting	P/N 055903	P/N 055902

### **Semi-Preparative Flow Cell**

The semi-preparative flow cell assembly (see <u>Figure 2-7</u>) is available in PEEK only. The cell is designed for use with semi-preparative flow applications and flow rates of 5.0 to 100.0 mL/min. The 0.4-mm path length minimizes detector absorbance for concentrated peaks.

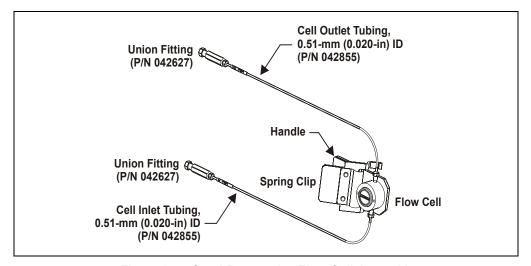


Figure 2-7. Semi-Preparative Flow Cell Assembly

### 2.4 Electronics



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



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



Halten Sie sich von der Elektronik des Dionex PDA 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 power source and provides regulated, low-voltage power for the Dionex PDA. Power entry components are shielded and filtered to prevent electromagnetic interference (EMI).



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



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



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

### **Lamp Power Supply Board**

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

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



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



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



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

### **Preamp Board**

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

## **Data Processing Board**

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

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

### 2.5 Rear Panel

<u>Figure 2-8</u> illustrates the rear panel of the Dionex PDA.

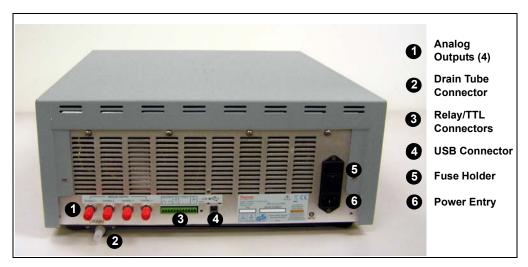


Figure 2-8. Dionex PDA Rear Panel

#### **ANALOG OUTPUT Connectors**

The four analog outputs are standard female BNC connectors. The output range is 0 to 1 volt. The full-scale absorbance range can be set to 0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale in Chromeleon. For details about programming the analog outputs, see Section 3.3.2.

The analog outputs will continue to output data after the PC is turned off or is disconnected from the Dionex PDA. When the detector power is turned on again, the analog output settings will be either the factory defaults (see <u>Section 3.3.3</u>) or the settings selected the last time the detector was controlled by the computer.

#### **Drain Tube Connector**

Waste exits the detector via a drain tube (P/N 055075) connected at the rear panel. When correctly installed, the drain tube is routed downward, below the leak tray. For installation instructions, see Section B.4.



Check periodically to verify that the drain tube is not clogged or bowed upward, and that the tubing remains routed below the leak tray. If the drain tube is above the drain port, the drip tray may back up and overflow inside the detector.

#### **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 Chromeleon 7 instrument method or Chromeleon 6.8 program (see Section 3.3.2), or controlled manually 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.

The Dionex PDA Ship Kit (P/N 060977) includes a 12-pin connector (P/N 923686) for relay and TTL connections. For installation instructions, see Section B.6.

#### **USB Connector**

The USB connector allows communication between the Dionex PDA and the PC on which Chromeleon is installed. The Dionex PDA Ship Kit (P/N 060977) includes one USB cable (P/N 063246). For installation instructions, see Section B.7.4.

### **Power Entry**

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

NOTE Always leave the main power switch on unless instructed to turn it off (for example, before performing a service procedure). Use the power button on the front of the detector for routine on/off control.



SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the Dionex PDA or connect it to AC power 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 Dionex PDA and is easily accessible.



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



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



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



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



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

#### **Fuses**

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



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



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



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

# 2.6 Operating Guidelines

#### 2.6.1 Mobile Phases

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

IMPORTANT

Strong bases can etch the fused 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. If strong base remains in the cell for 1 to 2 days, the cell windows may need to be replaced (see Section 5.5).

IMPORTANT

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

IMPORTANT

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

- Prepare all mobile phases with spectroscopy-grade solvents, reagent-grade chemicals, and ASTM Type I (18 megohm-cm) filtered, deionized water that meets the specifications listed in Table B-1.
- 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 Dionex PDA 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.

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

IMPORTANT

To avoid contamination, always wear latex gloves that are lint-free, particle-free, and oil-free when handling pump components in the flow path. Contamination may cause baseline disruptions, spurious peaks, and inconsistent results, especially in sensitive applications.

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

- 1. Remove the column and the suppressor (if installed).
- 2. Connect the line exiting the injection valve directly to the flow cell inlet tubing.
- 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 pump 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.

# 2.7 Chromatography Software

### 2.7.1 Overview

The Dionex PDA is controlled by a PC configured with either the Chromeleon 7 Chromatography Data System or the Chromeleon 6.8 Chromatography Data System. Both software products provide extensive instrument control, data management, reporting, and compliance features.

In Chromeleon 7, an *ePanel Set* provides centralized system control. You can use the ePanel Set to view system status information and issue commands for controlling each module. In Chromeleon 6.8, these functions are available on a *panel tabset*.

In both the ePanel Set and the panel tabset, a convenient **Home** panel shows the overall system status and provides basic module control

functions. The UV\_Vis tab provides access to Dionex PDA functions, as well as detailed status and diagnostics information. <u>Figure 2-9</u> shows the Dionex PDA ePanel in Chromeleon 7.

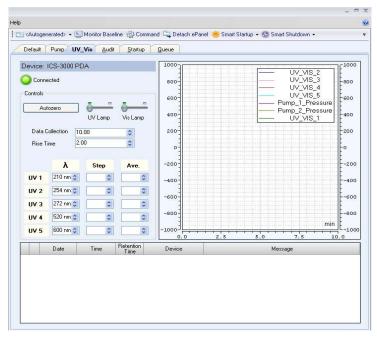
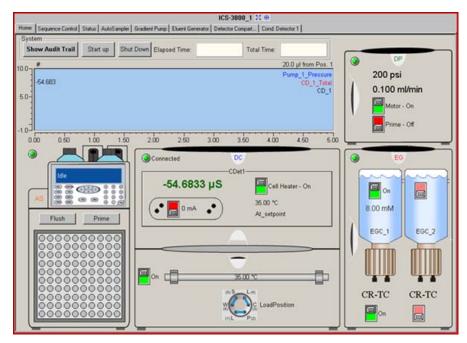


Figure 2-9. Example Dionex PDA ePanel in Chromeleon 7



<u>Figure 2-10</u> shows the Dionex PDA Control panel in Chromeleon 6.8.

Figure 2-10. Example Panel Tabset in Chromeleon 6.8

Two modes of software control are available: direct control and automated control.

- With *direct* control, you select operating parameters and commands from the Chromeleon 7 ePanels (or Command window) or the Chromeleon 6.8 Control panels (or Commands dialog box). Direct control commands are executed as soon as they are entered. See <u>Section 3.3</u> for details about direct control.
- With *automated* control, you create a Chromeleon 7 instrument method or a Chromeleon 6.8 program. An instrument method or program is a list of control commands, executed in chronological order, for automated operation of the detector. It can be created automatically (with the help of a software wizard) or manually (by editing an existing instrument method or program). See <u>Section 3.3</u> for details about automated control.

## 2.7.2 3D Data Acquisition License

To take full advantage of the Dionex PDA's capabilities, order Chromeleon with the 3D Data Acquisition license. This license is required in order to perform 3D data presentation and spectral analysis (real-time or post-run), peak purity analysis with selectable criteria, and spectral library search for positive peak identification.

Chromeleon supports a maximum of two detectors that can record a 3D field (for example, photodiode array or electrochemical detectors) on one Chromeleon 7 instrument controller or Chromeleon 6.8 instrument server. The total number of 3D data channels one instrument controller or server can acquire simultaneously depends on computer performance.

A 3D Data Acquisition license is not required to display and process data, only to acquire it. Thus, data acquired on a station equipped with the 3D Data Acquisition license can be reviewed and processed on any Chromeleon client station.

## 2.7.3 System Wellness and Predictive Performance

System Wellness monitors the overall "health" of the chromatographic system. It provides built-in diagnostic and calibration features that help prevent unscheduled system shutdowns and assure reliable operation of system devices.

*Predictive Performance* provides various functions for estimating the lifetime of replaceable parts and for planning and recording service and qualification procedures.

A partial list of the System Wellness and Predictive Performance features is provided below. Refer to the Chromeleon Help for a complete list of the available features.

- Recording of detector properties, including the serial number, firmware version, and operating hours
- Monitoring of lamp properties, including lamp ignitions, lamp operation time, and lamp intensity
- Setting of limits for the lamp intensity and lamp operation time, including display of warning messages when limits are exceeded
- Reminders for service and qualification periods, including display of warning messages when limits are exceeded

- Leak detection
- Wavelength verification

System Wellness and Predictive Performance commands and parameters are available in the Chromeleon 7 Command window or the Chromeleon 6.8 Commands dialog box.

## Dionex PDA Operator's Manual

## 3 • Operation and Maintenance

## 3.1 Getting Started

## 3.1.1 Initial Startup

- 1. If you are starting the Thermo Scientific Dionex ICS Series Photodiode Array Detector (Dionex PDA) for the first time, press the main power switch on the rear panel (see Figure 2-8).
- 2. Press the power button on the front of the detector (see <u>Figure 2-1</u>).

After initial startup, leave the main power switch on unless instructed to turn it off (for example, before performing a service procedure).

If the detector is installed with a Dionex ICS-5000 or Dionex ICS-3000 system, use the power button on the front of the Dionex Dual Pump (DP) or Dionex Single Pump (SP) for routine on/off control. To turn off the pump, press and hold the power button for 2 seconds.

If the detector is *not* installed with a Dionex ICS-5000 or Dionex ICS-3000 system, use the power button on the front of the Dionex PDA for routine on/off control.

- Check the status of the LEDs on the front of the detector: The following LEDs should be lighted: POWER, DEUTERIUM – UV, and/or TUNGSTEN – VIS LED.
- 4. Wait 20 to 30 minutes for the lamps to stabilize. (For sensitive applications work or for detector validation, allow at least 2 hours. After installing a new lamp, allow 8 to 24 hours.)

#### 3.1.2 Connect to the Chromeleon 7 Client

1. Start the Instrument Controller Service, if it is not already running. If the Chromeleon tray icon on the taskbar is crossed out in red , the Instrument Controller Service is not running. To start it, right-click the icon and select **Start Chromeleon Instrument Controller**. The icon changes to gold to indicate that the Instrument Controller Service is starting. When the Instrument Controller Service is running (idle), the icon changes to gray .

If the Chromeleon tray icon is not on the taskbar, click **Start > All Programs > Chromeleon 7 > Services Manager** to open the Services Manager and click **Start Instrument Controller**.

- 2. Start the Chromeleon 7 client:
  - a. Click **Start** on the Windows taskbar and select **All Programs > Chromeleon 7 > Chromeleon 7**.
  - b. Click the **Instruments** Category Bar on the Console.
  - c. On the Navigation Pane, click the instrument that includes the detector. The ePanel Set for the instrument appears in the Console Work Area.
  - d. To display the detector ePanel, click the **UV\_Vis** tab on the ePanel Set (see Figure 3-1).
  - e. Verify that the **Connected** check box is selected. If it is not, click the box to connect the Dionex PDA to Chromeleon 7.

NOTE After starting Chromeleon 7 and connecting to the ePanel, the Connected LED on the detector's status bar is blue.

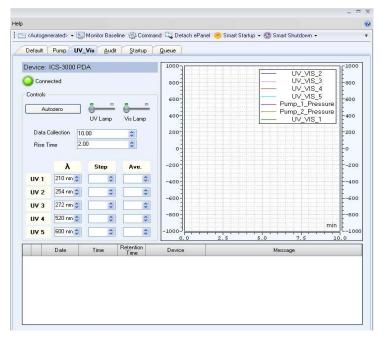


Figure 3-1. Example Dionex PDA ePanel in Chromeleon 7

## 3.1.3 Connect to the Chromeleon 6.8 Client

1. Start the Chromeleon server, if it is not already running. If the Chromeleon server icon on the taskbar is crossed out in red server is not running. To start the server, right-click the icon and select **Start Server**. When the server is running (and data acquisition is not occurring), the icon is gray

If the Server Monitor icon is not on the taskbar, click **Start** on the taskbar and select **All Programs > Chromeleon > Server Monitor**. Click **Start** to start the server.

- 2. Start the Chromeleon 6.8 client:
  - a. Click **Start** on the Windows taskbar and select **All Programs** > **Chromeleon** > **Chromeleon**.

- b. Select **View > Default Panel Tabset** or click the corresponding toolbar button to display the panel tabset.
- c. To display the Dionex PDA Control panel, select the **Dionex PDA** tab on the panel tabset (see Figure 3-1).

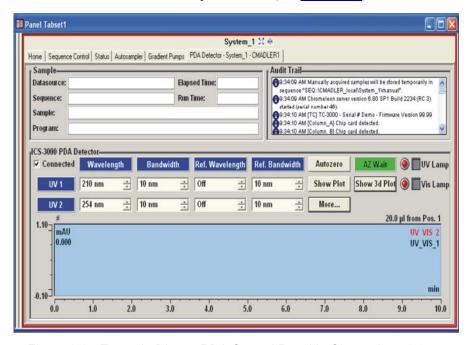


Figure 3-2. Example Dionex PDA Control Panel in Chromeleon 6.8

d. Verify that the **Connected** check box is selected. If it is not, click the box to connect the Dionex PDA to Chromeleon 6.8.

NOTE After starting Chromeleon 6.8 and connecting to the Control panel, the Connected LED on the detector's status bar is blue.

## 3.1.4 Turn On the Lamps

To turn on the lamps, click **UV Lamp** and **Vis Lamp** on the Chromeleon 7 ePanel (see <u>Figure 3-1</u>) or the Chromeleon 6.8 Control panel (see Figure 3-2).

When a detector lamp is turned on for the first time after the detector power is turned on, wavelength calibration is performed automatically. Calibration starts shortly after the lamp is ignited.

## 3.2 System Equilibration

Before you can begin using the Dionex PDA for sample analysis, the chromatography system must be equilibrated. To equilibrate the system, perform the following tasks:

- Pump the starting solvent through the entire system until the system is free of any other liquid composition.
- Heat or cool all temperature-controlled devices (for example, a column compartment) to the temperature required for the application.
- Set the detector wavelength(s) and turn on the lamp(s).
- Calibrate the wavelength(s).
- Monitor the pump pressure and verify that the reading is correct for the application and is stable.
- Monitor the detector signal and verify that the baseline signal is at the
  expected reading for your application and is stable. This may not be the case if
  the solvent composition has been modified, or if the light path contains air
  bubbles. See Section 4.5 and Section 4.6 for troubleshooting information.

NOTE To achieve optimum results, allow 20 to 30 minutes for the lamp(s) to stabilize before beginning operation. For sensitive applications work or for detector validation, allow at least 2 hours for the lamps to stabilize.

Select one of the following methods to perform the equilibration tasks:

• Select operating commands and parameters directly from the Chromeleon 7 ePanel Set or the Chromeleon 6.8 Control panel. See <u>Section 3.3.1</u> for details about direct control.

- Create and run an equilibration instrument method or program to automate the process. See Section 3.3.2 for details about automated control.
- If you are running Chromeleon, you can use the Smart Startup feature to automate system startup and equilibration. In Chromeleon 7, you first create an instrument method from which the Smart Startup settings are derived. In Chromeleon 6.8, you can use the Smart Startup Wizard to create a Smart Startup program. Refer to the Chromeleon Help for details.

## 3.3 Routine Operation

This section provides a brief overview of detector control with Chromeleon 7 or Chromeleon 6.8. For complete operating instructions, refer to the Chromeleon Help.

### 3.3.1 Direct Control

With direct control, you select operating commands and parameters from the following locations:

• Chromeleon 7 ePanel Sets or the Command window

-or-

• Chromeleon 6.8 Control panels or the Commands dialog box

Direct control commands are executed as soon as they are entered. Most routine direct control functions can be performed from ePanels or from Control panels.

For other functions, you can use the Command window or Commands dialog box. These provide access to all of the available commands and properties for the Dionex PDA, as well as commands for other devices in the instrument or timebase, respectively.

#### To issue direct control commands to the Dionex PDA:

- 1. Open the ePanel Set (see Section 3.1.2) or panel tabset (see Section 3.1.3) and click the tab for the detector.
- 2. Use the controls (buttons, sliders, etc.) on the panel to issue commands

3. If the function that you want to perform is not available from the panel, open the Command window (see the instructions below) or the Commands dialog box (see page 40).

The commands available in the Command window or Commands dialog box vary, depending on these variables:

- The version of Chromeleon installed
- The display filter level (**Normal**, **Advanced**, or **Expert**)
- The options selected for the detector in the Properties dialog box in the Chromeleon 7 Instrument Configuration program or the Chromeleon 6.8 Server Configuration program (see Section B.11.3)

### To open the Chromeleon 7 Command window:

- 1. Open the ePanel Set for the instrument that includes the Dionex PDA.
- 2. Click **Command** on the **Instrument** toolbar above the ePanel Set (or press the **F8** key).

The Command window appears (see Figure 3-3).

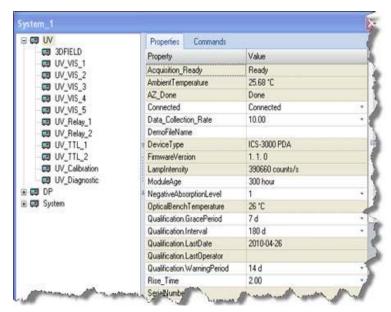


Figure 3-3. Command Window

- 3. Click **UV** and then click the **Properties** tab or **Commands** tab to view detector properties or commands, respectively. To execute a command, click the button next to the command.
  - NOTE To change the display filter level, right-click the list of commands and select the desired filter on the context menu.

#### To open the Chromeleon 6.8 Commands dialog box:

- 1. Open the Dionex PDA Control panel.
- Select Control > Commands (or press the F8 key).
   The Commands dialog box appears (see <u>Figure 3-4</u>).

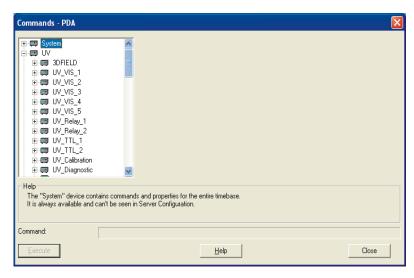


Figure 3-4. Commands Dialog Box

- 3. To see the commands available for the Dionex PDA, click the plus sign next to **UV**. To execute a command, click the **Execute** button.
  - NOTE To change the display filter level, right-click the list of commands and select the desired filter on the context menu.

#### 3.3.2 Automated Control

You can control the detector automatically by running instrument methods (in Chromeleon 7) or programs (in Chromeleon 6.8) that specify the function the detector and other system instruments should perform at a specific time. Instrument methods and programs can be created automatically (with the help of a software wizard) or manually (by editing an existing instrument method or program).

To prepare for automated control, create a *sequence* (a list of injections) and specify the instrument method or program to run on each injection. The sequence also includes a processing method (in Chromeleon 7) or a quantification method (in Chromeleon 6.8) for peak identification and area determination.

The table below lists the main steps required to set up and run the detector automatically. Refer to the Chromeleon Help for details about these steps.

In Chromeleon 7	In Chromeleon 6.8
Create a sequence and specify the instrument method files to use for each injection	Create a sequence and specify the program files to use for each injection
Load the sequence into the queue	Load the sequence into the batch
Start the queue	Start the batch

NOTE Before creating a program, review Section 3.4 for a description of the detector parameters, how they interact with each other, and guidelines for their selection.

## 3.3.3 Stand-Alone Analog Operation

This section describes limited, stand-alone operation of the Dionex PDA when the detector is not interfaced to a PC running Chromeleon. In standalone operation, TTL inputs on the Dionex PDA rear panel can be used to trigger autozero or to turn the lamps on and off. All other detector parameters remain at their factory default settings and cannot be changed.

#### **Factory Default Settings**

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

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

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

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

• Rise time: 2.0 seconds

• UV lamp: On

• Visible lamp: On

## **Stand-Alone Operation**

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

TTL Input	Dionex PDA Parame- ter
TTL 1 in	Autozero
TTL 2 in	Reserved
TTL 3 in	Deuterium lamp on/off
TTL 4 in	Tungsten lamp on/off

## 3.4 Optimizing Detector Performance

The performance of the Dionex PDA can be optimized by careful selection of key operating parameters. This section defines these parameters, describes how they interact, and offers guidelines for selecting them. The table below 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
Bandwidth	Sensitivity vs. baseline noise
Reference wavelength	Baseline drift
Reference bandwidth	Baseline noise, baseline drift
Bunch width	Spectral resolution, peak match, disk space
Step*	Baseline noise, rise time, data collection rate, disk space
Average*	Rise time, data collection rate
Negative absorbance	Peak detection, baseline noise, linearity
* 0	1 (0 1 111 ) (1 1 7

<sup>\*</sup> Set automatically by Chromeleon 6.8; not available in Chromeleon 7

#### Flow Cell Material

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



Strong bases can etch the fused 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. If strong base remains in the cell for 1 to 2 days, the cell windows may need to be replaced.



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

IMPORTANT

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

#### **Rise Time**

Rise time is a measure of how quickly the Dionex PDA responds to a change in signal. The selected rise time is applied to all data collected, as well as to both analog and digital output. The Dionex PDA rise time settings are 0.05, 0.1, 0.2, 0.5, 1.0, 2.0 (default), or 5.0 seconds.

Select a rise time that is about 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 <u>Figure 3-5</u>). 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.

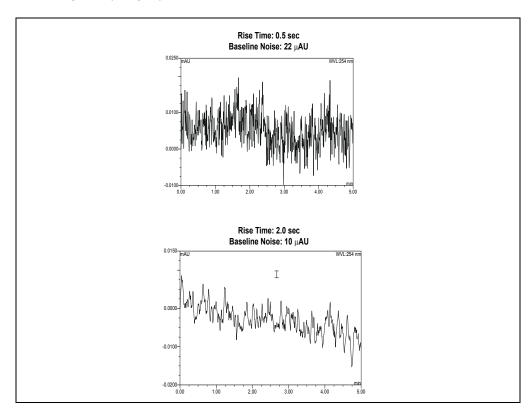


Figure 3-5. Effect of Rise Time on Baseline Noise

#### **Data Collection Rate**

The data collection rate (or *sample rate*) is the number of data points per second (Hz) at which the computer stores data from the Dionex PDA. (The detector electronics collect data at up to 20.0 Hz.)

The Dionex PDA data collection rate settings are 0.25, 0.5, 1.0, 2.0, 2.5 (default), 5.0, 10.0, or 20.0 Hz. The maximum number of data points is stored at 20.0 Hz.

- 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 (for example, 1.0 Hz).
- If any peaks of interest are less than a few seconds, select a faster data collection rate (for example, 5.0 Hz).
- If the data collection rate is too slow, the start and end points of peaks are not
  accurately determined. If the collection rate is too fast, data files may occupy
  excessive disk space and post-run analyses may require more processing time.
- A slow data collection rate with a fast rise time may result in a longer system response than indicated by the rise time. For example, a data collection rate of 0.5 Hz and a rise time of 0.5 second results in a system response longer than 2.0 seconds
- At a data collection rate of 20 Hz, the 3D sample wavelength range must be
   ≤310 nm. Regardless of the data collection rate, the reference wavelength (if set) must always be within the 3D range.
- These are the recommended pairings for the rise time and data collection rate:

Rise Time (seconds)	Data Collection Rate (Hz)
0.05	20.0
0.1	20.0
0.2	10.0
0.5	5.0
1.0	2.0
2.0	1.0
5.0	0.5

#### Sample Wavelength

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

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

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

#### Bandwidth

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

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

## Example Chromatogram: Selecting the Bandwidth

<u>Figure 3-6</u> illustrates how selecting a wide bandwidth for both the sample and reference wavelengths reduces noise by averaging over a wavelength range. The operating conditions are:

Sample wavelength = 254 nm

Reference wavelength = Off

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

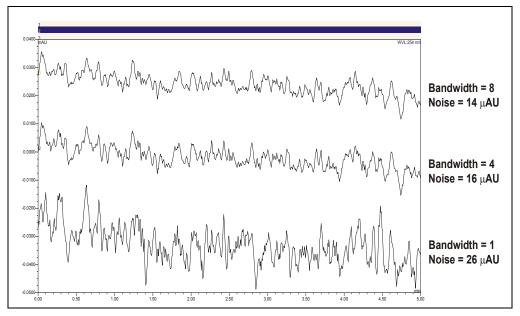


Figure 3-6. Effect of Bandwidth on Baseline Noise

### Reference Wavelength

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

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

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

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

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

Select a reference wavelength and reference bandwidth in a quiet area of the spectrum where little absorption occurs. The reference wavelength plus one-half the reference bandwidth must be within the sample wavelength range selected; if it is not, the reference wavelength will default to the longest wavelength. If the data collection rate is 20 Hz, the 3D sample wavelength range must be  $\leq$ 310 nm.

When programming a 3D channel, the reference wavelength (if set) must be within the 3D range. The reference wavelength plus one-half the reference bandwidth must be within the sample wavelength range selected; if it is not, the reference wavelength will default to the longest wavelength.

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

• To *cancel* the active reference mode, enter **Off** as the reference wavelength. The intensity reading at the time of autozero will be used as the reference intensity.

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

NOTE When you enter Off as the reference wavelength setting, the reference bandwidth is not used. The reference value is the first data point of the sample wavelength and bandwidth after autozero.

## Example Chromatogram: Selecting the Reference Wavelength

Figure 3-7 shows the results obtained from two different channels within the same run. When the reference wavelength mode is **Off** (i.e., the active reference mode is cancelled), the baseline drift is  $-120~\mu AU/hour$ . When the active reference mode is selected, the baseline drift is reduced to  $-40~\mu AU/hour$ .

The operating conditions are:

Sample wavelength = 520 nm

Bandwidth = 10 nm

Reference bandwidth = 50 nm (active reference mode only)

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

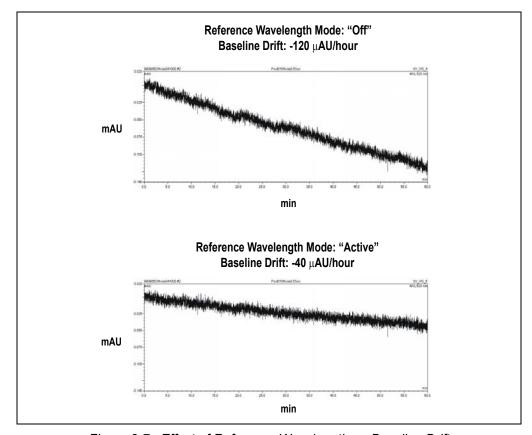


Figure 3-7. Effect of Reference Wavelength on Baseline Drift

#### Reference Bandwidth

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

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

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

- Select a reference bandwidth in an area of the spectrum where the sample does not absorb
- Select a reference bandwidth that is narrow enough not to interfere with nearby compounds. A wider bandwidth will reduce baseline noise, but may increase the chance that the sample absorbs in the reference bandwidth; this will reduce sensitivity.
- The reference wavelength plus one-half the reference bandwidth must be within the sample wavelength range selected. The reference wavelength (if set) must be within the 3D range.
- If the *active* reference mode is selected (see <u>"Reference Wavelength" on page 47</u>), select a reference wavelength and reference bandwidth in a quiet area of the spectrum where little absorption occurs. The reference wavelength plus one-half the reference bandwidth must be within the sample wavelength range selected; if it is not, the reference wavelength will default to the longest wavelength. If the data collection rate is 20 Hz, the 3D sample wavelength range must be ≤310 nm.

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

<u>Figure 3-8</u> shows the spectrum of caffeine. The operating conditions are:

Sample wavelength = 272 nm

Bandwidth = 5 nm

Reference wavelength = 325 nm

Reference bandwidth = 51 nm

These settings calculate the absorbance by the following equation:

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

Where:

 $I_K = Light intensity$ 

Offset = Absorbance offset at autozero

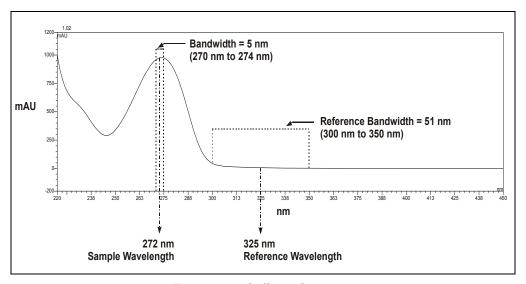


Figure 3-8. Caffeine Spectrum

#### **Bunch Width**

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

#### Step (Chromeleon 6.8 only)



It is not necessary for the user to select a step setting; the appropriate step setting is selected automatically by Chromeleon 6.8.

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

Chromeleon 6.8 automatically selects the step value that is the inverse of the data collection rate selected by the user. For example, if the data collection rate is 5.0 Hz, Chromeleon 6.8 sets the step to 0.2 second. The following table indicates the step setting automatically selected for each data collection rate.

Data Collection Rate (Hz)	Step (seconds)
0.25	4.0
0.5	2.0
1.0	1.0
2.0	0.5
2.5	0.4
5.0	0.2
10.0	0.1
20.0	0.05

#### Step Override



This section explains how to override the step setting automatically selected by Chromeleon 6.8. This information is provided for reference for advanced users only. Be aware that selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems.

In almost all cases, the step setting selected by Chromeleon 6.8 is the preferred setting. However, it is possible to override this setting by manually changing the step in the Chromeleon 6.8 program (or on the Control panel). For example, you may want to increase the step time for the 3D field or any of the five absorbance channels. For the 3D field, the

step determines the sampling rate at which the detector stores spectra. Ideally, the step is no smaller than the interval suggested for the rise time (see the table in "Data Collection Rate" on page 45). 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 (Chromeleon 6.8 only)" on page 53), 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 (Chromeleon 6.8 only)

The Average parameter operates in conjunction with the Step parameter (see <u>"Step (Chromeleon 6.8 only)" on page 52</u>).

When Average is **On**, Chromeleon 6.8 averages the data points between step intervals, reports that value, and noise is reduced. This setting is recommended for most applications.

When Average is **Off**, Chromeleon 6.8 reports the data points at the step interval. The data points between steps are skipped and noise is not reduced.



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

#### **Negative Absorbance**

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

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

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

<sup>\*</sup> These signals are saturated and will not report a further negative value.

- 1. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>), and select **UV**.
- 2. Select **NegativeAbsorptionLevel**, enter a new setting, and execute the command.
- 3. Select **Autozero** and execute the command to run the autozero routine.
- 4. Select the **LampIntensity** command to check the intensity of the deuterium lamp. The reading should be above 10 million counts for a standard or semi-preparative cell or above 3 million counts for a semi-micro cell.
  - If the reading is lower than it should be, select **UV\_Calibration Intensity** and execute the command to calibrate the intensity of the deuterium lamp. Afterward, check the lamp intensity again. If the reading is still too low, clean the flow cell (see Section 5.2) or replace the lamp (see Section 5.7).
- 5. You may continue increasing the negative absorbance level if the range is required; however, be aware that baseline noise may increase as the negative absorbance increases.

## 3.5 Shutdown

Before shutting down the Dionex PDA 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.

The Smart Shutdown feature in Chromeleon 7 or Chromeleon 6.8 can assist you in creating and running a shutdown or standby procedure.

- The shutdown process shuts down the system by automatically stopping the pump flow, turning off the detector lamps, and turning off heating in any temperature-controlled devices that are installed.
- The standby process lets you quickly restart the chromatography system from standby. A typical standby process includes steps such as reducing the pump flow rate and turning off heating in temperature-controlled devices. While the system is in standby mode, the detector lamps remain on.

Both processes maintain the equilibrated status of an instrument until the system is restarted.

## 3.6 Routine Maintenance

The following maintenance procedures may be performed by users. All other maintenance procedures for the Dionex PDA must be performed by Thermo Fisher Scientific personnel.

- Periodically check the drain tube connected to the leak tray at the bottom of the optical bench. Verify that the tubing is unclogged and that it is routed below the leak tray.
- Periodically check all tubing and fittings and replace as necessary (see <u>Section 5.1</u>). Tubing and fitting components may eventually become loose and leak, or pinched off and plugged. Look for these symptoms:

Decreasing pressure; this may indicate a leak.

A sudden increase in backpressure; this may indicate plugged tubing. (A gradual 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. Note that the semi-micro flow cell will develop approximately 1.03 MPa (150 psi) backpressure at 1.0 mL/min.

If the backpressure remains high, remove the fittings and tubing from part of the flow path, section by section; starting at the waste line, work back "upstream" until you identify the section causing the substantial backpressure. Replace tubing and fittings as necessary.

- Periodically check the flow cell tubing connections for leaks or restrictions. Replace tubing and fittings as necessary.
- Periodically repassivate stainless steel systems. This is especially important
  when operating with extreme pH levels (which will shorten the life of the
  passivation finish) or after a major disassembly. For instructions, see
  Passivation of Stainless Steel Systems and Components (Document No.
  031152).
- Monitor baseline noise. If the noise increases above an acceptable level, especially after a lamp(s) has been in service for an extended length of time, replace the lamp.

To check the lamp lifetime:

- 1. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
- 2. Select **UV**, and then select **UVLampAge** and **VisLampAge**. If a lamp has been in service for more than 2000 hours, replace it. See <u>Section 5.7</u> or <u>Section 5.8</u> for instructions on replacing the deuterium lamp or tungsten lamp, respectively.
- Check the deuterium lamp intensity approximately every 6 months:
  - 1. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
  - 2. Select **UV**, and then select **LampIntensity**. The reading should be above 10 million counts for a standard or semi-preparative cell or above 3 million counts for a semi-micro cell.
  - 3. If the lamp intensity reading is too low, calibrate the intensity: In the Command window or Commands dialog box, select **UV\_Calibration**, select **Intensity**, and execute the command.

- 4. After calibrating the lamp, check the intensity again. If the reading is still too low, follow the steps in <u>Section 4.7</u> to resolve the problem.
- Run the wavelength verification test for both the deuterium and tungsten lamps approximately every 6 months (see Section 4.14.2). If a lamp fails the test, calibrate the wavelength (see Section 4.14.2) and then run the wavelength verification test again.

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

## Dionex PDA Operator's Manual

## 4 • Troubleshooting

This chapter is a guide to troubleshooting problems that may occur while operating the Thermo Scientific Dionex ICS Series Photodiode Array Detector (Dionex PDA):

- Section 4.1 describes error messages and how to troubleshoot them.
- <u>Section 4.2</u> through <u>Section 4.14</u> describe routine operating problems and how to resolve them.
- <u>Section 4.14.2</u> describes some of the diagnostic features available in the Dionex PDA Moduleware and in Chromeleon chromatography software.

If you are unable to eliminate a problem, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

## 4.1 Alarms and Error Conditions

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

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

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

Dionex PDA-Related Alarms and Error Conditions	Default Severity Level	See
3DFIELD RefWavelength and/or RefBandwidth is out of range.	Error	page 62
Acquisition On for 3DFIELD rejected—no 3D Data Acquisition license found.	Abort	page 63
Autozero is already in progress.	Error	page 64
Calibration is running.	Abort	page 64
Cannot change 3DFIELD parameters while data acquisition is running.	Warning	page 64
Cannot set manual parameters for the required operation because the DSP is not ready.	Abort	page 64
Can't execute calibration command during data acquisition.	Error	page 65
Can't execute diagnostic command during data acquisition.	Error	page 65
Can't execute x command during data acquisition.	Error	page 65
Command not executed because an Autozero command is already running.	Warning	page 65
Data is unavailable.	Abort	page 65
Diagnostic is running.	Abort	page 66
DSP communication failure.	Abort	page 66
Filter wheel sensor could not detect blocked position.	Warning	page 66
Filter wheel sensor could not detect open position.	Warning	page 66
Invalid rise time. Valid rise times at 20 Hz: 0.05, 0.1, 0.2, 0.5, 1.0, and 2.0.	Abort	page 67
Invalid step for the 3DFIELD. Valid steps are 0.05, 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.	Error	page 67
Leak sensor error.	Warning	page 68
Not enough light to perform operation.	Warning	page 68
Over temperature error.	Abort	page 68
PDA is running a calibration or diagnostic function. Not ready to accept this command.	Error	<u>page 69</u>

Dionex PDA-Related Alarms and Error Conditions	Default Severity Level	See
The minimum wavelength must be less than the maximum wavelength. Min wavelength = $x$ ; max wavelength = $x$ .	Error	page 69
The range of wavelength bunching is 1 to 25 nm.	Error	page 69
The range of wavelengths is outside the 190 to 800 nm limit. Wavelength = $x$ ; Bandwidth = $x$ .	Error	page 69
UV lamp control circuitry error while lamp is off.	Abort	page 72
UV lamp error or control circuitry error while lamp is on.	Abort	page 70
UV lamp error.	Abort	page 70
UV lamp error while attempting to turn off.	Abort	page 70
UV lamp error while attempting to turn on.	Abort	page 70
UV lamp failed to turn on for the required operation.	Abort	page 70
Visible lamp control circuitry error while lamp is off.	Abort	page 72
Visible lamp error or control circuitry error while lamp is on.	Abort	page 70
Visible lamp error.	Abort	page 70
Visible lamp error while attempting to turn off.	Abort	page 72
Visible lamp error while attempting to turn on.	Abort	page 70
When collecting at 20 Hz, the spectrum range cannot be more than 310 nm. Please lower the data rate or narrow the spectrum range.	Abort	page 72

## Δ

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

#### To troubleshoot:

For all data collection rates other than 20 Hz, select parameters that meet the following criteria:

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

#### Where:

 $\lambda$  = Reference wavelength

BW = Reference bandwidth

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

Wavelength = 190 to 800 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

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

## The following parameters are NOT VALID:

Wavelength = 190 to 600 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

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

When the data collection rate is 20 Hz, select parameters that meet the following criteria:

$$Minimum \ \lambda \leq \lambda \ \pm \frac{BW}{2} \leq Maximum \ \lambda$$

Maximum  $\lambda$  –Minimum  $\lambda \leq 310$  nm

#### Where:

 $\lambda$  = Reference wavelength

BW = Reference bandwidth

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

Wavelength = 190 to 500 nm

Reference wavelength = 450 nm

Reference bandwidth = 50 nm

These parameters are valid because 190 to 500 nm is a range of  $\leq$  310 nm and 450 nm + 25 nm (50% of 50 nm) = 475 nm, which is less than 500 nm.

## The following parameters are NOT VALID:

Wavelength = 190 to 600 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are invalid because 190 to 600 nm is a range of more than 310 nm; also, 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is greater than 600 nm



# Acquisition On for 3DFIELD rejected—no 3D Data Acquisition license found.

#### To troubleshoot:

Chromeleon cannot perform digital data acquisition of a 3D data field unless the software includes the 3D Data Acquisition license. Contact Thermo Fisher Scientific if you want to order this option.



### Autozero is already in progress.

#### To troubleshoot:

This message appears if you select **Autozero** (in the Chromeleon 7 Command window or the Chromeleon 6.8 Commands dialog box) when the autozero routine is already in progress. Allow the autozero routine to continue running until completion.



### Calibration is running.

#### To troubleshoot:

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

## ①

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

#### To troubleshoot:

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



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

#### To troubleshoot:

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

- (1) Can't execute calibration command during data acquisition.
- ▲ Can't execute diagnostic command during data acquisition.
- ▲ Can't execute x command during data acquisition.

#### To troubleshoot:

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

Command not executed because an Autozero command is already running.

#### To troubleshoot:

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

Data is unavailable.

#### To troubleshoot:

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

# Diagnostic is running.

#### To troubleshoot:

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

**DSP** communication failure.

#### To troubleshoot:

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

- Filter wheel sensor could not detect blocked position.
- filter wheel sensor could not detect open position.

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

#### To troubleshoot:

- 1. Turn off the Dionex PDA power for 30 seconds, and then turn it on again.
- 2. If the error message appears again, check the deuterium lamp intensity:
  - a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).

- b. Select **UV**, and then select **LampIntensity**. The intensity reading should be above 10 million counts for a standard or semi-preparative cell *or* above 3 million counts for a semi-micro cell. If the reading is acceptable, resume normal operation. If the reading is too low, see <u>Section 4.7</u>.
- 3. If the error message appears again, there may be a faulty connection to the filter wheel sensor. Contact Thermo Fisher Scientific for assistance.

NOTE The Dionex PDA electronics cannot be serviced by users. All repairs of the electronics components must be performed by Thermo Fisher Scientific personnel.



Invalid rise time. Valid rise times at 20 Hz: 0.05, 0.1, 0.2, 0.5, 1.0, and 2.0.

#### To troubleshoot:

This message appears if you enter a rise time setting that is invalid for a data collection rate of 20 Hz. Select a valid rise time.



Invalid step for the 3DFIELD. Valid steps are 0.05, 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.

#### To troubleshoot:

This message appears if you enter an invalid step setting. Chromeleon 7 or Chromeleon 6.8 automatically selects the step value that is the inverse of the data collection rate (see "Step (Chromeleon 6.8 only)" on page 52). In future, accept the value selected by the software.



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

# ①

#### Leak sensor error.

#### To troubleshoot:

Locate the source of the leak and tighten (or replace) the liquid line connection. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Thermo Scientific Reference Library DVD (P/N 053891).

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

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

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

# Not enough light to perform operation.

#### To troubleshoot:

Follow the troubleshooting steps in <u>Section 4.7</u>.

# Over temperature error.

#### To troubleshoot:

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

NOTE Do not attempt to service the Dionex PDA electronics components. All repairs of the electronics components must be performed by Thermo Fisher Scientific personnel.



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

#### To troubleshoot:

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



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

#### To troubleshoot:

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



The range of wavelength bunching is 1 to 25 nm.

#### To troubleshoot:

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



The range of wavelengths is outside the 190 to 800 nm limit. Wavelength = x; Bandwidth = x.

#### To troubleshoot:

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

- **UV** lamp error.
- UV lamp error or control circuitry error while lamp is on.
- UV lamp failed to turn on for the required operation.
- UV lamp error while attempting to turn on.
- Visible lamp error.
- Visible lamp error or control circuitry error while lamp is on.
- Visible lamp error while attempting to turn on.
- Visible lamp failed to turn on for the required operation.

#### To troubleshoot:

- Check that the lamp referred to in the error message is turned on. When a lamp is on, the corresponding LED (DEUTERIUM UV or TUNGSTEN VIS) is lighted. If necessary, turn on the lamp from Chromeleon or the TTL input.
- 2. If you recently installed a new deuterium lamp, check that the UV lamp connector wires are routed through the wire guide in the optical bench (see <u>Figure 5-3</u>) and are not pinched under the lamp cover.
- 3. Check the number of hours the lamp has been in service:
  - Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>), and then select **UV**.
  - If the error message relates to the deuterium lamp, execute the **UVLampAge** command.

If the deuterium lamp has been in operation for *more* than 2000 hours, replace it (see Section 5.7). If the error message appears again, contact Thermo Fisher Scientific for assistance.

If the deuterium lamp has been in operation for *less* than 2000 hours, turn off the lamp for at least 3 minutes and then turn it on again. If the error message reappears, replace the lamp (see Section 5.7). If the error message does *not* appear again, run a manual data acquisition for about 30 minutes. If you notice a shift in the absorbance signal during this time, replace the lamp.

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

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

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

- UV lamp control circuitry error while lamp is off.
- UV lamp error while attempting to turn off.
- visible lamp control circuitry error while lamp is off.
- Visible lamp error while attempting to turn off.

#### To troubleshoot:

The lamp power supply may have failed or the CPU board may have failed. Contact Thermo Fisher Scientific for assistance.

NOTE The Dionex PDA electronics cannot be serviced by users.
All repairs of the electronics components must be performed by Thermo Fisher Scientific personnel.

When collecting at 20 Hz, the spectrum range cannot be more than 310 nm. Please lower the data rate or narrow the spectrum range.

#### To troubleshoot:

Reduce the data collection rate to less than 20 Hz or select a wavelength range that is  $\leq 310$  nm.

# 4.2 ALARM LED Is Lighted

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

After fixing the leak and drying the sensor, clear the **ALARM** LED.

- In the Chromeleon 7 Command window (see <u>page 31</u>): Click **UV**, and then execute the **ClearAlarmLED** command.
- In Chromeleon 6.8, click the **Clear Alarm LED** button on the Control panel.

-or-

In the Commands dialog box (see <u>page 32</u>), click **UV** and then execute the **ClearAlarmLED** command

# 4.3 Lamp Does Not Light/Lamp LED Is Flashing Rapidly

# • Lamp default setting is not in effect

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

To restore the default setting for a lamp:

- 1. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
- 2. Select **UV**, and then execute the required command:
  - UVLampPowerOnSetting (for the deuterium lamp)
  - VisLampPowerOnSetting (for the tungsten lamp)

# • Lamp is old or burned out

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

# 4.4 No Detector Response

#### Detector power not on

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

Check that the main power switch on the rear panel is turned on.

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

#### Lamp not turned on

Turn on the lamp from Chromeleon or the TTL input. When a lamp is on, the corresponding LED (**DEUTERIUM – UV** or **TUNGSTEN – VIS**) is lighted.

# • Lamp LED flashing rapidly

The corresponding lamp failed to power on properly. Open the Chromeleon 7 Command window (see <u>page 31</u>) or Chromeleon 6.8 Commands dialog box (see <u>page 32</u>), and select UV. Check that the required command (UVLampPowerOnSetting or VisLampPowerOnSetting) is On.

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

# • Detector and/or ePanel not connected to instrument (Chromeleon 7); detector and/or Control panel not connected to timebase (Chromeleon 6.8)

For Chromeleon 7: In the Instrument Configuration program, add the Dionex PDA device driver to an instrument.

For Chromeleon 6.8: In the Server Configuration program, add the Dionex PDA device driver to a timebase.

# • Windows operating system does not recognize Dionex PDA

When the computer is turned on, Windows scans the network for unknown devices. If the Dionex PDA power is off, the detector cannot be identified. If you connect the Dionex PDA to the PC before installing Chromeleon, the detector cannot be identified. See <u>Section 4.14</u> for instructions on how to resolve this.

# 4.5 Noisy Baseline

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

Prepare all mobile phases and reagents with spectro-grade solvents, reagent-grade chemicals, and ASTM Type I (or better) filtered, deionized water that meets the specifications in Table B-1.

# Leaking fittings

Locate the source of the leak and tighten (or replace) the liquid line connection. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Thermo Scientific Reference Library DVD (P/N 053891).

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

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

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

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

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

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

# • Plot scale exaggerates baseline noise

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

#### Air bubbles in flow cell

Remove the trapped air (see Section 5.3).

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

 Degas mobile phases and post-column reagents by vacuum degassing or sparging with helium.

• Connect a backpressure line to the cell (see <u>Section B.3</u>). This increases the backpressure on the cell, thereby shrinking bubbles and allowing them to pass more easily through the cell.

#### • Contaminants in flow cell

Clean the cell (see Section 5.2).

### Incorrect lamp selected

If a UV wavelength is selected, make sure the deuterium lamp is on. If a visible wavelength is selected, make sure the tungsten lamp is on. When a lamp is on, the corresponding LED (**DEUTERIUM – UV** or **TUNGSTEN – VIS**) is lighted.

#### • Lamp needs replacement

The light output of the lamp decreases over time. The lower light output may increase noise above an acceptable level, especially if the lamp has been in service for more than 2000 hours. If this occurs, replace the lamp. See Section 5.7 or Section 5.8 for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

# • Lamp incorrectly installed

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

# • Inappropriate reference wavelength

Select a reference wavelength in a quiet area of the spectrum where little absorption occurs. Thermo Fisher Scientific recommends selecting a reference wavelength below 380 nm for UV applications and above 600 nm for visible applications. For more information, see "Reference Wavelength" on page 47.

#### Bandwidth is too narrow

Select a wider bandwidth and reference bandwidth, if appropriate for the application. For guidelines when selecting the sample and reference bandwidths, see "Bandwidth" on page 46 and "Reference Bandwidth" on page 50.

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

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

# • Crimped or plugged tubing

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

# • Flow cell installed incorrectly

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

# • Inappropriate rise time

In general, the rise time should be approximately 25% of the peak width at half-height of the narrowest peak of interest. See <u>page 44</u> for more information.

# Detector exposed to high vibration

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

# • Filter paddle has malfunctioned

If you have recently autozeroed the detector, check the Chromeleon Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see <a href="mailto:page-66">page-66</a> for instructions.

# • Pump needs priming and/or maintenance

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

# • Negative absorbance level is too high

Baseline noise may increase as the negative absorbance increases. If the noise is unacceptable, reduce the negative absorbance level (see page 53).

# 4.6 Drifting Baseline

# Fluctuations in ambient temperature

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

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

Select the active reference mode by entering a reference wavelength (see page 47).

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

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

# Flow cell heat exchanger not in place

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

# Lamp not stabilized or burned in

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

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

# Leaking flow cell

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

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

#### Absorbance in the reference wavelength

Sample absorbance in the selected reference wavelength may cause excessive baseline drift. Select a reference wavelength and reference bandwidth in an area of the spectrum where little absorption occurs (see "Reference Bandwidth" on page 51).

Thermo Fisher Scientific recommends selecting a reference wavelength (i.e., active reference mode) for gradient analyses and turning off the reference wavelength for isocratic analyses in a laboratory with a stable ambient temperature (see "Reference Wavelength" on page 48).

# 4.7 Deuterium Lamp Intensity Reading Too Low

# Light signal is not reaching the data processor

Check that at least one lamp is turned on. (When a lamp is on, the corresponding LED (**DEUTERIUM – UV** or **TUNGSTEN – VIS**) is lighted.) If necessary, turn on one of the lamps from Chromeleon or the TTL input.

# • Low light throughput

- 1. Remove the cell from the optical bench (see <u>Section 5.4</u>).
- 2. Check the deuterium lamp intensity:
  - a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
  - b. Select UV, and then select LampIntensity.

If the lamp intensity reading is above 10 million counts for a standard or semi-preparative cell *or* above 3 million counts for a semi-micro cell, follow these steps:

- a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
- b. Select UV, and then select UV\_Calibration.
- c. Execute the **Intensity** command to calibrate the lamp intensity.
- d. Execute the **Autozero** command to run the autozero routine.
- e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in Section 5.2.

- f. Reinstall the cell in the optical bench.
- g. Autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Thermo Fisher Scientific for assistance

If the lamp intensity reading is below 10 million counts for a standard or semi-preparative cell *or* below 3 million counts for a semi-micro cell, follow these steps:

- a. Check that the flange of the deuterium lamp is aligned with the two locating pins in the optical bench (see Section 5.3). If the flange is aligned, the lamp is defective and should be replaced (see Section 5.7).
- b. If the error message appears again, contact Thermo Fisher Scientific for assistance.

#### Filter paddle has malfunctioned

Check the Chromeleon Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see page 66 for instructions.

# • Failure of an electronics component

Turn off the Dionex PDA power and contact Thermo Fisher Scientific for assistance

NOTE Do not attempt to service the Dionex PDA electronics components. All repairs of the electronics components must be performed by Thermo Fisher Scientific personnel.

# 4.8 Wavelength Calibration Fails

# Deuterium lamp may be defective

If the **DEUTERIUM – UV** LED is flashing rapidly—or if the LED fails to light—replace the deuterium lamp (see <u>Section 5.7</u>).

If the **DEUTERIUM – UV** LED is lighted but is not flashing, open the Dionex PDA Wellness panel and run the wavelength calibration and the wavelength verification test (see Section 4.14.2). If the Dionex PDA fails these procedures, remove the flow cell from the detector and repeat both

procedures. If the detector fails the wavelength calibration again, contact Thermo Fisher Scientific for assistance.

# Deuterium lamp intensity out of calibration

- 1. Remove the cell from the optical bench (see Section 5.4).
- 2. Check the deuterium lamp intensity:
  - a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
  - b. Select UV, and then select LampIntensity.

If the lamp intensity reading is above 10 million counts for a standard or semi-preparative cell *or* above 3 million counts for a semi-micro cell, follow these steps:

- a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
- b. Select UV, and then select UV Calibration.
- c. Execute the **Intensity** command to calibrate the lamp intensity.
- d. Execute the **Autozero** command to run the autozero routine.
- e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in Section 5.2.
- f. Reinstall the cell in the optical bench.
- g. Autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Thermo Fisher Scientific for assistance.

If the lamp intensity reading is below 10 million counts for a standard or semi-preparative cell *or* below 3 million counts for a semi-micro cell, follow these steps:

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

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

Flush the cell with the mobile phase for the application.

#### Cell not clear

Clean the cell (see Section 5.2).

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

# 4.9 Wavelength Verification Fails

# Detector wavelength requires recalibration

Open the Dionex PDA Wellness panel and run the wavelength calibration procedure (see Section 4.14.2).

# • No solvent flow through cell

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

# • Lamp(s) needs replacement

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

- 1. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
- Select UV, and then select UVLampAge and VisLampAge. If a lamp has been in service for more than 2000 hours, replace it. See <u>Section 5.7</u> or <u>Section 5.8</u> 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.

# • Filter paddle position error

Check the Chromeleon Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see <a href="mailto:page 66">page 66</a> for instructions.

# 4.10 No Spectra Collected

#### Spectral range was not programmed

In Chromeleon 7, select a spectral range in the instrument method or on the Dionex PDA ePanel.

In Chromeleon 6.8, select a spectral range in the program or on the Dionex PDA Control panel.

# • Detector and instrument method/program (or ePanel/Control panel) connected to different timebases

In Chromeleon 7, add the detector to the same instrument as the instrument method or the Dionex PDA ePanel.

In Chromeleon 6.8, add the detector to the same timebase as the program or the Dionex PDA Control panel.

# 4.11 Low Spectral Resolution

#### • Bunch width too wide

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

#### Bandwidth too wide

Select a bandwidth that includes only the absorbing wavelength.

#### • Incorrect reference wavelength

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

# Wavelength range too narrow

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

#### Reference bandwidth too wide

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

# 4.12 Peaks Too Large or Small

#### Analog output-related problem

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

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

# • System-level problem

Verify that the sample volume or concentration is appropriate.

Verify that the rise time (see <u>page 45</u>) and data collection rate (see <u>page 46</u>) are appropriate to capture the narrowest peak.

Prepare fresh post-column reagent.

Verify that the reagent flow rate is correct.

#### Reference bandwidth too wide

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

# 4.13 Poor Peak Shape

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

Dilute the standard.

Install a sample loop with a smaller volume.

#### Concentration of standard is too low

Increase the standard concentration

# • Inappropriate mobile phase

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

# • Incorrect sample wavelength selected

If little is known about the analytes in the sample, collect spectra over the full wavelength range (using both lamps). For guidelines to follow when selecting the sample wavelength, see <u>page 47</u>.

# • Incorrect reference wavelength selected

Select a different reference wavelength. For guidelines to follow when selecting the reference wavelength, see <u>page 48</u>.

#### • Bandwidth too wide

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

#### Reference bandwidth too wide

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

#### Cell flow is reversed.

Check that the flow cell inlet tubing is routed through the heat exchanger (see Figure 2-5) and is correctly connected to the inside front panel (see Figure 5-1). Check that solvent flow is directed to the cell inlet and out the cell outlet.

#### • Semi-micro cell flow rate selected when standard cell is installed

For operation at less than 1.0 mL/min, install a semi-micro flow cell.

# 4.14 Faulty USB Communication

#### Dionex PDA not recognized by Windows operating system

Thermo Fisher Scientific strongly recommends installing Chromeleon *before* connecting the Dionex PDA to the computer. When the chromatography software is installed first, USB driver information is loaded automatically. If Windows fails to detect the Dionex PDA, refer to the following table for corrective action.

	lem

#### Action

Windows Vista fails to detect the Dionex PDA and launches a wizard, instead This indicates that you connected the Dionex PDA to the computer and turned on the power for the first time before you installed Chromeleon. To resolve the problem:

- Click **Cancel** to exit the wizard.
- Turn off the detector and unplug the USB cable from the computer.
- Install Chromeleon.
- Reconnect the USB cable to the computer and turn on the power to the detector.

Windows Vista will now detect the Dionex PDA and automatically install the USB software for it.

Windows XP fails to detect the Dionex PDA and a message box asks for a USB configuration file (CmWdmUsb.inf) This indicates that you connected the Dionex PDA to the computer and turned on the power for the first time before you installed Chromeleon. To resolve the problem:

- Click Cancel in the Windows message box.
- Turn off the detector power and unplug the USB cable from the computer.
- Install Chromeleon.
- Reconnect the USB cable to the computer and turn on the power to the detector.

Windows will now automatically detect the Dionex PDA and launch the Found New Hardware Wizard.

# 4.14.1 Moduleware Run-Time Diagnostics

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

• Check the Audit Trail displayed on the Dionex PDA ePanel or Control panel.

-or-

• Retrieve the Audit Trail from a sequence by selecting the Audit Trail folder in the timebase and the file for that date

# 4.14.2 Software Diagnostics and Calibration

Chromeleon includes comprehensive diagnostics for the Dionex PDA. This section describes some of the most frequently used diagnostic procedures. For more information, refer to the Chromeleon Help.

# Lamp age

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

To reset the lamp age:

- a. After installing a new lamp, open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see page 32).
- Select UV, and then select the UVLampAge command (for the deuterium lamp) or VisLampAge command (for the tungsten lamp).
- c. Reset the number to zero.
- d. Execute the command.

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

# • Wavelength calibration

**Purpose:** Improves wavelength accuracy in the UV range by reassigning new wavelengths to each pixel, based on the emission spectrum from the deuterium lamp. The detector will perform the wavelength calibration routine and upload the result (pass or fail) to Chromeleon. The results of both the initial calibration and the last calibration are stored in the calibration log. You can view the results at any time.

#### In Chromeleon 7:

- a. Verify the following conditions: no solvent is flowing through the cell, the background absorbance is low, and there are no bubbles in the light path.
- b. Open the Command window (see <u>page 39</u>), select **UV**, and execute the **WavelengthCalibration** command.

#### In Chromeleon 6.8:

- a. Verify the following conditions: no solvent is flowing through the cell, the background absorbance is low, and there are no bubbles in the light path.
- b. To open the Wellness panel, click the **Wellness** button on the Dionex PDA Control panel.
- c. Click the wavelength script button under Calibration.

#### Wavelength verification

**Purpose:** Tests the accuracy of the selected wavelength. The accuracy is verified for the following wavelengths: 360.9, 418.8, and 536.5. The results of the test (pass or fail) are logged in the Audit Trail. The result of the last wavelength verification test is stored in the calibration log and can be retrieved at any time.

NOTE Wavelength verification can take up to 2 minutes. During this time, data acquisition is not possible.

#### In Chromeleon 7:

a. Open the Command window (see <u>page 39</u>), click **UV**, and execute the **WavelengthVerTest** command.

# In Chromeleon 6.8:

- a. To open the Wellness panel, click the **Wellness** button on the Dionex PDA Control panel.
- b. Click the **wavelength verification** script button under **Diagnostics**.

# Dionex PDA Operator's Manual

This chapter describes routine service procedures for the Thermo Scientific Dionex ICS Series Photodiode Array Detector (Dionex PDA) that users may perform. All procedures not described here, including electronics-related repair procedures, must be performed by Thermo Fisher Scientific personnel. For assistance, contact Technical Support for Dionex products. In the U.S. and Canada, call 1-800-346-6390. Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.



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

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



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

# 5.1 Liquid Leaks

The Dionex PDA is plumbed with the parts listed below. For tightening requirements, refer to *Installation of Dionex Liquid Line Fittings* (Document No. 031432). The manual is on the Thermo Scientific Reference Library DVD (P/N 053891).

Note that the tubing connected to the heat exchangers (standard and semi-micro cells only), as well as the semi-preparative cell inlet and outlet tubing, cannot be replaced. If a leak (at the cell inlet, for example) cannot be eliminated by tightening the fitting, you must replace the entire cell (see Section 5.6).

Table 5-2. Dionex PDA with Standard Cell

Component	PEEK Cell	SST Cell
Cell inlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A
Cell outlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A

Table 5-2. Dionex PDA with Standard Cell (Continued)

Component	PEEK Cell	SST Cell
Cell inlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 056124
Cell outlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 051650
Reducing union fittings	P/N 055903	P/N 055902
Ferrules	P/N 043276	P/N 010262
Nuts	P/N 043275	P/N 010261

Table 5-3. Dionex PDA with Semi-Micro Cell

Component	PEEK Cell	SST Cell
Cell inlet tubing: 0.12-mm (0.005-in) ID	P/N 063897	N/A
Cell outlet tubing: 0.38-mm (0.015-in) ID	P/N 057304	N/A
Cell inlet tubing: 0.12-mm (0.005-in) ID	N/A	P/N 063903
Cell outlet tubing: 0.25-mm (0.010-in) ID	N/A	P/N 051650
Reducing union fittings	P/N 055903	P/N 055902
Ferrules	P/N 043276	P/N 010262
Nuts	P/N 043275	P/N 010261

Table 5-4. Dionex PDA with Semi-Preparative Cell

Component	PEEK Cell
Cell inlet tubing: 0.51-mm (0.020-in) ID	P/N 042855
Cell outlet tubing: 0.51-mm (0.020-in) ID	P/N 042855
Union fittings	P/N 042627
Ferrules	P/N 043276
Nuts	P/N 043275

# 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 at least 10  $\mu$ L each of deionized water, acetone, and 3 M HNO<sub>3</sub>.
- 4. Reconnect the cell liquid lines.
- 5 Flush the cell with deionized water for 20 to 30 minutes at 1.0 mL/min

# 5.3 Removing Trapped Air from the Flow Cell

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

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

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

# 5.4 Removing the Flow Cell from the Optical Bench

The flow cell must be removed from the optical bench in order to inspect the cell for leaks or obstructions, replace the cell windows (see Section 5.5), or install a new cell (see Section 5.6).

- 1. Grasp the Dionex PDA front cover by the sides and pull straight off to remove.
- 2. Disconnect the tubing connected to the flow cell inlet and outlet (see Figure 5-1).
- 3. Remove the flow cell cover from the inside front panel (see <u>Figure 2-3</u>).
- 4. **Standard or semi-micro cell**: 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. Squeeze the handle of the flow cell and pull it out of the optical bench.



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

# 5.5 Replacing the Flow Cell Windows

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

NOTE The windows in the semi-preparative flow cell cannot be replaced. If cleaning the windows does not eliminate the problems described above, order a new semi-preparative cell.

The Dionex AD25/Dionex PDA/Dionex PDA-100 Flow Cell Windows Replacement Kit (P/N 060498) contains all of the parts required to replace the fused silica windows in a standard or semi-micro cell. The kit is optional and must be ordered separately from the Dionex PDA.

- 1. Remove the flow cell from the optical bench (see <u>Section 5.4</u>) and place the cell on a clean work surface.
- 2. Disassembling the cell with bare hands may introduce contaminants into the system. Before proceeding, put on a pair of latex gloves that are lint-free, particle-free, and oil-free.

IMPORTANT

Even minute particles of dust, dirt, etc. on the flow cell parts can contaminate the cell and result in excessive baseline noise and drift.

- 3. Using the windows replacement tool (P/N 060497), remove one of the flow cell retaining nuts from the cell (see <u>Figure 5-2</u>). (It does not matter which retaining nut is removed first.)
- 4. Remove and discard the old window.
- 5. Use tweezers to carefully remove the O-ring from the groove (see <u>Figure 5-2</u>). **Do not scratch the groove.**

IMPORTANT

The following factors will prevent the new O-ring from sealing properly and will cause leaks: scratches in the groove, particles of dust or dirt in the groove, and failure to seat the new O-ring in the groove.

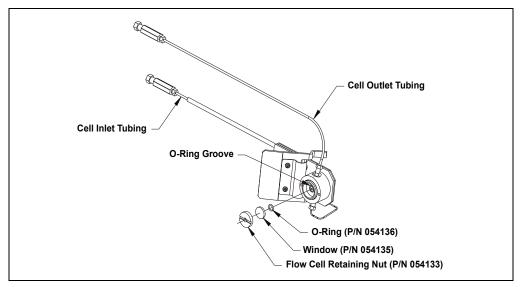


Figure 5-2. Replacing the Standard or Semi-Micro Flow Cell Windows (Light inlet side shown)

- 6. Check the groove to be sure it does not contain any dust or dirt particles, and then install a new O-ring (P/N 054136). Make sure the O-ring is seated in the groove.
- 7. Place a new window (P/N 054135) into the counterbore in the retaining nut. Screw the window into place.
- 8. Using the windows replacement tool, tighten the retaining 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 1.38 MPa (200 psi). Check for leaks around the windows. If there is any leakage, tighten the retaining nut(s) with the windows replacement tool just until the leak stops.

IMPORTANT

Do not overtighten the retaining nut, as this may fracture the window.

If tightening the retaining nut(s) stops the leakage, dry the cell thoroughly with a lint-free, optical-grade tissue and go on to <u>Step 11</u>.

If tightening the retaining nut(s) *does not* stop the leakage, remove the window and inspect the O-ring and groove for the cause of the leak:

- If necessary, clean the groove and reinstall the O-ring. Dry the cell thoroughly with a lint-free, optical-grade tissue and go on to Step 11.
- If the groove is scratched, the cell must be replaced. Contact Thermo Fisher Scientific to order a new cell
- 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 fused 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 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 cover into place.

# 5.7 Replacing the Deuterium Lamp

- 1. Stop the Chromeleon instrument controller or server.
- 2. Press the power button on the front of the Dionex PDA to turn off the power.
- 3. Turn off the main power switch on the Dionex PDA rear panel.



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



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



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



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



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



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

- 4. Grasp the detector front cover by the sides and pull straight off to remove.
- 5. **Standard or semi-micro cell**: Remove the tubing bracket from the lamp cover (see Figure 5-1).
- 6. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (see Figure 5-1).
- 7. Remove the flow cell cover from the inside front panel (see <u>Figure 5-1</u>).
- 8. 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.

9. Squeeze the clip on the UV lamp connector and disconnect it from the 6-pin bulkhead connector on the side wall (see Figure 5-3).

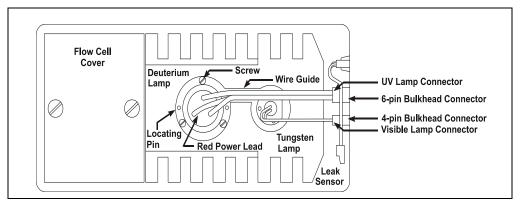


Figure 5-3. Lamp Connections

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

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

- 11. Inspect the new deuterium lamp (P/N 939016T) for fingerprints and dust. If necessary, clean the lamp with IPA (isopropyl alcohol).
- 12. Line up the flange of the new lamp with the two locating pins and three threaded holes in the optical bench (see <u>Figure 5-3</u>). When the flange is aligned, gently push the lamp into the optical bench.
- 13. 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.

14. 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).

15. Replace the lamp cover.

# IMPORTANT

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

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

# IMPORTANT

Do not pinch the tubing under the flow cell cover.

- 18. Replace the detector front cover.
- 19. Turn on the detector power.
- 20. Start the Chromeleon instrument controller or server.
- 21. Reset the lamp lifetime:
  - a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
  - b. Select UV, and then select the UVLampAge command.
  - c. Reset the lamp age to zero.
  - d. Execute the command.

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

22. Run the wavelength calibration and wavelength verification test for the new lamp (see Section 4.14.2).

## 5.8 Replacing the Tungsten Lamp

- 1. Stop the Chromeleon instrument controller or server.
- 2. Press the power button on the front of the Dionex PDA to turn off the power.
- 3. Turn off the main power switch on the Dionex PDA rear panel.



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.

- 4. Grasp the detector front cover by the sides and pull straight off to remove.
- 5. **Standard or semi-micro cell**: Remove the tubing bracket from the lamp cover (see Figure 5-1).
- 6. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (see Figure 5-1).
- 7. Remove the flow cell cover from the inside front panel (see Figure 5-1).
- 8. 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.
- 9. Squeeze the clip on the visible lamp connector and disconnect it from the 4-pin bulkhead connector on the side wall (see <u>Figure 5-3</u>).
- 10. 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.

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

12. 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.

- 13. 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 <u>Figure 5-3</u>).
- 14. Replace the lamp cover.

#### IMPORTANT

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

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

#### IMPORTANT

Do not pinch the tubing under the flow cell cover.

- 17. Replace the detector front cover.
- 18. Turn on the detector power.
- 19. Start the Chromeleon instrument controller or server.
- 20. Reset the lamp lifetime:
  - a. Open the Chromeleon 7 Command window (see <u>page 31</u>) or the Chromeleon 6.8 Commands dialog box (see <u>page 32</u>).
  - b. Select UV, and then select the VisLampAge command.
  - c. Reset the lamp age to zero.
  - d. Execute the command.

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 Dionex PDA.



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



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

- 1. Press the power button on the front of the Dionex PDA to turn off the power.
- 2. Turn off the main power switch on the Dionex PDA rear panel.
- 3. Disconnect the power cord from the Dionex PDA rear panel.
- 4. The fuse holder is part of the main power receptacle (see <u>Figure 5-4</u>). Note the recessed lock on each side of the fuse holder.

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

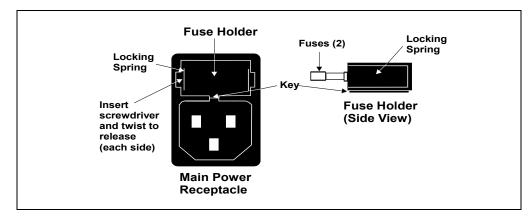


Figure 5-4. Main Power Fuse Holder

#### **Dionex PDA Operator's Manual**

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

# A • Specifications

#### A.1 Electrical

**Main Power** 85 to 265 VAC, 47 to 63 Hz (auto-sensing; no manual

adjustment required)

Typical input power: 100 W

Maximum line draw: 3.5 A at 110 VAC at power-up

Fuses Two fast-blow IEC 127 fuses (P/N 954763) rated at 4.0 A

## A.2 Physical

**Dimensions** Height: 17.4 cm (6.8 in)

Width: 44.4 cm (17.5 in) Depth: 50.3 cm (19.8 in)

Clearance required behind detector: 6 cm (2.4 in)

**Weight** 18.1 kg (40 lbs)

#### A.3 Environmental

**Operating** 4 to 40 °C (40 to 104 °F) constant temperature

**Temperature** 

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

Humidity

#### A.4 Detector

**Optical System** Single-beam, reverse-optics design with concave holographic

grating

**Light Sources** Deuterium lamp (30 W) for ultraviolet spectrum analysis

Tungsten lamp (15 W) for visible spectrum analysis Both lamps: Lifetime of 2000 hours with >50% of initial

intensity

**Photodiode Array** 1024-element photodiode array bench

**Wavelength** 190 to 800 nm

Range

**Pixel Resolution** 0.7 nm

**Wavelength** ±1 nm, self-calibration with deuterium lines, verification with

**Accuracy** built-in holmium oxide filter

**Noise with**  $\leq \pm 10 \,\mu\text{AU}$  at 254 nm, 2 second rise time, 4 nm bandwidth, and

**Standard or Semi-** flowing water at 1.0 mL/min

**Preparative Cell**  $\leq 15 \,\mu\text{AU}$  at 520 nm, 2 second rise time, 10 nm bandwidth, and

**Installed** flowing water at 1.0 mL/min

Noise with Semi-  $\leq \pm 15 \,\mu\text{AU}$  at 254 nm, 2 second rise time, 4 nm bandwidth, and

**Micro Cell** flowing water at 1.0 mL/min

**Installed** <± 15 μAU at 520 nm, 2 second rise time, 10 nm bandwidth, and

flowing water at 1.0 mL/min

**Drift** <1000 μAU/hour (after warm-up)

**Linearity** Deuterium lamp: >2 AU

Tungsten lamp: >2 AU

**Analog Outputs** Four; 0 to 3 AU, selectable, 1000 mV range

**Voltage Out** 1 V output

**Analog Output** 0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale

Range

**Control and Data** Provided by Chromeleon 7 or Chromeleon 6.8 software; the PC

**Evaluation** and the detector are connected through USB (Universal Serial

Bus)

#### A.5 Flow Cells

#### A.5.1 Standard Flow Cell

**Cell Body** PEEK or 316 stainless steel

**Volume and** PEEK cell: 13 μL volume; 10 mm (0.394 in) path length

Length

Maximum PEEK cell: 2 MPa (300 psi)
Operating Stainless steel cell: 3 MPa (500 psi)

Pressure

Chemical Compatibility

• Do not use bases stronger than 0.1 M; these solutions will etch the fused silica windows of the flow cell.

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

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

#### A.5.2 Semi-Micro Flow Cell

Cell Body PEEK or 316 stainless steel

Volume and PEEK cell: 3.1 µL volume; 9 mm (0.35 in) path length

Optical Path Stainless steel cell: 3.1  $\mu$ L volume; 9 mm (0.35 in) path length Length

Maximum PEEK cell: 2 MPa (300 psi)

Operating Stainless steel cell: 3 MPa (500 psi)

Pressure Stainless steel cell: 3 MPa (300 psi)

# Chemical Compatibility

- Do not use bases stronger than 0.1 M; these solutions will etch the fused silica windows of the flow cell.
- Do not use normal phase or chlorinated solvents with the PEEK flow cell; these solutions will damage the cell.
- Do not use low pH solutions with the stainless steel flow cell; these solutions may cause corrosion, contamination, and metal leaching.

#### A.5.3 Semi-Preparative Flow Cell

Cell Body PEEK

Volume and  $0.7 \mu L$  volume; 0.4 mm (0.02 in) path length

Optical Path Length

**Maximum** 10.34 MPa (1500 psi)

Operating Pressure

Chemical • Do not use bases stronger than 0.1 M; these solutions will etch the fused silica windows of the flow cell.

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

# A.6 Heat Exchangers

**Maximum** PEEK cell: 2 MPa (300 psi)

**Operating** Stainless steel cell: 3 MPa (500 psi)

Pressure

**Volume of** PEEK cell: 45 μL

Standard Cell Stainless steel cell: 20 µL

Heat Exchangers (including cell inlet tubing)

Volume of Semi-Micro Cell Heat Exchangers (including cell inlet tubing) PEEK cell: 5 μL

Stainless steel cell:  $5 \,\mu L$ 

# Dionex PDA Operator's Manual

#### **B.1** Facilities Requirements

- Make sure the installation site for the Thermo Scientific Dionex ICS Series Photodiode Array Detector (Dionex PDA) meets the power and environmental specifications listed in Appendix A.
- For mobile phase generation, or when manually preparing mobile phase and regenerant, use ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in Table B-1.

Contaminant	Specification
Ions-Resistivity	>18.0 (megohm-cm)
Organics-TOC	<10 ppb
Iron/Transition Metals*	<1 ppb
Pyrogens	<0.03 (Eu/mL)
Particulates $> 0.2 \mu m$	<1 (units/mL)
Colloids-Silica	<10 ppb
Bacteria	<1 (cfu/mL)
* Iron/transition metal content not specified for ASTM Type	

I water

Table B-1. ASTM Filtered, Type I Deionized Water Specifications for Ion Chromatography

- Optical detectors are sensitive to vibration. Provide a sturdy, vibration-free workbench of a height that allows convenient access to the interior of the Dionex PDA
- Allow at least 6 cm (2.4 in) behind the Dionex PDA for power connections and ventilation. **Do not block air flow to the detector.** During operation, room air must be inducted into the detector to cool the electronics and dissipate the heat generated by the lamps.

• The Dionex PDA can be positioned in many locations within a system; the options include:

Above a Dionex ICS-5000 Thermal Compartment

Below a Thermo Scientific Dionex AS-AP Autosampler

On either side of an integrated system (for example, the Dionex ICS-2100 Ion Chromatography System)

 Optical detectors are sensitive to changes in temperature and light. Protect the Dionex PDA, the columns, and all tubing connections from drafts. Do not operate the Dionex PDA unless the front cover, lamp cover, and flow cell cover are in place.

# **B.2** Unpacking

Carefully remove the Dionex PDA from the shipping container.



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

- Do not turn on the Dionex PDA unless it is at room temperature (4 to 40 °C; 40 to 104 °F). If you move the detector from a cold environment to a warm environment, wait at least 1 hour for condensation to evaporate before turning on the power.
- Unpack all items in the Dionex PDA Ship Kit (P/N 060977) and check them
  against the packing list. If there are any discrepancies, notify Thermo Fisher
  Scientific immediately.

NOTE Keep the original shipping container and all packing material. These will be needed if the detector is ever shipped or is moved to a new location.

## **B.3** Installing the Flow Cell

IMPORTANT

Do not touch the cell windows. If you touch a window, clean it with isopropyl alcohol (IPA) and a clean lens tissue.

IMPORTANT

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

IMPORTANT

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

- 1. Grasp the Dionex PDA front cover by the sides and pull straight off to remove.
- 2. Remove the flow cell cover from the inside front panel (see <u>Figure 2-3</u>).
- 3. **Standard or semi-micro cell**: Remove the tubing bracket from the lamp cover (see Figure 5-1).
- 4. Squeeze the handle of the flow cell 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.
- 5. Thread the flow cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket (see <u>Figure 5-1</u>).
- 6. 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.
- 7. **Standard or semi-micro cell:** Connect the cell inlet tubing to the reducing union fitting (P/N 055903 for a PEEK cell; P/N 055902 for a stainless steel cell) on the heat exchanger tubing (see Figure B-1).

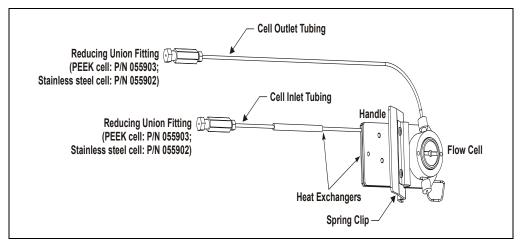


Figure B-1. Standard or Semi-Micro Flow Cell Connections

8. **Semi-preparative cell:** Connect the cell inlet tubing to a union fitting (P/N 042627) (see Figure B-2).

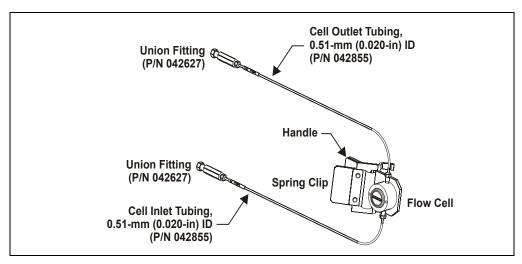


Figure B-2. Semi-Preparative Flow Cell Connections

9. Connect a length of 0.51-mm (0.020-in) ID tubing (P/N 052308) to the cell outlet connection (see <u>Figure B-1</u> or <u>Figure B-2</u>). This is the cell waste line.

- 10. Connect a backpressure line to the cell outlet. The backpressure line will provide enough restriction to generate the small amount of backpressure (0.14 to 0.34 MPa; 20 to 50 psi) required to help prevent bubbles from forming in the cell.
  - a. Locate the following items in the Dionex PDA Ship Kit (P/N 060977): 0.25-mm (0.010-in) ID tubing (P/N 052306), two split-cone 10-32 ferrule fittings (P/N 062978), and two 10-32 bolts (P/N 064980).
  - b. Cut the tubing to the required length: 1 meter for a flow rate of 1.0 mL/min, 2 meters for a flow rate of 0.5 mL/min, and so on.
  - c. Install a 10-32 ferrule fitting and bolt on each end of the tubing.
  - d. Using a union fitting (P/N 042627), connect the backpressure line to the cell waste line installed in Step 9.

#### IMPORTANT

The backpressure on the cell must not exceed 2 MPa (300 psi) for a PEEK standard or semi-micro cell, 3 MPa (500 psi) for a stainless steel standard or semi-micro cell, or 10.34 MPa (1500 psi) for a semi-preparative cell.

- 11. Thread the cell outlet tubing through the slot near the bottom of the front cover. Thread the cell inlet tubing through the slot on the right side.
- 12. Push the detector front cover into place.
- 13. Connect the cell inlet tubing to the separator column outlet.

#### **B.4** Connecting the Waste Line

Connect the waste line (P/N 055075) to the Dionex PDA rear panel (see <u>Figure B-3</u>). Place the free end of the waste line in a waste container below the level of the workbench on which the Dionex PDA 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.

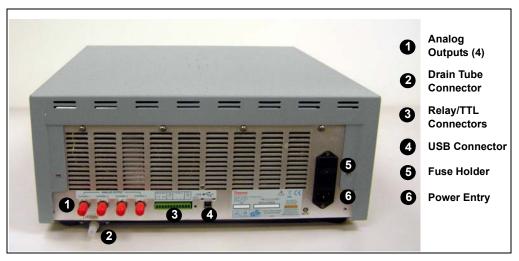


Figure B-3. Dionex PDA Rear Panel

# **B.5** Connecting the Analog Outputs (Optional)

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

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

#### B.6 Connecting the Relays/TTLs (Optional)



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



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



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



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

The Dionex PDA Ship Kit (P/N 060977) includes a 12-pin connector (P/N 923686) for relay and TTL connections (see <u>Figure B-4</u>). To attach individual wires and twisted pairs to the connectors, strip the end of the wire(s), insert into the connector, and tighten the locking screw(s) with a small screwdriver (P/N 046985).

NOTE A twisted wire assembly (P/N 043598) is available as an option.

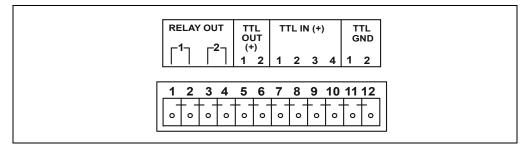


Figure B-4. Relay and TTL Connector

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

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

## **B.7** Connecting to the Chromeleon PC

#### B.7.1 Before You Begin

Verify that the following tasks have been completed:

- Chromeleon 7 or Chromeleon 6.8 is installed on the computer.
- The Chromeleon 7 or Chromeleon 6.8 license is installed.

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

If installation is required, follow the instructions in the appropriate installation guide. The guides are provided on the Thermo Scientific Reference Library DVD (P/N 053891).

- For Chromeleon 7, refer to *Chromeleon 7 Installation Guide*.
- For Chromeleon 6.8, refer to *Installing the Chromeleon Chromatography Management System with a Dionex Ion Chromatograph* (Document No. 031883).

#### **B.7.2** Chromeleon 7: Starting the Instrument Controller Service

To start the Instrument Controller Service, right-click the Chromeleon tray icon (which is crossed out in red ) and click **Start Chromeleon Instrument Controller**. The icon changes to gold to indicate that the Instrument Controller Service is starting. When the Instrument Controller Service is running (idle), the icon changes to gray .

If the Chromeleon tray icon is not on the taskbar, click **Start > All Programs > Chromeleon 7 > Services Manager** to open the Services
Manager and click **Start Instrument Controller**.

#### B.7.3 Chromeleon 6.8: Starting the Server

To start the Chromeleon server, right-click the Chromeleon Server Monitor tray icon (which is crossed out in red (and click Start Server). The icon changes to gold (and indicate that the Chromeleon server is starting. When the server is running (idle), the icon is gray (and indicate that the Chromeleon server) is starting.

If the Server Monitor icon is not on the taskbar, click **Start** on the taskbar and select **All Programs > Chromeleon > Server Monitor**. Click **Start** to start the server.

#### **B.7.4** Connecting the USB Cable

Select one of the following methods to connect the Dionex PDA to the Chromeleon computer:

- Connect the detector directly to a USB port on the PC.
- Connect the detector to the internal USB hub of another module in the system, and connect the other module directly to the PC.
- Connect the detector to an external USB hub (P/N 060392).

#### To connect the Dionex PDA directly to the PC:

- 1. Plug the "A" connector of the USB cable (P/N 063246) into the USB port on the computer (see Figure B-5).
- 2. Plug the "B" connector of the USB cable into a USB receptacle on the Dionex PDA rear panel.

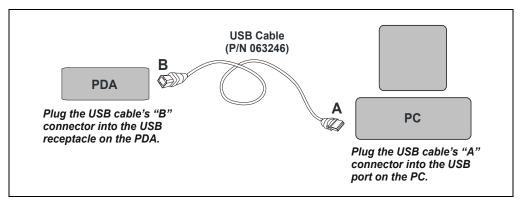


Figure B-5. Dionex PDA Connected Directly to the Chromeleon PC

To connect the Dionex PDA to the internal USB hub of another module:

NOTE If you are installing a Dionex PDA with a Dionex ICS-5000 or Dionex ICS-3000 Ion Chromatography System, refer to the system installation instructions for information about USB compatibility issues with these systems. The manuals are available on the Thermo Scientific Reference Library DVD (P/N 053891).

#### IMPORTANT

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

- 1. Plug the "A" connector of the USB cable (P/N 063246) into the USB port on the module with the internal hub (see <u>Figure B-6</u>).
- 2. Plug the "B" connector of the USB cable into the USB receptacle on the Dionex PDA rear panel.

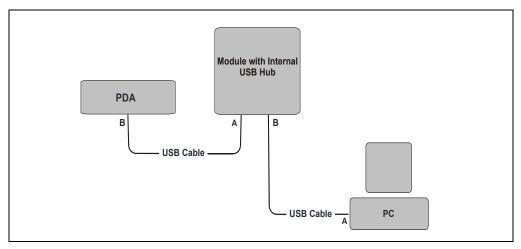


Figure B-6. Example USB Connections:

Dionex PDA Connected to a Module with an Internal USB Hub

#### To connect the Dionex PDA to an external USB hub:

The Dionex PDA Ship Kit (P/N 060977) contains one USB cable (P/N 063246). Contact Thermo Fisher Scientific to order the external USB hub (P/N 060392) and additional USB cable (P/N 063246) required for this configuration.

#### IMPORTANT

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

#### IMPORTANT

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

- 1. Plug the "A" connector of a USB cable (P/N 063246) into a port on the external USB hub (P/N 060392) (see Figure B-7).
- 2. Plug the "B" connector of the cable into the USB receptacle on the Dionex PDA rear panel.
- 3. Plug the "A" connector of the second USB cable (P/N 063246) into a USB port on the computer on which Chromeleon is installed.
- 4. Plug the "B" connector of the USB cable into a port on the external USB hub (see Figure B-7).

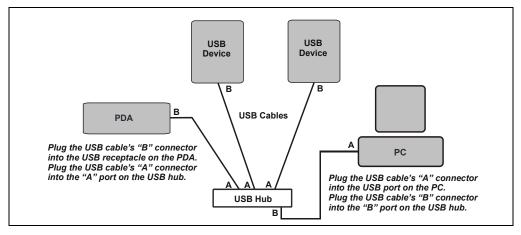


Figure B-7. Example USB Connections: Modules Connected to an External Hub

#### **B.8** Connecting the Power Cord

- 1. Verify that the main power switch on the rear panel of the Dionex PDA is turned off. (The main power switch may be turned on accidentally when the detector is unpacked.)
- 2. Connect a modular power cord (IEC 320 C13) from the Dionex PDA main power receptacle (see <u>Figure B-3</u>) to a grounded, single-phase power source. The power supply is auto-sensing: No adjustment is needed to select the line voltage.



SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the Dionex PDA or connect it to AC power 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 Dionex PDA and is easily accessible.



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



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



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



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



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

## **B.9 Installing the USB Device Drivers**

- 1. Turn on the computer power.
- Log onto Windows Vista or Windows XP as an administrator.
   For a network computer, log on as a user with local computer administrator privileges.

IMPORTANT

Before turning on the Dionex PDA power, verify that Chromeleon is installed on the PC and the license code was entered (see <u>Section B.7.1</u>). If the chromatography software is not installed first, Windows will be unable to identify the detector.

- 3. Turn on the main power switch on the rear panel of the Dionex PDA.
  - NOTE Always leave the main power switch on unless instructed to turn it off (for example, before performing a service procedure). Use the power button on the front of the Dionex PDA for routine on/off control.
- 4. **For Windows Vista:** Windows Vista detects the Dionex PDA and performs the USB driver installation.
  - a. When the driver installation is complete, the Chromeleon USB Auto Configuration Wizard automatically appears. Accept the default option for Chromeleon 7 (Create a new instrument) or Chromeleon 6.8 (Create a new timebase) and click OK. This opens the Auto Configuration dialog box. Leave the dialog box open.
  - b. Go on to Section B.10.
- 5. **For Windows XP:** Windows XP detects the Dionex PDA and launches the Found New Hardware Wizard. Use the wizard to install the Dionex PDA, as described below
  - a. If asked whether Windows can connect to Windows Update to search for software, select **No, not this time**.
  - Accept the default option (Install the software automatically) and click Next>.
  - c. When the hardware wizard reports that the software for the detector has been installed, click **Finish**.

- d. The Chromeleon USB Auto Configuration Wizard automatically appears. Accept the default option for Chromeleon 7 (**Create a new instrument**) or Chromeleon 6.8 (**Create a new timebase**) and click **OK**. This opens the Auto Configuration dialog box. Leave the dialog box open.
- e. Go on to Section B.10.

NOTE If Windows fails to detect the Dionex PDA, refer to Section 4.14 for corrective action.

# **B.10 Configuring the Dionex PDA in Chromeleon**

The Chromeleon USB Auto Configuration Wizard appears automatically when a USB device that is not assigned to a Chromeleon 7 instrument or a Chromeleon 6.8 timebase is powered up. You can use the wizard to create a new instrument or timebase for the Dionex PDA (see Section B.10.1).

If you prefer, click **Cancel** to close the wizard, and then manually create an instrument or timebase:

- For instructions on how to manually create an instrument, refer to the Chromeleon 7 Help or *Chromeleon 7 Installation Guide*.
- For instructions on how to manually create a timebase, refer to the Chromeleon 6.8 Help or to *Installing Chromeleon with a Dionex Ion Chromatograph* (Document No. 031883).

The software installation manuals are provided on the Thermo Scientific Reference Library DVD (P/N 053891)

#### **B.10.1 Using the USB Auto Configuration Wizard**

- Review the names displayed under Unassigned USB Devices
   Detected. Clear the check mark before the name of all devices other
   than the Dionex PDA.
- 2. Under **Mode**, select a predefined configuration template for the new instrument or timebase.
- 3. (Optional) Select the Launch Instrument Controller Service upon closing this wizard check box (Chromeleon 7) or the Launch Server Configuration upon closing this wizard check box (Chromeleon 6.8). The check box is disabled if the program is already running.

- 4. (Optional) Select the Customize instrument names (For advanced users only) check box (Chromeleon 7) or the Customize timebase names (For advanced users only) check box (Chromeleon 6.8).
- 5. Click **Accept** to create a new instrument or timebase.
- 6. If you selected the **Customize instrument...** or **Customize timebase...** option, the Change Instrument Names or Change Timebase Names dialog box appears now.

Enter a *new and unique* name and click the **Use Custom Names** button. (To cancel, click the **Use Default Names** button.)

- NOTE Selecting an instrument or timebase name already in use on another instrument controller or server can cause unpredictable behavior.
- 7. If you selected the **Launch Instrument Controller Service...** or **Launch Server Configuration...** option, the program automatically starts running when you close the wizard. If the Instrument Controller Service or Server Configuration was already running, it is updated with the new configuration information.

#### **B.10.2 Selecting Configuration Properties**

When the Dionex PDA is added to an instrument or timebase, default configuration properties are selected. Verify the default settings for the detector and select other settings as required.

1. To open the configuration properties dialog box, right-click the detector in the instrument or timebase and click **Properties**.

The Properties dialog box for the detector appears (see Figure B-8).

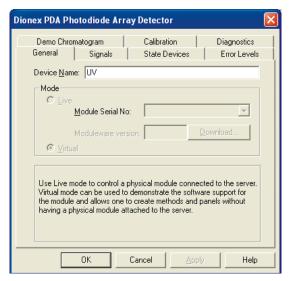


Figure B-8. Dionex PDA Properties Dialog Box

- 2. Review the default settings for the detector and change any settings that are not correct for your system. If you have questions about parameters, click the **Help** button.
- 3. When you finish, click **OK** to close the dialog box.
- 4. On the **File** menu, click **Save Installation** to save the configuration.

## **B.11 Starting the Chromeleon Client**

#### **Chromeleon 7 Client**

- 1. Click Start > All Programs > Chromeleon 7 > Chromeleon 7.
- To display the Chromeleon 7 ePanel Set, click the Instruments Category Bar on the Console. Chromeleon 7 connects to the instrument and displays the ePanel Set.
- 3. To view the Dionex PDA ePanel (see <u>Figure B-9</u>), click the **UV\_Vis** tab on the ePanel Set. The ePanel provides access to Dionex PDA functions, as well as detailed status and diagnostics information.

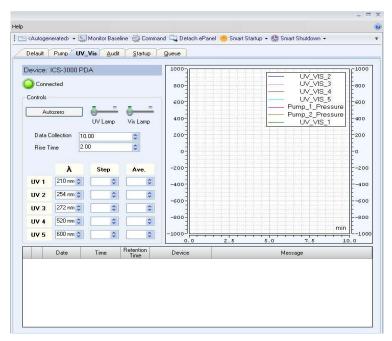


Figure B-9. Example Dionex PDA ePanel in Chromeleon 7

#### Chromeleon 6.8 Client

- 1. Click **Start** on the Windows taskbar and select **All Programs > Chromeleon** > **Chromeleon**

3. To display the Dionex PDA Control panel (see <u>Figure B-10</u>), select the **Dionex PDA** tab on the panel tabset. The Control panel provides access to Dionex PDA functions, as well as detailed status and diagnostics information.

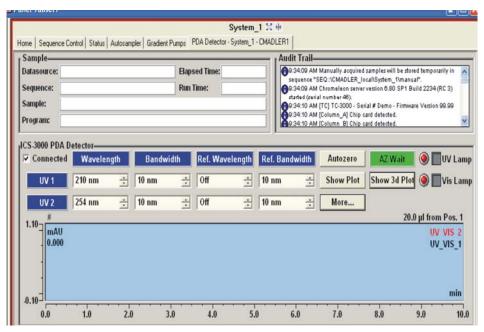


Figure B-10. Example Dionex PDA Control Panel in Chromeleon 6.8

# Dionex PDA Operator's Manual

# **C** • Reordering Information

Part Number	Item
939016T	Deuterium lamp
056123T	Tungsten lamp
954763	Fuse, 4.0 amps (fast-blow IEC 127)
923686	Relay/TTL connector, 12-pin
043598	Twisted wire assembly, 2 meters (8 ft)
063246	USB cable, 1 m (3 ft)
	Standard Flow Cell: PEEK
056346	PEEK standard flow cell (with heat exchangers)
057304	PEEK standard flow cell outlet tubing assembly
055903	Reducing union fitting (PEEK)
043276	Ferrule
043275	Nut
060498	Dionex AD25/Dionex PDA/Dionex PDA-100 Flow Cell Windows Replacement Kit
	Standard Flow Cell: 316 Stainless Steel
056126	Stainless steel standard flow cell (with heat exchangers)
056124	Stainless steel standard flow cell outlet tubing assembly
055902	Reducing union fitting (stainless steel)
010262	Ferrule
010261	Nut
060498	Dionex AD25/Dionex PDA/Dionex PDA-100 Flow Cell Windows Replacement Kit
	Semi-Micro Flow Cell: PEEK
064169	PEEK semi-micro flow cell (with heat exchangers)
057304	PEEK semi-micro flow cell outlet tubing assembly
055903	Reducing union fitting (PEEK)
043276	Ferrule
043275	Nut

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Part Number	Item
060498	Dionex AD25/Dionex PDA/Dionex PDA-100 Flow Cell
	Windows Replacement Kit
	Semi-Micro Flow Cell: 316 Stainless Steel
063884	Stainless steel semi-micro flow cell (with heat exchangers)
051650	Stainless steel semi-micro flow cell outlet tubing assembly
055902	Reducing union fitting (stainless steel)
010262	Ferrule
010261	Nut
060498	Dionex AD25/Dionex PDA/Dionex PDA-100 Flow Cell
	Windows Replacement Kit
	Semi-Preparative Flow Cell: PEEK
064167	PEEK semi-preparative flow cell
042855	PEEK semi-preparative flow cell outlet tubing assembly
042627	Union fitting
043276	Ferrule
043275	Nut

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