



Thermo Scientific Dionex UltiMate 3000 Series

PCM-3000

pH and Conductivity Monitor

for DAD-3000(RS), MWD-3000(RS), and VWD-3x00(RS)

Operating Instructions (Original Operating Instructions)



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1 Introduction

1.1 How to Use this Manual

The layout of this manual is designed to provide quick reference to the sections of interest to the reader when operating the Thermo ScientificTM DionexTM pH and conductivity monitor. However, in order to obtain a full understanding of the monitor, Thermo Fisher Scientific recommends that you review the manual thoroughly before beginning operation.

Almost all descriptions in the manual apply to all variants of pH and conductivity monitor in the UltiMate 3000 series.

The following conventions apply to the descriptions throughout this manual:

- The term "the monitor" or "the device" is used throughout the manual.
- If not otherwise stated, the descriptions for the ViperTM capillary connections apply also to the nanoViperTM and possible other Viper capillary connections.
- The representation of a component in this manual may be slightly different from the real component. However, this does not influence the descriptions.
- The descriptions in this manual refer to ChromeleonTM 6.80 SR12 and detector firmware version 2.40 (DAD/MWD) or 3.70 (VWD). If you want to operate the detector from Chromeleon 7, note the information on page 15.

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1.2 Safety

1.2.1 Symbols in the Manual

At various points throughout the manual, messages of particular importance are indicated by certain symbols:

i Tip: Indicates general information, as well as information intended to optimize the performance of the instrument. **M** Important: Indicates that failure to take note of the accompanying information could cause wrong results or may result in damage to the instrument. **Important:** Indique que ne pas tenir compte de l'information jointe peut conduire à de faux résultat ou endommager l'instrument. **STOP** Warning: Indicates that failure to take note of the accompanying information may result in personal injury. **Avertissement:** Indique que ne pas tenir compte de l'information jointe peut entraîner des blessures corporelles.

1.2.2 Safety Precautions

When working with analytical instrumentation, you must know the potential hazards of using chemical solvents.

I Tips: Before operating the monitor for the first time, read this manual once to make vourself familiar with the contents of this manual

> Please also note the information in the related sections and in the Operating Instructions of the pump and the optical detector.



STOP Warning: All users of the device must observe the following safety precautions and all additional safety precautions in this manual to avoid the possibility of personal injury or damage to the device when operating the device or carrying out any maintenance or service procedures.

Protective equipment •

When performing any work on or near the HPLC system, wear personal protective equipment (protective clothing, safety gloves, safety glasses) as required by the hazard of the mobile phase and sample. For information about the proper handling of a particular substance and for advice on specific hazards, refer to the material safety data sheet for the substance you are using. Observe the guidelines of Good Laboratory Practice (GLP).

An eyewash facility and a sink should be close to the device. If any substance splashes on the eyes or skin, wash the affected area and seek medical attention.

• Hazardous substances

Many organic solvents, mobile phases, and samples are harmful to health. Be sure that you know the toxic and infectious properties of all substances that you are using. You may not know the toxic or infectious properties of many substances that you are using. If you have any doubt about a substance, treat it as if it contains a potentially harmful substance. For advice on the proper handling of a particular substance, refer to the Safety Data Sheet (SDS) of the manufacturer. Observe the guidelines of Good Laboratory Practice (GLP).

Dispose of waste substance in an environmentally safe manner that is consistent with all local regulations. Do not allow flammable, toxic, and/or infectious substances to accumulate. Follow a regulated, approved waste disposal program. Never dispose of flammable, toxic, and/or infectious substances through the municipal sewage system.

• Hazardous gases

Install the HPLC system in a well-ventilated laboratory. If the mobile phase or sample includes volatile or flammable solvents, do not allow them to enter the workspace. If the mobile phase or sample includes volatile or flammable solvents, avoid open flames and sparks.

• Electrostatic discharge

Discharge of electrostatic energy may lead to sparking and can constitute a fire hazard. Keep in mind that liquid flowing through capillaries can generate static electricity. This effect is particularly pronounced in insulating capillaries and with non-conductive solvents (for example, pure acetonitrile).

Take appropriate measures to prevent the generation of static electricity near the HPLC system. For example, make sure that the air humidity level in the laboratory is sufficiently high and provide proper ventilation, wear anti-static clothing or shoes, prevent accumulation of air bubbles in waste lines, and use grounded waste containers. Use only non-conductive capillaries to direct solvents into the waste container. With electrically conductive capillaries, make sure that they are properly grounded.

• Self-ignition of solvents

Do not use solvents for which the self-ignition temperature is below 150 °C. In case of leakage, these solvents may self-ignite on a hot surface.

• Capillaries, capillary connections, open connections

- Capillaries, especially non-metallic capillaries may burst, slip out of their fittings or may not be screwed in. This may result in substances spraying out of the open connections.
- ◆ In an UltiMate 3000 system, some components are made of PEEK[™]. This polymer has superb chemical resistance to most organic solvents. However, it tends to swell when in contact with trichlormethane (CHCl₃), dimethyl sulfoxide (DMSO), or tetrahydrofuran (THF). In addition, it is attacked by concentrated acids, such as, sulfuric acid and nitric acid or a mixture of hexane, ethyl acetate, and methanol.

In both cases, capillaries may start leaking or they can burst. Swelling or attack by concentrated acids is not a problem with brief flushing procedures.

- Do not use tubing that is stressed, bent, kinked, or damaged.
- Capillary connections can be contaminated by harmful substances or harmful substances can escape from open connections.
- In an UltiMate 3000 Bio RS system, some system capillaries are made of MP35N[®], a nickel-cobalt based alloy. Individuals with sensitivity to nickel/cobalt may show an allergic reaction from skin contact.
- Always wear safety glasses when handling fused silica tubing, for example, during installation or when cutting capillaries to the length.
- Be aware that liquid may have been spilled when removing the hood. If a leak occurs, remedy the situation immediately.
- Replace faulty communication cables.
- Replace faulty power cords. Never use a power cord other than the power cords provided for the device.
- Use only the original spare parts and accessories authorized for the device by Thermo Fisher Scientific.
- When operating the HPLC system, always set a lower pressure limit for the pump. This prevents damage resulting from leakage or from running the pump dry.
- The module is primed with 2-propanol when being shipped from the factory. Make sure to flush the device with water or a buffer solution before first use of the pH electrode.
- After operation, rinse out buffers and solutions that form peroxides.
- Before switching from buffer to organic solution, rinse the analytical system thoroughly with de-ionized or HPLC grade water.
- When switching to another solvent, ensure that the new solvent is miscible with the one contained in the HPLC system. If the solvents are not miscible, the system can be damaged, for example, by flocculation.
- Use only standard solvents (HPLC grade) and buffers that are compatible with all parts in the monitor that may be exposed to solvents.
- Before interrupting operation for several days or more or when preparing the monitor for transport, observe the precautions for shutting down the monitor (\rightarrow page 47).
- Do not use the module in ways other than those described in these Operating *Instructions*.
- Keep the operating instructions near the device to be available for quick reference.

1.2.3 Consignes de Sécurité

Si vous utilisez d'instrumentation analytique, vous devez connaître les risques d'utilisation de produit chimiques.

Veuillez noter: Avant de commencer à utiliser l'instrument, assurez-vous que vous vous êtes familiarisés avec le contenu de ce manuel.

Référez-vous aux sections correspondantes dans ce manuel et aux consignes de sécurité dans les manuels du detecteur et de la pompe.

Avertissement: Toutes les personnes utilisant l'instrument doivent observer les consignes de sécurité suivantes et dans les autres chapitres de ce manuel pour éviter une mise en danger de sa personne ou de dommage à l'instrument pendant l'utilisation et des opérations de maintenance ou service de l'instrument.

• Equipment de protection

Pour tous le travaux sur le système HPLC ou à proximité, portez l'équipement de protection personnel (vêtements de protection, gant de sécurité, lunettes de protection) qui correspond aux risque découlant de la phase mobile et/ou de l'échantillon. Pour les informations sur la manipulation correcte des composés et des recommandations pour les situations de risque spécifiques, veuillez consulter la fiche de données de sécurité des substances que vous utilisez. Veuillez respecter des directives des Bonnes Pratiques de Laboratoire (BPL).

Une installation permettant de se laver les yeux ainsi qu'un lavabo doivent se trouver à proximité du système. Si une substance, quelle qu'elle soit, entre en contact avec vos yeux ou votre peau, rincez abondamment la zone affectée à l'eau, puis.

Substances dangereuses

De nombreux solvants organiques, phases mobiles et échantillons sont nuisibles à la santé. Informez-vous de propriétés toxicologiques et infectieuses de toutes les substances que vous utilisez. Les propriétés toxicologiques et infectieuses de nombreuses substances peuvent être mal connues. Au moindre doute concernant une substance, traitez-la comme s'il contenait une substance potentiellement dangereuse. Pour des instructions comment utiliser correctement des composés particuliers, veuillez consulter à la fiche de données des sécurités du fabricant respectif. Veuillez respecter des directives des Bonnes Pratiques de Laboratoire (BPL).

Débarrassez-vous de tous les déchets de substances de manière écologique, conformément à la règlementation en vigueur au niveau local. Empêchez impérativement l'accumulation de solvants inflammables, toxiques et/ou infectieux. Suivez un programme d'élimination des déchets règlementé et approuvé. Ne jetez jamais de solvants inflammables, toxiques et/ou infectieux dans le système municipal d'évacuation des eaux usées.

• Gaz dangereux

Installez le système HPLC dans un laboratoire bien ventilé. Si la phase mobile ou l'échantillon contient des solvants volatils ou inflammables, vous devez assurer qu'ils ne pénètrent dans l'espace de travail. Si la phase mobile ou l'échantillon contient des solvants volatils ou inflammables, évitez les flammes nues et les sources d'étincelles à proximité.

• Décharge électrostatique

Décharge électrostatique peut provoquer la formation d'étincelles et peut présenter un risque d'incendie. Veuillez noter que des solvants fluides dans les capillaires peuvent se charger automatiquement. Cet effet se peut produire particulièrement forte dans les capillaires isolants et avec des solvants non-conducteurs (par exemple, l'acetonitrile pur).

Prenez des mesures appropriées pour éviter les charges électrostatiques à proximité du système HPLC. Par exemple, s'assurez qu'il y a une humidité de l'air suffisante et une ventilation adéquate dans le laboratoire, portez des vêtements ou équipement de protection antistatique, évitez l'accumulation de bulles d'air dans les lignes de déchets et utilisez des réservoirs à déchets mis à la terre.

Utilisez uniquement des capillaires non-conducteurs pour diriger solvants au réservoir de déchets. Capillaires électriquement conducteur devrait être mis à la terre.

• Inflammation spontanée des solvants

N'utilisez aucun solvants avec une température d'auto-inflammabilité inférieure à 150° C. Si une fuite se produit, ces solvants peuvent s'auto-enflammer au contact d'une surface chaude.

• Capillaires, connecteur capillaires, connexions ouvertes

- Descapillaires, en particulier les capillaires non-métalliques, pourraient fendre ou glisser des connecteurs ou ne peuvent pas être vissés. Ceci peut en résulter aussi que des substances pourraient jaillir des connexions ouvertes.
- Dans un système UltiMate 3000, certaines composantes sont en PEEK. Bien que ce polymère présente une excellente résistance chimique à la plupart des solvants organiques, il a tendance à gonfler lorsqu'il est en contact prolongé avec du chloroforme (CHCl3), du diméthyle sulfoxyde (DMSO) ou du tétrahydrofurane (THF). De plus, il est attaqué par des acides concentrés tels que l'acide sulfurique et l'acide nitrique ou d'un composé du hexane, éthyle acétate et méthanol. Ceci peut causer des capillaires de fuite ou risquer des capillaires d'éclater. Ces acides peuvent cependant être utilisés dans le cadre de procédures de nettoyage, à condition que l'exposition soit brève.
- N'utilisez pas de capillaires écrasés, pliés, abimés ou endommagés.
- Les connecteurs capillaires pour pourrait être contaminé par des substances dangereuses ou des substances dangereuses pourrait sortir des connexions ouvertes.
- Dans un système UltiMate 3000 Bio RS, certains capillaires du système Viper sont faits d'alliage de nickel-cobalt MP35N. Contact avec la peau peut provoquer une réaction chez les personnes qui sont sensibles au nickel/cobalt.

- Portez des lunettes de protection lorsque vous manipulez des capillaires en silice fondue (pendant l'installation, découpe, etc.).
- Quand vous enlevez le capot de protection de l'appareil, prenez en compte le fait que liquide peut fuir. Si une fuite se produit, remédiez immédiatement au problème.
- Remplacez les câbles de communication défectueux.
- Remplacez les cordons d'alimentation électrique défectueux. Utilisez uniquement les cordons d'alimentation électrique spécifique à l'instrument.
- Utilisez seulement des pièces de rechange originales et des accessoires autorisés par Thermo Fisher Scientific.
- L'instrument est stocké sous 2-propanol. Avant utilisation de la sonde à pH, effectuez un rinçage à l'eau ou une solution tampon.
- Après utilisation, purgez le système des tampons et des susceptibles de former des peroxydes.
- Lorsque vous passez d'une solution saline à un solvant organique, effectuez un rinçage intermédiaire du système HPLC à l'eau dé-ionisée ou qualité HPLC.
- Lorsque vous passez à un autre solvant, assurez-vous que le nouveau solvant soit miscible avec celui qui se trouve dans le système HPLC. Dans le cas contraire, le système HPLC peut être endommagé; par exemple, par des floculations.
- Utilisez uniquement des solvants (qualité HPLC) et des solutions salines compatibles avec les matériaux exposés phase mobiles.
- Avant d'interrompre le fonctionnement pendant plusieurs jours ou plus, observez les précautions figurant en page 47).
- N'utilisez pas l'instrument de manière autre que celles décrites dans ce manuel.
- Conservez ce manuel à proximité de l'instrument pour pouvoir le consulter facilement.

1.3 Intended Use

For Research Use Only. Not for use in diagnostic procedures.

The device is designed to be operated only be qualified and authorized personnel. All users must know the hazards presented by the device and the used substances.

PCM-3000 pH and conductivity monitor is designed as a module for measuring the pH and conductivity value of liquids for laboratory research use in HPLC or UHPLC (ultra-high performance liquid chromatography) applications in combination with an optical UV-VIS detector (diode array detector, multiple wavelength detector, or variable wavelength detector) of the UltiMateTM 3000 series.

The monitor can be controlled by the Chromeleon Chromatography Management System. Being part of the UltiMate 3000 system, it can also be operated with other data systems, such as

- XcaliburTM, CompassTM/HyStarTM, or Analyst[®]. Installation of the DCMSLink (Thermo Scientific Dionex Chromatography Mass Spectrometry Link) software is required in addition to the installation of the data system.
- EmpowerTM. Installation of the Thermo Scientific Dionex Instrument Integration Software is required in addition to the installation of the data system.

Observe the following:

- Note that the monitor may be operated only with accessories and spare parts recommended by Thermo Fisher Scientific (→ page 65) and within its technical specifications (→ page 63).
- Use only standard solvents (HPLC grade or LC-MS grade: 0.2 µm filtered). When using the pH electrode, do not use organic solvents.
- Also observe the information about the solvent compatibility of the other UltiMate 3000 system modules, in particular of the optical detector and the pump. For more information, refer to the *Operating Instructions* for the modules.

If there arise any questions regarding appropriate usage of the module, contact Thermo Fisher Scientific before proceeding.

SUP Warning:

If the device is used in a manner not specified by Thermo Fisher Scientific, the protection provided by the device could be impaired. Thermo Fcisher Scientific assumes no responsibility and will not be liable for operator injury and/or instrument damage. Whenever it is likely that the protection is impaired, the instrument must be disconnected from all power sources and be secured against any intended operation.

Avertissement: Si l'instrument est utilisé de façon non spécifiée par Thermo Fisher Scientific, la protection prévue par l'instrument pourrait être altérée. Thermo Fisher Scientific n'assume aucune responsabilité et ne sera pas responsable des blessures de l'operateur et/ou des dommages de l'instrument. Si la protection de l'instrument n'est pas garanti à tout moment, débranchez l'instrument de toutes les sources d'alimentation électrique et assurez-vous que l'instrument n'est pas utilisé involontairement.

2 Overview

2.1 Unit Description

The PCM-3000 pH and conductivity monitor is designed as a module for measuring the pH and conductivity value of liquids in combination with a diode array detector, multiple wavelength detector, or variable wavelength detector of the UltiMate 3000 series.

- A pH electrode is used to monitor over a period of time the pH value of a liquid that is flowing through the pH flow cell.
- The pH flow cell is equipped with a temperature sensor. Chromeleon automatically considers the measured temperature value when calculating the pH value, thus improving the accuracy of the measurement.
- A conductivity flow cell is used to monitor over a period of time the conductivity value of a liquid that is flowing through the flow cell.
- The conductivity flow cell is equipped with a temperature sensor. Chromeleon automatically considers the measured temperature value when calculating the conductivity value.
- Calibration of the conductivity flow cell and pH electrode is easy and can be performed directly in Chromeleon.
- Features for monitoring the calibration are provided in Chromeleon to ensure system reliability.

2.2 Configurations

The pH and conductivity monitor measures the pH value and conductivity of a flow of liquid. The measured values are processed by the instrument driver of the detector and can be viewed in Chromeleon as well as on the detector display. The module is available in the following configurations:

Description	Part no.
PCM-3000 for DAD/MWD detectors of the UltiMate 3000 series.	6082.2000
PCM-3000 for VWD detectors of the UltiMate 3000 series.	6082.2005

The shipment includes a suitable plug-in extension board for installation in the detector, a flow cell carrier with pre-mounted pH and conductivity flow cells, a hood, a pH electrode, and the required accessories (\rightarrow page 65). The pH electrode and both flow cells are available as spare parts (\rightarrow page 66).

The figure below shows the components of the pH and conductivity monitor.



2.3 pH Flow Cell and pH Electrode

The pH flow cell is pre-installed on the flow cell carrier. A dummy electrode protects the flow cell from dust. As the temperature of the liquid is required for calculating the pH value, the pH flow cell is equipped with a temperature sensor, which is connected to the PCM-3000 extension board on the detector rear panel via a cable. The temperature values can be monitored in Chromeleon as a separate signal.



Fig. 2: pH flow cell with dummy electrode

The pH electrode shipped with the monitor is placed in the flow cell during pH measurements. An electrode storage container is mounted on the flow cell carrier for storing the electrode "offline"; that is, outside of measurements. Before initial operation, the pH electrode must be calibrated.

2.4 Conductivity Flow Cell

The conductivity flow cell is pre-installed on the flow cell carrier.

An identification chip is fitted to the flow cell in the factory. The chip stores the cell calibration data, among other information. When the flow cell is connected to the detector rear panel, the chip is connected to the electronics. It is therefore not mandatory to calibrate the cell before initial operation. To achieve optimum accuracy with the used substances, however, calibrating the flow cell is recommended before operation (\rightarrow page 42).

As the conductivity of a substance is influenced by its temperature, the conductivity flow cell is equipped with a temperature sensor. The temperature values can be monitored in Chromeleon as a separate signal. Chromeleon can then perform a temperature compensation of the conductivity value.



Fig. 3: Conductivity flow cell with connection cable

2.5 Fluid Connections

The capillary connections are premounted at the factory. A recess is provided in the hood to accommodate the connection from the optical detector to the conductivity flow cell. In the detector enclosure, the capillary slot on the left can be used for this connection.

The capillary from the pH flow cell to the waste can be guided through another small recess at the rear of the hood.

2.6 Operation with Chromeleon

The monitor is controlled by the Chromeleon Chromatography Management System. This requires that a supported optical detector is installed and also controlled by Chromeleon. For information about how to control the optical detector by Chromeleon, refer to the detector's Operating Instructions.

One of the following Chromeleon versions is required to control the PCM-3000:

- Chromeleon 6.80 DU10b (Driver Update) or later
- Chromeleon 7.1 DU0a (Driver Update) or later

All software details in this manual refer to Chromeleon 6.80.

If you want to operate the system with Chromeleon 7, refer to the following documents for information about how to perform the related processes in Chromeleon 7 (all documents are included in the Chromeleon 7 shipment):

- Chromeleon 7 Help—provides extensive information and comprehensive reference material for all aspects of the software.
- Quick Start Guide—describes the main elements of the user interface and guides you step-by-step through the most important workflows.
- Reference Card—provides a concise overview of the most important workflows.
- Installation Guide—provides basic information about module installation and configuration. For specific information about a certain module, refer to the Chromeleon 7 Instrument Configuration Manager Help.

Please also note that Chromeleon 7 terminology is different from the terminology used in Chromeleon 6.80. For details, refer to the 'Glossary - Chromeleon 7,' which is available in the Documents folder of your Chromeleon 7 installation.

3 Installation

i Tips: The pictures in this section show installation with a DAD-3000. Note that the installation steps are identical for other optical detectors, although the rear panel of the extension board may be different.

Operation of the PCM-3000 requires detector firmware version 2.20 (DAD-3000(RS) and MWD-3000(RS) or 3.40 (VWD-3x00(RS)), or higher. Perform a firmware update, if necessary.

You can install either a PCM-3000 or a DAC board (to provide analog outputs) in a detector. If a DAC board is already installed, remove the DAC board first before you install the PCM-3000.

3.1 Facility Requirements

- Make sure that the installation site meets the power and environmental specifications listed in the Technical Information section (→ page 63).
- Install the monitor in the laboratory on a stable surface that is free of vibrations.
- Make sure that the surface is resistant to solvents.
- Avoid locations with extreme changes in temperature.
- Avoid direct sunlight and high humidity.
- Allow sufficient clearance behind and to the sides of the optical detector for power connections and ventilation, even after the PCM-3000 is installed.

3.2 Unpacking

All electrical and mechanical components of the module are carefully tested before the module is shipped from the factory. After unpacking, inspect the instrument for any signs of mechanical damage, which might have occurred during transit.

Tips: Immediately report any shipping damage to both, the incoming carrier and Thermo Fisher Scientific. Shipping insurance will compensate for the damage only if reported immediately.

Keep the original shipping container and packing material. They provide excellent protection for the instrument in case of future transit. For warranty information, see the warranty statement in the terms of sale.

- 1. Place the shipping container on the floor and remove all parts.
- 2. Remove any foam spacers and plastic packaging.

3. Before installing the extension board, wait approximately four hours to allow the instrument to come to room temperature and to allow any condensation that might have occurred during shipping to evaporate. After four hours, check the monitor; if condensation still exists, allow the monitor to continue to warm up (without connecting it to the power source) until the condensation is completely gone.

3.3 Installing the Extension Board

- Warning: Before installing the extension board, turn off the main power switch at the detector's rear panel and disconnect the power cord from its source.
 - Avertissement: Avant de installer la carte d'extension, arrêtez l'instrument. Assurezvous de bien débrancher le cordon d'alimentation de la source secteur.
- 1. Use appropriate grounding protection to avoid electrostatic discharge, which may result in damage to electronic components.
- 2. Loosen the four screws indicated in the picture using a size 1 Phillips screwdriver and remove the access panel from the rear panel.



Fig. 4: Screws on the access panel

3. Slide the left guide bar of the board onto the green edge next to the USB port.



Fig. 5: Installing the extension board

4. Push the board partially into the detector enclosure. Using your finger, apply gentle pressure on the TEMP connection until the board locks in place.

5. Attach the extension board to the rear panel, using the four screws from the access panel. Conductivity



Fig. 6: PCM-3000 connections on the detector rear panel

3.4 Installing the Flow Cell Carrier

The flow cell carrier can be mounted on the detector. To facilitate installation, the detector should be placed with the left side protruding the edge of the lab bench for a few centimeters.

- **I** Tip: Mounting the flow cell carrier on the detector is not possible for older VWD detectors, as the borings for the screws shown below are not available. In this case, place the flow cell carrier on the side or on top of the HPLC system.
- 1. Three square recesses on the bottom of the detector are provided to accommodate the flow cell carrier profile. The screws for fixing the flow cell carrier must be screwed into the borings next to the two outer recesses (\rightarrow Fig. 7).



Fig. 7: Recesses on the bottom of the detector

2. Place the flow cell carrier in the recesses from below (\rightarrow Fig. 8). The borings on the flow cell carrier must be aligned with the borings on the bottom of the detector as shown. The guide bars of the left recess are well visible from the side and may help you to locate the proper position.



Fig. 8: Installing the flow cell carrier

- 3. Use the two screws shipped with the monitor and insert them in the two borings, then fix them with a screwdriver (TX20, included in accessories).
- 4. Move the detector to place it completely on the lab bench and/or position it in the HPLC System as described in the Operating Instructions for the detector, if necessary. When lifting or moving the detector, always lift by the bottom or sides of the instrument. Do not lift the detector by the flow cell carrier.
- Connect the conductivity flow cell cable (6-pin Mini DIN connector) to the COND port on the detector rear panel (→ Fig. 6); note the arrow that is visible on the upper side of the connector when connected.
- 6. Connect the pH flow cell temperature sensor cable (4-pin Mini DIN connector) to the TEMP port on the detector rear panel (\rightarrow Fig. 6); note the arrow that is visible on the upper side of the connector when connected.
- 7. Place the pH electrode (\rightarrow section 3.5) in the offline storage container.

3.5 Placing the pH Electrode in the Offline Storage Container

Before initial operation, the pH electrode is placed in the offline storage container. Leave the electrode in the storage container for at least 30 minutes before performing measurements. Observe the general guidelines for pH electrodes and flow cells (\rightarrow page 28).

If you do not want to measure pH, do not unpack or install the electrode.

1 Tips: Always handle the pH electrode with care, as the glass membrane is easily damaged or contaminated.

To protect the electrode from drying out, always store it in the offline storage container when no pH measurements are performed. The electrode must not be stored in deionized water.

Fill the storage container for the pH electrode (→ Fig. 1: Flow cell carrier) with a solution of pH 4 buffer and potassium chloride (100 g KCl in 1 L of pH4 buffer, based on potassium hydrogen phthalate (KHP)). Fill the liquid until it reaches the bottom edge of the slant.



Fig. 9: Filling level for the storage container

- 2. Remove the pH electrode from its packaging, then carefully remove the soaker cap that is filled with a mixture of pH4 buffer and potassium chloride. Examine the electrode for damage. Finally rinse the cap with water and keep it together with the packaging.
- 3. Locate the lock nut that is shipped with the monitor and thread the cable of the pH electrode through the nut as shown in Fig. 10. This is required to fix the pH electrode to the pH flow cell during operation.



Fig. 10: Preparing the pH electrode

- Place the pH electrode in the offline storage container. Leave the electrode in the offline storage container until it is used for a measurement. Refer to section 5.2.1 (→ page 35) for information how to place the pH electrode in the pH flow cell for measurements.
- 5. Connect the pH electrode cable (BNC connector) to the BNC connector of the PCM-3000 extension board on the detector rear panel. The connector has a slot to be aligned with the pin on the female connector. Turn the connector clock-wise to lock it.
- 6. Move the strain relief (\rightarrow Fig. 11) aside and route the electrode cable through the dedicated (middle) guide.



Fig. 11: Strain relief (left: closed, right: open)

7. Reconnect the detector to the power source.

3.6 Setting Up the PCM-3000 in Chromeleon or DCMSLink

After the pH and conductivity monitor is installed, it must be set up in the chromatography software. This requires that an optical detector (DAD-3000(RS), MWD-3000(RS) or VWD-3000(RS) has been installed and configured.

- For details on setting up the detector in Chromeleon, see the Operating Instructions for the detector, or the Chromeleon Help.
- For details on setting up the detector in DCMSLink, see the Operating Instructions for the detector, or the *DCMSLink Installation Guide*, which is provided on the DCMSLink DVD in the *Additional Documents**DCMSLink User Documents* folder.
- 1. Turn on the detector power.
- 2. In Chromeleon, open the **Properties** dialog for the detector in the **Server Configuration** program. On the **Detector** page, click the **PCM** option. Only if this option is clicked, the channels for pH and conductivity and the temperature sensors will be available in Chromeleon.

DAD-3000(RS) Configuration	x
General Detector Signals PCM	
DAD Type C DAD-3000 © DAD-3000RS	
Device Device Name:	
IV Lamp IV VIS Lamp	
Board Extension C None C DAC © PCM	

Fig. 12: Detector page (here: DAD-3000RS)

3. The **PCM** tab page lists all signals that the monitor can record. The signal type and name of each signal are displayed. To allow raw data collection for a signal, select the **Enabled** check box next to the signal name. If the check box is cleared, Chromeleon cannot collect raw data for the signal. To change a signal name, overwrite the existing name directly in the Name line.

The **Device Name** box displays the name used to identify the monitor in the installation environment and in the Chromeleon Client program (default: **UV_PCM**). Accept the default name. If you enter a different name, you may have to re-link the controls on the control panels and edit the name in the program files.

DAD-3000(RS) Configuration General Detector Signals PCM Device Name: UV_PCM						
		Enabled	, _ Туре	Name		
	•		рН	рН		
		•	Temperature	pH_Temp		
		•	Conductivity	Cond		
		•	Temperature	Cond_Temp		

Fig. 13: Signals tab page (here: DAD-3000RS)

4 Preparation for Operation (Startup)

4.1 Overview

M Important: The device is filled with 2-propanol when being shipped from the factory. Make sure to flush the device with water or a buffer solution before first use of the pH electrode.

Important: L'instrument est stocké sous 2-propanol. Avant utilisation de la sonde à pH, effectuez un rinçage à l'eau ou une solution tampon.

After you have installed and connected the pH and conductivity monitor as described in sections 3.3 through 3.6 (\rightarrow page 17 and following pages), prepare the monitor for operation:

- 1. Connect a drain tubing (\rightarrow section 4.2).
- 2. Connect the flow cell capillaries (\rightarrow section 4.3).
- 3. Place the pH electrode in the pH flow cell (\rightarrow section 5.2.1, page 35).
- 4. Calibrate the pH electrode (\rightarrow section 5.2.2, page 35).
- 5. Calibrate the conductivity flow cell in the measuring range if necessary (\rightarrow section 5.3.2, page 42).

4.2 Connecting a Drain Tube

To discharge liquid leaks from a flow cell or capillary connection, the flow cell carrier has a drain port (\rightarrow Fig. 1: Flow cell carrier, page 12). Discharge liquid to an appropriate waste container. Push the tubing that is shipped with the monitor into the connecting piece as far as it goes in to establish a self-sealing connection.

4.3 Fluid Connections

When connecting capillaries, please observe the following general precautions:

- Observe the precautionary statements for capillaries and capillary connections in section 1.2.2 (→ page 3).
- Use only the capillaries shipped with the module and original spare PEEK capillaries. Do *not* use steel or titanium capillaries.
- Capillary connections between the detector flow cell and the flow cell inlet should be as short as possible to avoid peak broadening effects due to excessive dead volume.
- Do not overtighten fitting connections. If you observe leakage on the connection, tighten a little further.

If leakage still exists, first consider cleaning the connection port with a cleaning swab (part no. 6040.0006). Replace the capillary and/or fitting if this does not eliminate the problem.

Reuse used fittings and ferrules only for the same capillary connection. This is to avoid increased dead volume or damage to the system and leakage

• When connecting capillaries to the pH and conductivity flow cells, always connect the inlet capillary at the bottom and the outlet capillary at the top. Inlet and outlet must not be exchanged. This ensures that air in the flow cell can escape.

Connecting the pH flow cell and conductivity flow cell

The capillary connections are premounted at the factory.

- 1. Connect the conductivity flow cell inlet capillary to the flow cell outlet of the optical detector. A recess is provided in the hood for this capillary. When closing the detector front panel door, avoid bending the capillary and make sure that it is routed to the outside through the slot on the left side.
- 2. Connect a waste tubing to the waste capillary (included in accessories). A recess is provided in the hood for the waste capillary.



Fig. 14: Capillary connections on the PCM-3000

No.	Description		
1	Capillary from flow cell outlet (through capillary slot on the optical detector)		
2 Conductivity flow cell inlet (lower connection)			
3	Conductivity flow cell outlet (upper connection)		
4	pH flow cell inlet (lower connection)		
5	pH flow cell outlet (upper connection)		
6	Clamp for the waste capillary		

As an alternative, you can use the pH flow cell or conductivity flow cell only. In this case, the capillaries should be connected to bypass the unused flow cell.

Connecting the pH flow cell without the conductivity flow cell

- 1. Remove the installed capillary between the conductivity flow cell and the pH flow cell.
- 2. Remove the capillary connection at the conductivity flow cell inlet, and connect the flow cell outlet of the optical detector to the pH flow cell inlet instead.

Connecting the conductivity flow cell without the pH flow cell

- 1. Connect the flow cell outlet of the optical detector to the conductivity flow cell inlet.
- 2. Remove the installed capillary between the conductivity flow cell and the pH flow cell.
- 3. Remove the waste capillary from the pH flow cell outlet and connect the waste capillary to the conductivity flow cell outlet instead.

Even if you do not want to perform pH measurements, we recommend that you place the pH electrode in the offline storage container as described in section 3.5 and connect it with the BNC connector on the rear panel of the detector.

4.4 General Notes for Operation

4.4.1 Mobile Phases

Observe the notes on mobile phase quality and delivery in an HPLC system as outlined in the Operating Instructions for the detector and the HPLC pump.

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 flow cells with a buffer-compatible solvent (in most cases, HPLC-grade water) immediately after the analysis. Do not allow buffers to remain in the flow cells for extended periods.

4.4.2 pH Flow Cell and Electrode

Observe the following notes on the pH flow cell:

- To protect the flow cell from dust, the dummy electrode that is shipped with the monitor should be installed on the pH flow cell whenever it is not used; that is, whenever the pH electrode is in the offline storage container.
- The backpressure on the flow cell must not exceed 7 bar. Do not connect any capillaries or instruments to the flow cell out line, as this might generate a higher backpressure. When switching valves while the flow is turned on, pressure peaks may occur and destroy the flow cell or pH electrode. Thermo Fisher Scientific recommends using a capillary or tube with an internal diameter of at least 0.75 mm at the flow cell out line.
- Do not try to clean the flow cell mechanically with abrasives, a brush, or similar. If necessary, use a soft, lint-free tissue. For information about how to clean the flow cell, see page 58.
- Do not pull the flow cell connection cable.

Observe the following notes on the pH electrode:

- Always handle the pH electrode with care, as the glass membrane is easily damaged or contaminated.
- The electrode must not be stored at temperatures below -11°C, as this may damage the glass.
- To protect the electrode from drying out, always store it in the offline storage container on the flow cell carrier when no pH measurements are performed; the storage container should always have a sufficient liquid level (→ page 21). The electrode must not be stored in deionized water.
- When using organic solvents, the pH electrode should not be used. Place the dummy electrode in the pH flow cell instead or avoid the flow cell as described on page 28.
- Never try to clean the pH electrode with abrasives, a brush, etc.. For information about how to clean the pH electrode, refer to page 56.
- pH electrodes have a limited lifetime and should be replaced if the performance has considerably deteriorated. Chromeleon supports functions for monitoring the performance (→ page 39).

4.4.3 Conductivity Flow Cell

Observe the following notes on the conductivity flow cell:

- The backpressure on the flow cell must not exceed 50 bar. Do not connect any capillaries or instruments to the flow cell out line as this might generate a higher backpressure. When switching valves while the flow is turned on, pressure peaks may occur and destroy the flow cell.
- If you suspect that the conductivity flow cell is blocked, disconnect it immediately from the flow cell of the optical detector, as the latter may be destroyed through the increased backpressure.
- For information about how to clean the flow cell, see page 60.
- Never hold the flow cell by the connection cable.

5 Operation and Maintenance

The pH and conductivity monitor is controlled by the Chromeleon Chromatography Management System. This requires that a supported optical detector is installed and also controlled by Chromeleon. For information about how to control the detector by Chromeleon, refer to the detector's Operating Instructions.

Turn on the detector by pressing the power switch on the rear of the detector. This will also supply power for the pH and conductivity monitor.

In addition, you can monitor the measured values for pH and conductivity on the detector display.

5.1 General Information about Control with Chromeleon

Before you begin, verify that

- 1. The Chromeleon software is installed on the computer and the license code is entered.
- 2. The optical detector is connected to the Chromeleon computer via a USB connection.
 - **Tip:** Verify that Chromeleon is installed on the computer and that the license code is entered *before* you connect the detector to the USB port on the Chromeleon computer and turn on the detector power. Only then, the USB driver for the detector is automatically loaded and the Windows[®] operating system can detect the detector when the power is turned on.
- 3. The optical detector is set up in Chromeleon, as described in the Operating Instructions for the detector.
- 4. The monitor is set up in Chromeleon, as described in section 3.6 (\rightarrow page 23).

Before you can operate the monitor with Chromeleon, you have to connect the timebase in which the detector is installed to the Chromeleon client program ($\rightarrow Operating$ *Instructions of the detector*).

5.1.1 Direct Control

Use direct control for calibration procedures and for defining limits. The commands are selected in the **Commands** (F8) dialog box or on a panel and are directly executed.

To open the Commands dialog box for the PCM-3000

- 1. Open a control panel (any panel is possible). To open a control panel, open the Chromeleon Browser and double-click a control panel in the **Dionex Templates/Panels** folder.
- 2. Connect the control panel to the timebase in which the detector is installed. On the Control menu, select Connect to Timebase, and then select the timebase on the Timebase tab. (The Control menu is visible only when a control panel is open.) For information about the **Timebase** dialog, click Help.
- 3. Press the F8 key or select **Command** on the **Control** menu.
- 4. To see the parameters and commands that are available for the detector, click the plus sign next to UV.
- 5. Scroll down to the area where the detector channels are listed. To see the parameters and commands that are available for the PCM-3000, click the plus sign next to a PCM-3000 channel:

UV_PCM: Module parameters, for example, for calibration and monitoring

pH_Temp: Temperature signal of the pH flow cell

pH: Signal of the pH value

Cond_Temp: Temperature signal of the conductivity flow cell

Cond: Signal of the conductivity value

6. The commands and parameters available in the dialog box may vary, depending on the display filter level (**Normal**, **Advanced**, or **Expert**). Change the display filter level if necessary. Right-click in the commands list and select the filter level on the menu.



Fig. 15: Commands dialog box
7. Verify that the detector is connected to Chromeleon. If it is not, select Connect to connect the detector.

To open a control panel for the PCM-3000

1. On the **View** menu, click **Default Panel Tabset** or click the corresponding icon on the toolbar **1**, and then connect to the Chromeleon server.

Chromeleon creates centralized control panels, called panel tabsets, for all timebases available on the Chromeleon server. A panel tabset provides control panels for the individual instruments in a timebase and, in addition, one or more panels for performing system-wide functions, for example, creating and running sequences. For more information about panel tabsets, see the Chromeleon Help.

- 2. On the Panel Tabset for your timebase, click the page for the PCM-3000.
- 3. Verify that the detector is connected to Chromeleon. If it is not, click Connect on the Detector page.

CLABOR22 xPG-3x00(RS/SD/BM) Sampler Col. Comp. Vw/D PCM-3000	2_PCM3000_23, 34
PCM Board Status	pH / Conductivity
Serial No.:	pH Conductivity
PCM Board Beta3	Current Value: 5.82 0.003 mS/cm
pH Flowcell	
Conductivity Flowcell	Temperature: 23.9 ° C 24.2 ° C
Conductivity	Calibration / Limits
Status: Calibrated ATC: 1.00 %/°C Progress: NotExecutedYet Ref. Temp.: 25 °C	Calibration Limits
рН	Signals
Status: Calibrated ATC: 1.00 %/°C Progress: NotExecutedYet Last Date: 2011-03-29	14.0 pH
Audit Trail	
□2:02:12 PM 0.000 SampleHeight = 2.000 🔼	
2:02:12 PM 0.000 InjectWash = NoWash 2:02:12 PM 0.000 LoopWashFactor = 0.000	0.00 0.20 0.40 0.60 0.80 1.00
2:02:12 PM 0.000 E00pWashPactor = 0.000 = 2:02:12 PM 0.000 PunctureOffset = 0.0	Conductivity [mS/cm]
= 2:02:12 PM 0.000 PumpDevice = "Pump"	
2:02:12 PM 0.000 InjectMode = Normal 2:02:12 PM 0.000 ColumnOven Temp.Step = 0.01	
=2:02:12 PM 0.000 ColumnOven_Temp.Average = Off	
2:02:12 PM 0.000 Pump_Pressure.Step = 0.01 2:02:12 PM 0.000 Pump_Pressure.Average = Off	
2:02:12 PM 0.000 Pdmp_Pressure.Average = 011 2:02:12 PM 0.000 %A.Equate = "Water"	0.00 0.20 0.40 0.60 0.80 1.00
]

Fig. 16: PCM-3000 control panel

The most important control functions are available on the control panel. All parameters and commands are also available in the **Commands** dialog box. To open the Commands box from the panel tabset, select Command on the Control menu.

5.1.2 Automated Control

The channels for pH and conductivity and the temperature sensors can be recorded by using a program (PGM file). Programs can be created automatically with the help of a software wizard or manually by editing an existing program. For information about how to create and run a program, refer to the Chromeleon Help or to the Operating Instructions for the optical detector.

You can activate and deactivate the channels for monitoring the pH value (**pH**) and conductivity value (**Cond**) as well as the temperature channels for the two flow cells (**pH_Temp** and **Cond_Temp**) in the Program Wizard on the page with the detector settings (**UV Options**, \rightarrow Fig. 17).

	No	Channel name	AcqOn[min]	AcqOff[min]	Wavel.[nm]	Bandw.[nm]	RefWavel.[nm]	^
	1	UV_VIS_1	0,000	30,000	230,0	1	Off	
	2	UV_VIS_2	0,000	30,000	254,0	1	Off	
	3	UV_VIS_3	0,000	30,000	270,0	1	Off	
	4	UV_VIS_4	0,000	30,000	300,0	1	Off	Ξ
	5	Temp_Lampho	0,000	30,000				
1	6	🔽 pH_Temp	0,000	30,000				
4	7	🔽 pH	0,000	30,000				
ר״	8	Cond_Temp	0,000	30,000				_
l U	9	Cond	0,000	30,000				
9	Signal Parameter Assistant III Please specify the peak width at half height for the narrowest expected peak in your chromatogram. III Peak Width 0,100 [0,002 min] Data Collection Rate 5,0 [Hz] Response Time 2,000 [s]			_ ,002 min] z]	3D Min. Wave 3D Max. Wave 3D Bunchwidth Slit Width: Data Collection Response Tim	elength: 800,(h: 1 Narrow h Rate: 10,0		im]

Fig. 17: UV Options page in the Program Wizard - (here: DAD-3000)

5.1.3 Displaying the Channels in the Chromatogram

If the channels for pH and conductivity and the temperature sensors are activated in the Program Wizard, the data will be available in Chromeleon. During data acquisition, you can monitor the signals in the two Online Plots on the control panel (\rightarrow Fig. 16). In the chromatogram, you can overlay the recorded channels with the channels of the optical detector ("Overlay"):

- In the Browser, right-click a sample, point to **Open**, and then click All Channels. All channels of a sample are displayed.
- If you want to display the PCM-3000 channels in separate panes, right-click in the chromatogram, and then click **Decoration**. In the **Chromatogram Decoration** dialog box, on the **Comparison** tab page, click the **Mixed** option in the **Arrangement** group.

To remove a channel from the overlay, right-click the chromatogram caption of the channel to be removed, and then click **Remove Chromatogram**.

For more information about channel overlays, see the Chromeleon Help.

5.2 pH Measurement

As described in section 5.1.2 (\rightarrow page 34), the **pH** channel is used to monitor the pH value in Chromeleon over a period of time. A program to monitor pH must include the **pH.AcqOn** (start data acquisition) and **pH.AcqOff** (end data acquisition) commands.

5.2.1 Placing the pH Electrode in the Flow Cell

Observe the general guidelines for pH electrodes and flow cells (\rightarrow page 28).

- **Tip:** Before you place the pH electrode in the flow cell, verify that the flow cell is free of salt deposits, and clean the flow cell if necessary (\rightarrow page 58), otherwise the electrode may be damaged.
- 1. Loosen the lock nut of the dummy electrode and remove the dummy electrode from the flow cell. Check that the o-rings (\rightarrow Fig. 18) are still in their place, and push them back into their grooves (using gloves) if necessary. (A set of replacement o-rings is available under part no. 6082.2035.)



Fig. 18: O-rings in the pH flow cell

- 2. Place the pH electrode in the pH flow cell and tighten the lock nut hand-tight. The nut and the electrode must be tightened such that they can no longer be moved.
- 3. Place the dummy electrode in the offline storage container and store the lock nut on the dummy electrode.

5.2.2 Calibrating the pH Electrode

The pH electrode must be calibrated on a regular basis. Daily calibration will ensure optimum accuracy. In each case, a calibration is required after the electrode was replaced, or when the ambient temperature changes significantly. The pH electrode can be calibrated directly inside the flow cell ("internally") or in separate vessels ("externally"). When you are calibrating externally, you must know the temperatures of the buffer solutions, or measure them separately with the help of a thermometer.

In general, choose the buffer solution(s) in the usual range of your measurements. For example; select pH=7.0 and pH=4.0 if you are measuring in a range below pH=7. Select pH=7.0 and pH=10.0 if you are measuring in a range above pH=7.

Calibration is performed with the help of Chromeleon. There are two types of calibration: one-point and two-point calibration.

Rinsing the pH electrode for external calibration

When you are calibrating in an external vessel, the electrode must be rinsed before and after calibration, as well as between the two buffer solutions (two-point calibration). Stir the electrode in a rinse solution (for example; deionized water) until it is thoroughly rinsed. Carefully shake the electrode afterward to remove residual drops of rinse solution.

To perform two-point calibration (recommended)

During a two-point calibration, the offset as well as the slope of the pH electrode calibration are recalculated by using two buffer solutions. This provides the best calibration results. The two buffer solutions must differ by at least 1 pH unit.

- **Tip:** The following description refers to the **Commands** dialog box. All commands and input fields for two-point calibration are also available on the PCM-3000 control panel (\rightarrow page 33).
- 1. Place the electrode in the flow cell and pump the first buffer solution through the system, or use the injection/flushing kit to directly inject the solution into the flow cell. If you are calibrating in an external vessel, immerse the electrode in the buffer solution. Stirring the electrode in the solution may speed up the process. In both internal and external calibration, wait for the displayed pH value (on the detector display or in Chromeleon) to stabilize.
- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 2. Select the **pHClearCalibrationPoints** command and click **Execute**.
- 3. Select the **pHSetCalibrationPoint1** parameter. In the pH box, enter the pH value of the buffer solution; for example, 7.0. Entering the temperature value in the **Temperature** box is only required if you are calibrating externally. If you do not enter a temperature value, Chromeleon uses the value measured by the temperature sensor in the flow cell.
- 4. Click **Execute**. The new calibration point is stored in Chromeleon.
- 5. Pump the second buffer solution through the system. If you are calibrating in an external vessel, rinse the electrode and then immerse it in the second buffer solution. Stirring the electrode in the solution may speed up the process. Again, wait for the displayed pH value (on the detector display or in Chromeleon) to stabilize.
- 6. Select the **pHSetCalibrationPoint2** parameter. In the **pH** box, enter the pH value of the buffer solution; for example, 4.0. Entering the temperature value in the **Temperature** box is only required if you are calibrating externally. If you do not enter a temperature value, Chromeleon uses the value measured by the temperature sensor in the flow cell.
- 7. Click **Execute**. The second calibration point is stored in Chromeleon.
- 8. Select the **pHTwoPointCalibration** command and click **Execute**. The new calibration values are sent to the monitor.
- 9. *When calibrating inside the pH flow cell:* Rinse the buffer solution from the HPLC system.

i Tip: You can repeat the measurement and re-enter the calibration points any number of times without changing the calibration. The new values will not be calculated and saved before the **pHTwoPointCalibration** command is executed.

To perform one-point calibration

During a one-point calibration, only the slope of the pH electrode calibration is recalculated. The used buffer solution must not have pH value 7.0 (< pH 6.0 or > pH 8.0, the greater the deviation from 7, the better the calibration).

- 1. Place the electrode in the flow cell and pump the buffer solution through the system. If you are calibrating in an external vessel, immerse the electrode in the buffer solution. Stirring the electrode in the solution may speed up the process. In both internal and external calibration, wait for the displayed pH value (on the detector display or in Chromeleon) to stabilize.
- 2. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 3. Select the **pHOnePointCalibration** command. In the **pH** box, enter the pH value of the buffer solution; for example, 4.0. Entering the temperature value in the **Temperature** box is only required if you are calibrating externally. If you do not enter a temperature value, Chromeleon uses the value measured by the temperature sensor in the flow cell.
- 4. Click **Execute**. The new slope value is sent to the monitor.
- 5. *When calibrating inside the pH flow cell:* Rinse the buffer solution from the HPLC system.

Error during calibration

If the calibration fails with an error, the calculated slope and/or offset values deviate considerably from the expected values (\rightarrow section 5.2.3). In this case the new values will not be stored. Check if you have entered the correct pH value for the calibration solution. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).

5.2.3 Monitoring pH Calibration

When the pH electrode ages, the zero point voltage shifts, and the slope of the calibration curve decreases. Therefore, Chromeleon provides functions for monitoring these calibration parameters, which function to detect electrode aging in time, such that the electrode can be cleaned or replaced. The limits for the shift of the zero point voltage (offset) and the slope can be determined by the user. For remedial action look up your exact error message in section 6.1 (\rightarrow page 49)

If the values calculated during pH calibration fall within the warning range, a warning is issued in the Chromeleon Audit Trail. If a value is outside of the defined limits, the calibration is invalid and the new values will not be stored.

Tip: The following description refers to the **Commands** dialog box. However, you can also enter the limits from the PCM-3000 control panel (**Limits** button, \rightarrow page 33).

To define the limits for the slope

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 2. Enter the preferred values for the **pHCalibSlope** parameter and click **Execute** for each value:
- 3. **LowerWarning**: Lower warning limit for the slope. A warning appears in the Chromeleon Audit Trail when a lower value is found during calibration.

LowerLimit: Lower limit for the slope. Calibration will fail if a lower value is found.

UpperWarning: Upper warning limit for the slope. A warning appears in the Chromeleon Audit Trail when a higher value is found during calibration.

UpperLimit: Upper limit for the slope. Calibration will fail if a higher value is found.

To define the limits for the offset

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 2. Enter the preferred values for the **pHCalibOffset** parameter and click **Execute** for each value:

LowerWarning: Lower warning limit for the offset. A warning appears in the Chromeleon Audit Trail when a lower value is found during calibration.

LowerLimit: Lower limit for the offset. Calibration will fail if a lower value is found.

UpperWarning: Upper warning limit for the offset. A warning appears in the Chromeleon Audit Trail when a higher value is found during calibration.

UpperLimit: Upper limit for the offset. Calibration will fail if a higher value is found.

5.2.4 pH Temperature Compensation

A temperature sensor measures the temperature of the eluent when it flows through the pH flow cell; when Chromeleon calculates the pH value, these temperature values are included in the calculation. If Chromeleon does not receive a signal from the temperature sensor, for example, because the sensor cable is not connected properly, it is not possible to start data acquisition. In this case, you can turn off temperature compensation in Chromeleon by using the **pHTempCompensation = Never** parameter. However, note that calculation of the pH value without considering the current temperature can lead to measuring errors if the temperature is not kept constantly at roughly 25°C.

5.3 Conductivity Measurement

Observe the notes on the conductivity flow cell (\rightarrow page 29).

As described in section 5.1.2 (\rightarrow page 34), the **Cond** channel is used to monitor the conductivity value in Chromeleon over a period of time. A program to monitor conductivity must include the **Cond.AcqOn** (start data acquisition) and **Cond.AcqOff** (end data acquisition) commands.

5.3.1 Conductivity Temperature Compensation

The conductivity of a solution increases as temperature increases, as the mobility of ions is temperature-sensitive. If the conductivity is used to monitor the eluent composition, it is required to convert all measured values to a defined reference temperature (usually 25°C), so that the values can be compared. How much the conductivity varies with the temperature (temperature coefficient) depends on the solution. Set the temperature coefficient for your solution before measurement is performed. A table with temperature coefficients of some common solvents can be found on page 67. The value of 2%/°C provides good results for most common solvents if you do not know the exact value. In addition, you can change the reference temperature.

The displayed conductivity value shows the conductivity of the solution at the defined reference temperature, usually 25°C. Therefore, the value does not change when temperature fluctuations occur (if the temperature coefficient is correct). However, you can turn off conductivity temperature compensation. In this case, the displayed conductivity value shows the actually measured conductivity (influence by the temperature). The resulting values are not only influenced by the solution composition, but also by the temperature.

To set the temperature coefficient

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level, if necessary.
- 2. Select the **CondTempCompensation** parameter and enter a value between -1.00 and 10.00%/°C in the box.
- 3. Click Execute.

To turn off temperature compensation

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level, if necessary.
- 2. Select the **CondTempCompensation** parameter, and select **Off** in the box.
- 3. Click Execute.
- **Tip:** When temperature compensation is turned off, signal changes must not be interpreted as changes in the composition of the solution, as the changes also reflect temperature fluctuations.

To change the reference temperature

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands. Select the **Expert** level, if necessary.
- 2. Select the **CondReferenceTemp** parameter and enter a value between 10.00 and 40.00°C.
- 3. Click Execute.

5.3.2 Calibrating the Conductivity Flow Cell

During calibration, the effective cell constant of the conductivity flow cell is recalculated. Calibration of the conductivity flow cell makes sense if very accurate measurements are required. In any case, calibration is required after cleaning the flow cell.

Calibration of the conductivity flow cell is performed by pumping a calibration solution with a defined conductivity through the flow cell. The conductivity is generally entered together with the temperature at which the conductivity applies. The currently measured eluent temperature is automatically used for temperature compensation by using the defined temperature coefficient (\rightarrow page 40).

Calibration is performed with the help of Chromeleon. There are two types of calibration: one-point with a single calibration solution, and two-point calibration with two calibration solutions with a different conductivity. For most solvents, one-point calibration provides optimum results. With high conductivity values (> 100 mS/cm), a two-point calibration may improve the accuracy.

Tip: The following descriptions refer to the **Commands** dialog box. All commands and input fields for calibration are also available on the PCM-3000 control panel (\rightarrow page 33).

To perform one-point calibration

A calibration solution with a known conductivity is required for one-point calibration. In general, choose the calibration solution in the usual range of your measurements.

- 1. Pump the calibration solution through the system, or use the injection/flushing kit to directly inject the solution into the flow cell. Wait for the displayed conductivity value (on the detector display or in Chromeleon) to stabilize.
- 2. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 3. Select the **CondOnePointCalibration** command. In the **Conductivity** box, enter the known conductivity value of the calibration solution, for example, 10.00 mS/cm. Then enter the related temperature value at which this conductivity applies, for example, 25°C, in the **Temperature** box.
- 4. Click **Execute**. The new value is sent to the monitor.

To perform two-point calibration

Two calibration solutions with a known conductivity are required for two-point calibration. The conductivity of the two calibration solutions must differ at least by a factor 2. It is important that you choose the calibration solutions in the usual range of your measurements.

- 1. Pump the first calibration solution through the system, or use the injection/flushing kit to directly inject the solution into the flow cell. Wait for the displayed conductivity value (on the detector display or in Chromeleon) to stabilize.
- 2. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level.
- 3. Select the **CondClearCalibrationPoints** command and click **Execute**.
- 4. Select the **CondSetCalibrationPoint1** parameter. In the **Conductivity** box, enter the known conductivity value of the calibration solution, for example, 100.00 mS/cm. Then enter the related temperature value at which this conductivity applies, for example, 25°C, in the **Temperature** box.
- 5. Click **Execute**. The new calibration point is stored in Chromeleon.
- 6. Pump the second calibration solution through the flow cell. Again, wait for the displayed conductivity value (on the detector display or in Chromeleon) to stabilize.
- Select the CondSetCalibrationPoint2 parameter. In the Conductivity box, enter the known conductivity value of the calibration solution, for example, 123.45 mS/cm. Then enter the related temperature value at which this conductivity applies, for example, 25°C, in the Temperature box.
- 8. Click **Execute**. The second calibration point is stored in Chromeleon.
- 9. Select the **CondTwoPointCalibration** command and click **Execute**. The new calibration values are calculated and stored in the flow cell.
- **Tip:** You can repeat the measurement and re-enter the calibration points any number of times without changing the calibration. The new values will not be calculated and saved before the Cond**TwoPointCalibration** command is executed.

Error during calibration

If the calibration fails with an error, the calculated effective cell constant deviates considerably from the nominal cell constant. In this case the new values will not be stored. Check if you have entered the correct conductivity value for the calibration solution. Clean the conductivity flow cell (\rightarrow page 60) and recalibrate. See also section 5.3.3.

5.3.3 Monitoring the Effective Cell Constant

The effective cell constant of a conductivity flow cell describes the sensitivity of the flow cell at a defined conductance value of the cell and should be in the range of the nominal cell constant after recalibration.

Therefore, Chromeleon provides functions for monitoring the deviation of this value from the nominal cell constant, which function to detect errors during calibration, such that the calibration can be repeated. Ideally, the effective cell constant and nominal cell constant are identical, in which case the **CellDeviation** is 100%. The limits for the allowed deviation can be defined by the user.

If the values calculated during calibration of the conductivity electrode fall within the defined warning range, a warning is issued in the Chromeleon Audit Trail. If a value is outside of the defined limits, the calibration is invalid and the new values will not be stored.

i Tip: The following description refers to the Commands dialog box. However, you can also enter the limits from the PCM-3000 control panel (Limits button, \rightarrow page 33).

To define the limits for the effective cell constant deviation

- 1. Open the **Commands** dialog box and go to the PCM-3000 commands (**UV_PCM**). Select the **Expert** level, if necessary.
- 2. Enter the preferred values for the **CellDeviation** parameter and click **Execute** for each value:
- 3. **LowerWarning**: Lower warning limit for the cell deviation. A warning appears in the Chromeleon Audit Trail when a lower value is found during calibration. This indicates a calibration error or aging and/or contamination of the flow cell.

LowerLimit: Lower limit for the cell deviation. Calibration will fail if a lower value is found. This indicates a calibration error or aging and/or contamination of the flow cell.

UpperWarning: Upper warning limit for the cell deviation. A warning appears in the Chromeleon Audit Trail when a higher value is found during calibration. This indicates a calibration error.

UpperLimit: Upper limit for the cell deviation. Calibration will fail if a higher value is found. This indicates a calibration error.

5.4 Noise Reduction (Response Time)

The response time is a measure of how quickly the monitor responds to a change in signal. It can be set separately for the pH and conductivity signals and independently of the Response Time of the detector signal.

As a rule, the Response Time should be very small or turned off. You can increase the Response Time to smooth the signal if strong noise occurs. However, remember that this will lead to a delay of the pH and conductivity measuring values when gradients change quickly.

The Response Time can be changed from the **Commands** dialog box (\rightarrow page 32) or, with automated control, directly in the program. Select the **pHResponseTime** parameter for the Response Time of the **pH** signal, and **CondResponseTime** for the Response Time of the **Cond** signal.

5.5 Changing the Detector Display

If a pH and conductivity monitor is installed in the detector, the values for the pH and conductivity will be displayed on the detector front panel display in place of the third and fourth UV-VIS channels. You can switch between this display mode and the standard mode with four detector channels at any time.



Fig. 19: Display of pH and conductivity (here: DAD-3000RS)

To change the detector display

A function key is provided on the detector display for toggling between the display modes "Standard" and "pH/Cond". White spots on the front panel mark the positions of the function keys.

- 1. To show the keys, touch the position of the utmost left spot on the display with a menu pen (part no. 6300.0100). The menu pen is included in the UltiMate 3000 autosampler accessories. Depending on the display mode you are currently using, the second function key shows "Standard" or "pH/Cond".
- 2. Touch the white spot under the second function key to toggle between the two display modes.

	DAD-3	000RS	
1:	220 nm	2:	254 nm
3:	272 nm	4:	520 nm
Menu	pH/Cond		

Fig. 20: Function keys (here: DAD-3000RS)

5.6 Shutting Down the Monitor

Observe the following precautions before interrupting the operation or before shipping the monitor:

• Remove the pH electrode from the flow cell or the offline storage container. Clean the pH electrode (→ page 56). Fill the original cap with a solution of pH 4 buffer and potassium chloride (100 g KCl in 1 L of pH4 buffer based on potassium hydrogen phthalate (KHP)) and place it on the electrode. To protect the pH electrode during shipment, place it in a separate box within the shipping package.

When storing the pH electrode for a longer period of time, cool the electrode to extend its lifetime. The electrode must not be stored at temperatures below -11°C, as this may damage the glass.

- Flush the flow cells to remove buffers and salts. You can flush the flow cells by using the optional syringe injection/flushing kit (part no. 6078.4200).
- Clean the pH flow cell (→ page 58) and place the dummy electrode in the flow cell to seal it.
- Clean the flow cells and the flow cell carrier from the outside.
- If possible, ship the monitor in the original shipping container. For warranty information, see the warranty statement in the terms of sale.

5.7 Routine and Preventive Maintenance

The monitor is made of high-quality components and materials to minimize maintenance requirements. All surfaces are well resistant to weak acids, alkali, and organic solvents. Nevertheless, immediately wipe up all liquids spilled onto the detector surface by using lint-free cloth or paper. If surfaces are exposed for longer periods, these liquids can cause damage.

Perform the maintenance procedures listed in the table below at regular intervals to ensure optimum performance and maximum uptime of the monitor. The exact maintenance schedule for the monitor will depend on a number of factors.

Frequency	What you should do
Daily	Inspect the fluid connections and flow cells for indications of leakage or restrictions.
	When buffer solutions are used, flush the system thoroughly after use. Use a solvent that does not contain buffers or salts.
Regularly	Check the drain tube connected to the drain port (\rightarrow page 25). Verify that the tubing is unclogged and is routed below the drain port. Check the volume of the liquid in the waste container and empty as needed.
When required	Clean the pH electrode (\rightarrow page 56). Clean the conductivity flow cell (\rightarrow page 60). Exchange the pH electrode (\rightarrow page 57).
Annually	Have a service representative check the detector once a year to prevent contamination and excessive wear.

I Tip: Chromeleon supports functions for monitoring the pH electrode (\rightarrow page 39).

6 Troubleshooting

If a fault or error is detected during the operation of the monitor, a message appears in the Chromeleon Audit Trail. For a description of the messages and recommended courses of action, see section 6.1.

If you are unable to eliminate a problem following the instructions given here, contact Thermo Fisher Scientific Service for Dionex HPLC Products.

6.1 Messages in the Chromeleon Audit Trail

The following table lists the most important messages related to the pH and conductivity monitor along with recommended remedial actions. In addition to the messages in the table, other messages may appear. In this case, write down the exact wording of the message and contact Thermo Fisher Scientific Service for Dionex HPLC Products if you are unable to eliminate the problem.

Message	Remedial Action
Calibration points with pH value x.x and pH value x.x are not accepted. The pH values of the calibration points must have a difference of at least 1.	The error occurs during two-point calibration. The pH values of the used buffers must differ by at least 1.
Cannot change calibration point because the conductivity parameter is missing.	The error occurs during calibration. Enter the conductivity value of the solution to perform calibration.
Cannot change calibration point because the pH value is missing.	The error occurs during calibration. Enter the pH value of the buffer to perform calibration.
Cannot determine the pH value because of missing temperature.	Check the pH temperature sensor connection (TEMP) at the detector rear panel (\rightarrow Fig. 6). The temperature sensor may be defective - please contact Service. Turn off temperature compensation if you want to monitor pH anyway (\rightarrow page 40).
Cannot determine the conductivity value because of missing temperature.	Check the conductivity temperature sensor connection (COND) at the detector rear panel (\rightarrow Fig. 6). The temperature sensor may be defective - please contact Service. Turn off temperature compensation if you want to monitor conductivity anyway (\rightarrow page 40).
Cannot execute one-point calibration because the pH value is missing.	The error occurs during one-point calibration. Enter the pH value of the buffer to perform calibration.
Cell deviation xx % is above xx %. Please check the conductivity sensor.	This warning is issued during calibration, if the cell deviation exceeds the defined upper warning limit (CellDeviation.UpperWarning), see page 44.
Cell deviation xx % is below xxx %. Please check the conductivity sensor.	This warning is issued during calibration, if the cell deviation is below the defined lower warning limit (CellDeviation.LowerWarning), see page 44.

Message	Remedial Action
Cell deviation xxx % must have a value between xx % and xxx %. Please check the conductivity sensor.	The error occurs during calibration, if the cell deviation is outside the defined limits (CellDeviation.UpperLimit or CellDeviation.LowerLimit), see page 44.
Conductivity flow cell not available. Please check the conductivity flow cell and conductivity flow cell connection.	No conductivity flow cell is connected. Check the conductivity temperature sensor connection (COND) at the detector rear panel (\rightarrow Fig. 6).
Conductivity two-point calibration needs two calibration points with different conductivity values.	The error occurs during two-point calibration. The conductivity of the calibration solutions used must differ at least by a factor 2.
Internal pH sensor calibration is not possible because the temperature cannot be measured.	The new calibration values cannot be calculated, because the temperature signal is missing. Check the pH temperature sensor connection (TEMP) at the detector rear panel (\rightarrow Fig. 6). The temperature sensor may be defective - please contact Service. Perform an external calibration (manual temperature input).
Offset xxx mV is above xxx mV. Please check the pH sensor.	This warning is issued during calibration, if the offset exceeds the defined upper warning limit (pHCalibOffset.UpperWarning), see page 39. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).
Offset xxx mV is below xxx mV. Please check the pH sensor.	This warning is issued during calibration, if the offset is below the defined lower warning limit (pHCalibOffset.LowerWarning), see page 39. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).
Offset xxx mV must have a value between xxx mV and xxx mV. Please check the pH sensor.	The error occurs during calibration, if the offset is outside the defined limits (pHCalibOffset.UpperLimit or pHCalibOffset.LowerLimit), see page 39. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).
One-point calibration must not use a pH value 7.	The error occurs during one-point calibration. The pH of the buffer must not be 7 (recommended: < pH6 or > pH 8).
PCM-3000 board defective.	Verify that the extension board is installed correctly. The board may be defective. Contact Service.
PCM-3000 configured, but not present. Please check configuration using the Server Configuration program.	A PCM-3000 was configured in the Chromeleon Server Configuration, but is not installed. Correct the configuration $(\rightarrow page 23)$.
PCM-3000 board not available.	Verify that the extension board is seated properly and the screws tightened.
PCM-3000 not configured, but present. Please check configuration using the Server Configuration program.	A PCM-3000 was installed, but not configured in the Chromeleon Server Configuration. Correct the configuration (\rightarrow page 23).
pH flow cell not available.	No pH flow cell is connected. Check the pH temperature sensor

Message	Remedial Action
Please check the pH flow cell and pH flow cell connection.	connection (TEMP) at the detector rear panel (\rightarrow Fig. 6). Chromeleon requires the pH flow cell to save the calibration values.
pH sensor provides an overloaded signal. Please check the pH sensor connection and the pH sensor.	Check the pH electrode connection (BNC connector) at the detector rear panel (\rightarrow Fig. 6).
Slope must have a value between xx % and xxx %. Please check the pH sensor.	The error occurs during calibration, if the slope is outside the defined limits (pHCalibSlope.UpperLimit or pHCalibSlope.LowerLimit), see page 39. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).
Slope xx % is above xx %. Please check the pH sensor.	This warning is issued during calibration, if the slope exceeds the defined upper warning limit (pHCalibSlope.UpperWarning), see page 39. Check if you have entered the correct pH value for the buffers. Only use the pH electrode that is shipped with the monitor.
Slope xx % is below xx %. Please check the pH sensor.	This warning is issued during calibration, if the slope is below the defined lower warning limit (pHCalibSlope.LowerWarning), see page 39. Check if you have entered the correct pH value for the buffers. Clean the pH electrode (\rightarrow page 56). The pH electrode may have to be replaced (\rightarrow page 57).
Temperature unit of the pH flow cell is not connected.	Check the pH temperature sensor connection (TEMP) at the detector rear panel (\rightarrow Fig. 6). Turn off temperature compensation if you want to monitor pH anyway (\rightarrow page 40).
Temperature unit of the conductivity flow cell is not connected.	Check the conductivity temperature sensor connection (COND) at the detector rear panel (\rightarrow Fig. 6).
The conductivity calibration needs the nominal conductivity and the related temperature.	The error occurs during calibration, of one of the two values (known conductivity of the calibration solution or related temperature value) is missing. Make sure to enter both values.
The conductivity of calibration points must differ at least by factor 2.	The error occurs during calibration, if the conductivity of the two calibration solutions does not differ by at least factor 2.
The acquisition is stopped because the connection to pH flow cell is lost.	Check the pH temperature sensor connection (TEMP) at the detector rear panel (\rightarrow Fig. 6). Restart data acquisition after you have eliminated the error.

6.2 Operating Problems

The following table provides information about common operating problems that might occur with an UltiMate 3000 system and lists probable causes, as well as remedial actions. For more information, also see the manuals for the other modules of the UltiMate 3000 system.

Problem	Probable Cause	Remedial Action
Problems during control under Chromeleon	There is no connection between the detector and the Chromeleon computer.	Check the USB cable and connection from the detector to the computer.
	The USB port on the computer is not ready for operation.	Check the USB port on the computer. It must comply to the USB 2.0 standard when an MWD or DAD is used.
	The sensor cables are not properly connected.	Check that the cables are connected properly to the detector.
pH value does not change	The pH electrode is defective.	Exchange the pH electrode $(\rightarrow page 57)$.
Monitor shows slow response to changes in pH	The pH electrode is contaminated.	Clean the pH electrode (\rightarrow page 56). Exchange the pH electrode (\rightarrow page 57).
The pH value is not correct or instable	The pH electrode cable is not properly connected.	Check that the cable is connected properly to the detector.
	The pH electrode is not calibrated correctly, or not calibrated in the measuring range.	Calibrate the pH electrode (→ page 36). Make sure that temperature values are stable. Choose the buffer solution(s) in the usual range of your measurements.
	The pH electrode is contaminated.	Clean the pH electrode $(\rightarrow page 56)$.
	Note that stable pH measurements are not possible for organic solvents such as ethanol, methanol, or acetonitrile.	Do not use organic solvents if you want to measure pH. Consider deactivating the pH electrode signal.
	Air bubbles in the pH flow cell (e.g.; if the injection/flushing kit is used)	Loosening the lock nut to let the air escape, then retighten.
	The electrode is not seated properly.	Check that the electrode is properly seated and that the lock nut is hand-tightened.
Wrong conductivity value	The flow cell is not correctly calibrated.	Calibrate the conductivity flow cell $(\rightarrow \text{ page 42})$. Verify that the calibration solution was prepared correctly.

Problem	Probable Cause	Remedial Action
The conductivity value is not correct or instable	Pressure or flow fluctuation occurs in the pump.	Purge the pump; check general function (\rightarrow Pump Manual).
	Steel capillaries are used.	Verify that only PEEK capillaries are used.
	The flow cell was not calibrated in the measuring range.	Choose the calibration solution(s) in the usual range of your measurements.
	The conductivity flow cell cable is not properly connected.	Check that the cable is connected properly to the detector.
	The conductivity flow cell is contaminated.	Clean the flow cell (\rightarrow page 60).
	The measured values reflect temperature fluctuations.	Verify that temperature compensation is turned on and that the correct temperature coefficient is used (\rightarrow page 40).
Conductivity value decreases or increases with same solvent over time	The conductivity flow cell is contaminated.	Clean the flow cell (\rightarrow page 60).
	The measured values reflect temperature fluctuations.	Verify that temperature compensation is turned on and that the correct temperature coefficient is used (\rightarrow page 40).
Baseline drift or noise in the conductivity signal	Pressure or flow fluctuation occurs in the pump.	Purge the pump; check general function (\rightarrow Pump Manual).
	Air bubbles in the conductivity flow cell	Check all fluid connections for tightness. Degas the mobile phase and/or install a restrictor at the conductivity flow cell outlet.
	The conductivity flow cell is contaminated.	Clean the flow cell (\rightarrow page 60).
	The eluents are degraded or inhomogeneous.	Before you start an analysis, homogenize eluents already in their reservoir. Use fresh solvent and check the eluent filter frits. In aqueous solvents, growth of microorganisms is possible.
	The column is contaminated.	Clean or replace the column.
	The selected response time is too small.	Select a higher response time $(\rightarrow page 45)$.
Ghost peaks in the conductivity signal	The solution contains a charged sample, e.g., a protein.	By selecting a suitable higher response time, peaks in the conductivity signal may be reduced $(\rightarrow page 45)$.

Problem	Probable Cause	Remedial Action
Negative peaks in the conductivity signal	Air bubbles in the conductivity flow cell	Check all fluid connections for tightness. Degas the mobile phase and/or install a restrictor at the conductivity flow cell outlet.

7 Service

7.1 General Notes and Safety Precautions

The following sections describe all procedures for the instrument that the user may perform. All other maintenance and service procedures must be performed by Thermo Fisher Scientific service personnel.

Remove the hood from the flow cell carrier for all service work. To protect the components during operation, reinstall the hood afterward.

Warning: The fluid components of the device may be filled with solvents that are harmful to health. Wear appropriate personal protective equipment. Rinse the fluid components with an appropriate solvent to remove harmful substances.

For information about the proper handling of a particular substance and for advice on specific hazards, refer to the material safety data sheet for the substance you are using. Observe the guidelines of Good Laboratory Practice (GLP).

Avertissement: Les composants fluidiques de l'instrument peuvent être remplis de solvants nocifs. Portez l'équipement de protection personnel approprié. Rincez les composants fluidiques avec un solvant approprié afin d'éliminer les substances nocives.

Pour les informations sur la manipulation correcte des composés et des recommandations pour les situations de risque spécifiques, veuillez consulter la fiche de données de sécurité des substances que vous utilisez. Veuillez respecter des directives des Bonnes Pratiques de Laboratoire (BPL).

Before starting maintenance or service procedures, observe the following precautions:

- For all service and repair procedures, observe all precautionary statements provided in these operating instructions.
- Use only the original spare parts and accessories authorized for the device by Thermo Fisher Scientific.
- Before returning the monitor for repair, contact Thermo Fisher Scientific Service for Dionex HPLC Products. An RMA (Return Material Authorization) number is required to track your instrument. Always use the original shipping container.

For instructions on shutting down the monitor, see page 47.

7.2 pH Electrode

7.2.1 Cleaning the pH Electrode

Occasionally, eluent or sample compounds may deposit on the pH electrode, thus resulting in wrong values and a reduced slope of the calibration curve.

To clean the pH electrode, immerse it in a vessel filled with 5-10% hydrochloric acid (HCl). Rinse the pH electrode in water afterward.



ning: To avoid damage to the skin and eyes, wear appropriate protective clothing and goggles when using hydrochloric acid.

Avertissement: Afin d'éviter des brûlures cutanées ou oculaires, portez des vêtements de protection appropriés et des lunettes de protection lorsque vous utilisez de l'acide chlorhydrique.

I Tip: Never try to clean the pH electrode with abrasives, a brush, or similar.

If cleaning the pH electrode does not help, replace the electrode.

7.2.2 Reconditioning the pH Electrode

When the pH electrode ages, reconditioning the electrode may help. The substances used for reconditioning are caustic and partially very toxic. Therefore, Thermo Fisher Scientific recommends replacing the electrode rather than trying to recondition it.

This section describes various chemical treatments. Start with the first treatment and then check if the performance has improved. Only then continue with the further treatments. Note that these treatments attack the pH glass. In some cases, the performance may deteriorate after the treatment. If performance was not improved, the pH electrode must be replaced.

Warning: To avoid damage to the skin and eyes, wear appropriate protective clothing and appropriate goggles when using the substances mentioned below.

Avertissement: Afin d'éviter des brûlures cutanées ou oculaires, portez des vêtements de protection appropriés et des lunettes de protection appropriées lorsque vous utilisez les substances mentionnées cidessous.

Warning: Ammonium bifluoride and hydrofluoric acid are caustic and very toxic and should only be used by qualified personnel. Carefully read the manufacturer's safety data sheet. In case of contact or inhalation, seek medical aid immediately.



Avertissement: Hydrogénofluorure d'ammonium et acide fluorhydrique sont des solutions corrisives et très toxiques. Ces substances ne doivent être utilizer que par du personnel qualifié. Observez la Fiche de Données de Sécurité du producteur. En cas de contact ou inhalation, consultez immédiatement un médecin.

- Immerse the electrode tip in 0.1 M hydrochloric acid (HCl) for 15 seconds, rinse in • water, and then immerse the electrode in 0.1 M sodium hydroxide (NaOH) for 15 seconds. Again, rinse the electrode in water. Repeat three times.
- Immerse the electrode tip in a 20% solution of ammonium bifluoride ((NH₄)HF₂) for 2 to • 3 minutes, and rinse the electrode in water afterward.
- Immerse the electrode tip in 5% hydrofluoric acid (HF) for 10-15 seconds, and • thoroughly rinse the electrode in water afterward. Quickly rinse the electrode in 5 M hydrochloric acid (HCl) and again rinse thoroughly in water.

7.2.3 Replacing the pH Electrode

Part no.	Description
6082.2020	pH electrode for PCM-3000 incl. o-rings for pH flow cell

If the pH electrode does not respond or slowly respond to changes in pH, or if the slope of the calibration curve is reduced, this indicates that the electrode has aged. If cleaning does not improve the results, the electrode must be replaced. Observe the general guidelines for pH electrodes (\rightarrow page 28).

- Disconnect the pH electrode cable (BNC connector) from the BNC connector on the 1. detector rear panel.
- 2. Loosen the lock nut of the mounted pH electrode and remove the electrode from the flow cell.
- 3. Verify that the flow cell is free of salt deposits, and clean the flow cell if necessary $(\rightarrow \text{ page 58})$ before you place the new electrode in it, otherwise the new electrode may be damaged.
- 4. Remove the new pH electrode from its packaging, then carefully remove the soaker cap that is filled with liquid. Examine the electrode for damage.
- 5. Thread the cable of the new pH electrode through the lock nut as shown in Fig. 10 $(\rightarrow page 22).$
- 6. Check that the o-rings (\rightarrow Fig. 18, page 36) are still in their place in the flow cell, and push them back into their grooves (using gloves) if necessary. (A set of replacement orings is shipped with the new pH electrode).

- 7. Place the new pH electrode in the pH flow cell and tighten the lock nut hand-tight. The nut and the electrode must be tightened such that they can no longer be moved.
- 8. Connect the pH electrode cable (BNC connector) to the BNC connector of the PCM-3000 extension board on the detector rear panel. The connector has a slot to be aligned with the pin on the female connector. Turn the connector clock-wise to lock it.
- 9. Calibrate the pH electrode (\rightarrow page 36).

7.3 pH Flow Cell

7.3.1 Cleaning the pH Flow Cell

Occasionally, eluent or sample compounds may deposit on the cell walls or windows, thus resulting in a higher noise level, high backpressure, or wrong values. Cleaning may help to remove deposits. Observe the general notes on the pH flow cell (\rightarrow page 28).

Tips: Do not try to clean the flow cell mechanically with abrasives, a brush, or similar. If necessary, use a soft, lint-free tissue.

You can flush the flow cell in the HPLC system or using the optional syringe injection/flushing kit (part no. 6078.4200).

Complete the following steps:

- 1. Turn off the detector by pressing the power switch on the rear of the detector.
- 2. Remove the pH electrode from the flow cell and install the dummy electrode in the flow cell.
- 3. Flush the flow cell with deionized water. If necessary, fill the flow cell with 1 M NaOH and allow it to take effect for 30 minutes.



STOP

To avoid damage to the skin and eyes, wear appropriate protective clothing and goggles when using NaOH.

Avertissement: Afin d'éviter des brûlures cutanées ou oculaires, portez des vêtements de protection appropriés et des lunettes de protection lorsque vous utilisez de la soude caustique.

4. Flush the flow cell with a buffer solution.

If flushing does not help, clean the pH flow cell externally:

- 1. Remove the inlet and outlet capillaries on the flow cell.
- 2. Remove the pH flow cell from its holder.

- 3. Immerse two thirds of the pH flow cell in a suitable cleaning solution (e.g., heated HPLC water). Do not immerse the part where the cable is attached.
- If cleaning the flow cell does not eliminate the problem, install a new flow cell (→ page 59).

7.3.2 Replacing the pH Flow Cell

Part no.	Description
6082.2030	pH flow cell for PCM-3000, 28 µL, PEEK

Observe the general notes on the pH flow cell (\rightarrow page 28). No tools are required to remove and install a flow cell.

1 Tips: The pH flow cell is symmetrical, however, when connecting capillaries, always connect the inlet capillary at the bottom connection and the outlet capillary at the top connection. This ensures that air in the flow cell can escape. Inlet and outlet must not be exchanged.

When replacing a flow cell, prevent dust and other contamination from entering the open capillary connections.

- 1. Remove the pH electrode or dummy electrode from the flow cell.
- 2. Remove the inlet and outlet capillaries on the flow cell.
- Move the strain relief away from the cable guides, so that you can remove the temperature sensor cable of the pH flow cell from the guide (→ Fig. 11 right). Disconnect the temperature sensor cable from the TEMP connector on the detector rear panel (→□Fig. 6, page 19).
- 4. Remove the pH flow cell from its holder.
- 5. Place the new pH flow cell in the holder until it locks in place. To do so, hold the flow cell at a correct angle (capillary outlets pointing to the top and bottom).
- 6. Loosen the lock nut of the new pH electrode and remove the dummy electrode from the flow cell. Check that the o-rings (→ Fig. 18, page 36) are still in their place, and push them back into their grooves (using gloves) if necessary. (A set of replacement o-rings is available under part no. 6082.2035.)
- 7. Verify that the new pH flow cell is free of dust and clean it, if necessary (\rightarrow page 58).
- 8. Route the temperature sensor cable below the flow cell holder and then through the cable guide, and then move the strain relief back into its closing position (\rightarrow Fig. 11 left, page 22). Then connect the cable to the TEMP connector of the PCM-3000 extension board on the detector rear panel; note the arrow that is visible on the upper side of the connector when connected.

- 9. Connect the inlet capillary to the bottom connection of the flow cell.
- 10. Connect the outlet capillary to the top side connection of the flow cell.
- 11. Place the pH electrode in the pH flow cell and calibrate the electrode (\rightarrow page 36).

7.4 Conductivity Flow Cell

7.4.1 Cleaning the Conductivity Flow Cell

Occasionally, eluent or sample compounds may deposit on the cell walls or windows and lead to a change in sensitivity, shifted baseline, poor linearity, instable measurements, or a higher noise level. Cleaning may help to remove deposits. Observe the general notes on the conductivity flow cell (\rightarrow page 29).

Tip: You can flush the flow cell in the HPLC system or using the optional syringe injection/flushing kit (part no. 6078.4200).

Complete the following steps:

1. If necessary, fill the flow cell with 1 M NaOH and allow it to take effect for 30 minutes.

Warning:

To avoid damage to the skin and eyes, wear appropriate protective clothing and goggles when using NaOH.

STOP

Avertissement: Afin d'éviter des brûlures cutanées ou oculaires, portez des vêtements de protection appropriés et des lunettes de protection lorsque vous utilisez de la soude caustique.

- 2. Flush the flow cell with deionized water (apprx. 100 mL).
- 3. Calibrate the conductivity flow cell (\rightarrow page 42).

After the cleaning, the conductivity flow cell may have to be recalibrated at shorter time intevals.

When the flow cell is blocked, you can try to to unblock it by pushing a thin needle through the flow cell

If cleaning the flow cell does not eliminate the problem, install a new flow cell (\rightarrow page 61).

7.4.2 Replacing the Conductivity Flow Cell

Part no.	Description
6082.2060	Conductivity flow cell for PCM-3000, 21 µL, titanium

Observe the general notes on the conductivity flow cell (\rightarrow page 29).

i Tip: When connecting capillaries, always connect the inlet capillary at the bottom connection and the outlet capillary at the top connection. Inlet and outlet must not be exchanged.

When replacing a flow cell, prevent dust and other contamination from entering the open capillary connections.

- 1. Remove the inlet and outlet capillaries on the flow cell.
- 2. Loosen the screw of the clamp that fixes the flow cell cable (\rightarrow Fig. 21) by using a screwdriver (TX10, included in accessories).



Fig. 21: Remove the clamp for the conductivity flow cell cable

- Move the strain relief away from the cable guides, so that you can remove the temperature sensor cable of the conductivity flow cell from the guide (→ Fig. 11 right, page 22). Disconnect the cable from the COND connector on the detector rear panel (→□Fig. 6, page 19).
- 4. Loosen the two screws that attach the flow cell to the holder by using a screwdriver (TX10, included in accessories) and remove the flow cell from the holder (→□Fig. 22).



Fig. 22: Remove the conductivity flow cell

- 5. Place the new conductivity flow cell in the holder with the four screws facing downward. Hold the flow cell while you tighten the two screws to mount it in the holder.
- 6. Route the cable of the new flow cell though the clamp and fix the clamp to the flow cell carrier as shown in Fig. 21. Then route the cable through the cable guide and move the strain relief back into its closing position (\rightarrow Fig. 11 left, page 22).
- 7. Connect the cable to the COND connector of the PCM-3000 extension board on the detector rear panel; note the arrow that is visible on the upper side of the connector when connected.
- 8. Connect the inlet capillary to the bottom connection of the flow cell.
- 9. Connect the outlet capillary to the top side connection of the flow cell.

Thermo Fisher Scientific recommends calibrating the conductivity flow cell (\rightarrow page 42).

8 Technical Information

General technical information

Data collection rate:	10 Hz		
Control:	All parameters and functions software-controlled		
GLP features:	In Chromeleon: All system parameters are recorded in the audit trail.		
Power requirements:	5 V DC, max. 1,5 W (internally via DAD/MWD/VWD)		
Environmental conditions:	Temperature: Air humidity: Overvoltage category:	Indoor use 10 °C to 35 °C (50 to 95°F) 80% relative humidity, non-condensing II 2	
Dimensions (h × w × d):	18 x 14 x 29 cm (7.1 x 5.5 x 11.4 in) (when placed stand-alone) 18 x 11 x 22 cm (7.1 x 4.3 x 8.7 in) (when mounted on detector)		
Weight:	Approx. 1.5 kg (approx. 3.3 lbs)		

pН

Measuring range:	pH 0 – 14 (specifications valid between pH 2-12)
Accuracy:	± 0.1 pH in a range of +20 to +30°C ± 0.35 pH in a range of +30 to +40°C
Drift:	Max. deviation: 0.1 pH/10h
Flow cell/pH electrode:	Cell volume: 28 μL wetted materials: PEEK, Epoxy, glass, HDPE, EPDM max. flow rate: 100 mL/min pressure limit: 0.7 MPA (100 psi) Temperature range: +4 to +50°C (liquid) Identification of flow cell type and serial number and storage of calibration data by means of a built-in ID chip in the flow cell.
Temperature compensated:	Yes, automatically by Chromeleon

Conductivity

Measuring range:	1 µS/cm to 1 S/cm		
Flow cell:	Cell constant: 50 /cm (\pm 20 /cm)		
	Cell volume: 21 μ L (fitting to fitting, active volume 2 μ L)		
	wetted materials: PEEK, titanium		
	max. flow rate: 100 mL/min		
	pressure limit: 5 MPA (725 psi)		
	Temperature range: +4°C to +50°C (liquid)		
	Identification of flow cell type and serial number and storage of calibration data by means of a built-in ID chip in the flow cell.		
Accuracy:	\pm 2% of the calibration range with full-scale output in the range of 1µS/cm-100mS/cm with 1-point calibration,		
	\pm 2% of the calibration range with full-scale output in the range of 1µS/cm-200mS/cm with 2-point calibration,		
	or $\pm 10 \ \mu$ S/cm, whichever is greater		
	KCL in HPLC grade water, 1 mL/min, 25°C		
Temperature compensated:	Yes, automatically by Chromeleon		

Technical information: October 2013.

All technical specifications are subject to change without notice.

9 Accessories, Consumables, and Spare Parts

Accessories, spare parts, and consumables for the module are always maintained at the latest technical standard. Therefore, part numbers are subject to alteration. However, updated parts will always be compatible with the parts they replace. The part number always refers to the packing unit. For more information, contact the Thermo Fisher Scientific sales organization for Dionex HPLC Products.

9.1 Standard Accessories

The following accessories are shipped with the monitor. (The list is subject to change without notice).

Description	Part No.	Qty in the accessories kit
Accessories of PCM-3000, including:		
Flexible hose (11.4 mm OD x 8.4 mm ID)	Included in 6040.0005 (drain system kit)	1.5 m
Silicone tubing (2.80 mm O.D. x 1.30 mm I.D.)	6007.9100	1.5 m
Torx® screw	-	2
Torx offset screwdriver, size TX20	Included in 6040.0010	1
Torx offset screwdriver, size TX10	Included in 6040.0010	1
Lock nut for pH flow cell	6082.2043	1

9.2 Optional Accessories

Description	Part no.	Note
Menu pen	6300.0100	
Flow cell syringe injection/flushing kit	6078.4200	Includes a syringe and capillaries for direct injection into a flow cell.

9.3 Consumables and Spare Parts

Description	Part no.
Capillary (PEEK, 1/16" x 0.25 mm OD x ID, 1 m)	6251.6001
Capillary (PEEK, 1/16" x 0.75 mm OD x ID, 1 m)	2251.6003
Conductivity flow cell for PCM-3000, 21 µL, titanium	6082.2060
Drain system kit for UltiMate 3000 systems	6040.0005
Dummy electrode for PCM-3000	6082.2042
Flow cell syringe injection/flushing kit	6078.4200
Lock nut for pH flow cell	6082.2034
O-rings for pH flow cell	6082.2035
Offline storage container for pH electrode	6082.2050
pH electrode for PCM-3000 incl. o-rings for pH flow cell Note: limited shelf life	6082.2020
pH flow cell for PCM-3000, 28 µL, PEEK	6082.2030
RheFlex [®] fittings (PEEK, 1/16", finger-tight), 10 pieces	6000.0012
Silicone tubing (2.80 mm O.D. x 1.30 mm I.D.)	6007.9100
Torx offset screwdriver kit, sizes TX10 and TX20	6040.0010

10 Appendix: Temperature Coefficients of Common Solvents

How much the conductivity varies with the temperature (temperature coefficient) depends on the solution. The value of 2%/°C for the temperature coefficient provides good results for most common buffer solutions if you do not know the exact value for the solution (\rightarrow page 40).

Table 1 lists the temperature coefficients of common solvents overr the indicated temperature range.

Solvent	Temperature coefficient in %/°C	Temperature range	Reference temperature
NaCl 1 mol/L	2.01	18-25°C	25°C
KCl 0,001 mol/L	1.94	18-25°C	25°C
KCl 0,01 mol/L	1.90	18-25°C	25°C
KCl 0,01 mol/L	1.87	18-25°C	25°C
KCl 1 mol/L	1.74	18-25°C	25°C

Table 1: Temperature coefficients of common solvents in $\%^{\circ}C$

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