Thermo Scientific Dionex UltiMate 3000 Series

Electrochemical Detector

ECD-3000RS

Operating Instructions

(Original Operating Instructions)

Revision: 1.0

Date: December 2012

Doc. No. 4820.7001

CE

Declaration of Conformity

(Original Declaration of Conformity)

Product: Thermo Scientific Dionex UltiMate 3000 - Detector

Type: **ECD-3000RS**

Dionex Softron GmbH herewith declares conformity of the above products with the respective requirements of the following regulations:

- Low-Voltage Directive 2006/95/EC
- EMC Directive 2004/108/EC

The electrical safety of the products was evaluated based on the following standard:

- DIN EN 61010-1:2010
  Safety requirements for electrical equipment for measurement, control and laboratory use, Part 1: General Requirements

The Electromagnetic Compatibility (EMC) of the products was evaluated based on the following standard:

- DIN EN 61326: 2006
  Electrical equipment for measurement, control and laboratory use
  EMC Requirements

Responsible for the technical CE documentation is the manufacturer (see further down).

This declaration is issued for the manufacturer

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by the President, Dr. Peter Jochum.

November 15, 2012

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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. However, in order to obtain a full understanding of the detector, Thermo Fisher Scientific recommends that you review the manual thoroughly before beginning operation.

Almost all descriptions in the manual apply to the ECD-3000RS detector of the Thermo Scientific Dionex™ UltiMate™ 3000 series. Therefore, the term "the detector" is used throughout the manual. If some detail applies to only one detector version, the version is identified by name. The same applies to the descriptions of the Viper™ capillary connections throughout this manual. They apply also to the nanoViper™ capillary connections if not otherwise stated.

Notes: The detector configuration may vary. Therefore, not all descriptions necessarily apply to your particular detector.

It may happen that the representation of a component in this manual is different from the real component. However, this does not influence the descriptions.

The descriptions in this manual refer to firmware version 1.01 and Chromeleon™ 6.80 Service Release 12. If you want to operate the detector with Chromeleon 7, note the information on page 24.

This manual is provided "as is". All technical specifications and programs have been developed with utmost care. But the information contained in this manual should not be construed as a commitment by Thermo Fisher Scientific. Thermo Fisher Scientific assumes no responsibility for any errors that may appear in this document that is believed to be complete and accurate at the time of publication and, in no event, shall Thermo Fisher Scientific be liable for incidental or consequential damages in connection with or arising from the use of this document. We appreciate your help in eliminating any errors that may appear in this document.

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1.2 Safety Information

The CE Mark label and cTUVus Mark safety label on the instrument indicate that the detector is compliant with the related standards (→ page I).

1.2.1 Symbols on the Instrument and in the Manual

The table shows the symbols used on the instrument:

Symbol	Description
~	Alternating current—Courant alternatif
	Power supply is on (-)—L'instrument est mis sous tension (-) and Power supply is off (O)—L'instrument est mis hors tension (O)
	Protective grounding—Mise à la terre de protection
	Refer to the Operating Instructions to prevent risk of harm to the operator and to protect the instrument against damage. Référez-vous à ce manuel pour éviter tout risque de blessure à l'opérateur et/ou protéger l'instrument contre tout dommage
	Label according to the "Measures for Administration of the Pollution Control of Electronic Information Products" (China RoHS) guideline Étiquette "Measures for Administration of the Pollution Control of Electronic Information Products" (China RoHS)
	WEEE (Waste Electrical and Electronic Equipment) label—For more information, see the WEEE Information section in the "Installation and Qualification Documents for Chromatography Instruments" binder. Étiquette WEEE (Waste Electrical and Electronic Equipment)—Pour plus d'informations, référez-vous au chapitre WEEE Information dans le classeur "Installation and Qualification Documents for Chromatography Instruments"

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

Tip: Indicates general information, as well as information intended to optimize the performance of the instrument.

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.

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 General Safety Precautions

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

Tips: Before initial operation of the detector, make sure that you are familiar with the contents of this manual.

Observe any warning labels on the device and see the related sections in these *Operating Instructions*.

To avoid the possibility of personal injury and damage to the instrument, observe the following general safety precautions when operating the detector or carrying out any maintenance work:

- Install the HPLC system in a well-ventilated laboratory. If the mobile phase includes volatile or flammable solvents, do not allow them to enter the workspace.
- For minimum interference effects, all components of the analytical system should be connected to the same mains output (same phase).
- When lifting or moving the detector, always lift by the bottom or sides of the instrument. Do not lift the detector by the front panel door. This may damage the door.
- The open front panel door is not designed to carry weight. Therefore, you should not place any objects on the open door.
- After operation, rinse out buffers and solutions that form peroxides.
- If the mobile phase includes volatile or flammable solvents, avoid open flames and sparks.

- If a leak occurs, turn off the instrument and remedy the situation immediately.
- Disconnect the module from all power sources before removing any panels. When the panels are removed, dangerous electrical connections will be exposed.
- Always replace blown fuses with original spare part fuses (→ page 124).
- Replace faulty power cords and communication cables.
- Many organic solvents and buffers are toxic. Know the toxicological properties of all mobile phases that you are using.
- The toxicological properties of many samples may not be well known. If you have any doubt about a sample, treat it as if it contains a potentially harmful substance.
- Wear goggles when handling mobile phases or operating the instrument. An eyewash facility and a sink should be close to the unit. If any mobile phase splashes on the eyes or skin, wash the affected area and seek medical attention.
- Dispose of waste mobile phase in an environmentally safe manner that is consistent with all local regulations. Do not allow flammable or toxic solvents to accumulate. Follow a regulated, approved waste disposal program. Never dispose of flammable or toxic solvents through the municipal sewage system.
- Use only standard solvents (HPLC grade) and buffers that are compatible with all parts that may be exposed to solvents.
- 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 ($CHCl_3$), 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 (the concentrated acids are not an issue when used in short wash cycles).
- Do not use PEEK tubing that is stressed, bent, or kinked.
- Before interrupting operation for several days or more or when preparing the detector for transport, observe the precautions for shutting down the detector (→ page 83).
- Use original spare parts only. Substituting original spare parts or using accessories other than those recommended by Thermo Fisher Scientific may impair the performance of the instrument.
- Do not use the detector in ways other than those described in this manual.
- Always maintain flow when potential is applied. Never run the electrochemical cell dry while applying potential to the electrodes, as this may damage the electrodes. Make sure that the mobile phase flow is established before the cell is turned on.
- Use mobile phases with a supporting electrolyte for potential control. Observe the general guidelines for mobile phases with the electrochemical detector (→ page 57).

- When using the detector in a system that contains metals, such as stainless steel, passivate the instruments and components before use to prevent unwanted oxidation of electroactive compounds in the mobile phase. For further information about passivation, refer to page 137.
- Do not use the detector and its accessories in a manner not specified by Thermo Fisher Scientific. Otherwise the safety protection provided by the equipment may be impaired.

1.1.1 Consignes Générales de Sécurité

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

Observez les étiquettes d'avertissement sur l'appareil et référez-vous aux sections correspondantes dans ce mode d'emploi.

- Veuillez observer les consignes générales de sécurité suivantes lorsque vous utilisez l'instrument ou que vous procédez à des opérations de maintenance:
- Installez le système HPLC dans un laboratoire bien ventilé. Si la phase mobile contient des solvants volatils ou inflammables, empêchez qu'ils ne pénètrent dans l'espace de travail.
- Afin d'éviter au maximum les interférences, tous les éléments du système analytique doivent être raccordés à la même ligne secteur (même phase).
- Lorsque vous soulevez l'instrument, tenez-le toujours par le dessous ou par les côtés de l'unité. Soulever l'instrument par la partie avant inférieure ou par le panneau avant peut endommager la porte.
- Ne placez aucun objet lourd sur la porte ouverte du panneau avant. Ceci pourrait endommager la porte.
- Après utilisation, purgez le système des tampons et des susceptibles de former des peroxydes.
- Si la phase mobile contient des solvants volatils ou inflammables, évitez les flammes nues et les sources d'étincelles à proximité.
- Si une fuite se produit, arrêtez immédiatement l'instrument et remédiez au problème.
- Quand les capots de protection de l'appareil sont démontés, vous êtes exposés à des connexions électriques sous haute tension deviennent accessibles. Débranchez le passeur d'échantillon de toute source d'alimentation électrique avant de retirer les capots. Ne démontez les capots de protection que si cela est explicitement demandé au cours de ces instructions.
- Remplacez toujours les fusibles grillés par des fusibles de rechange d'origine (→ page 124).

- Remplacez les cordons d'alimentation électrique et les câbles de communication défectueux.
- De nombreux solvants organiques et solutions salines sont toxiques. Informez-vous des propriétés toxicologiques de toutes les phases mobiles que vous utilisez.
- Les propriétés toxicologiques de nombreux échantillons peuvent être mal connues. Au moindre doute concernant un échantillon, traitez-le comme s'il contenait une substance potentiellement dangereuse.
- Portez des lunettes de protection lorsque vous manipulez des phases mobiles ou que vous utilisez l'instrument. Une installation permettant de se laver les yeux ainsi qu'un lavabo doivent se trouver à proximité du système. Si une phase mobile, quelle qu'elle soit, entre en contact avec vos yeux ou votre peau, rincez abondamment la zone affectée à l'eau, puis.
- Débarrassez-vous de tous les déchets de phase mobile de manière écologique, conformément à la règlementation en vigueur au niveau local. Empêchez impérativement l'accumulation de solvants inflammables et/ou toxiques. Suivez un programme d'élimination des déchets règlementé et approuvé. Ne jetez jamais de solvants inflammables et/ou toxiques dans le système municipal d'évacuation des eaux usées.
- Utilisez uniquement des solvants (qualité HPLC) et des solutions salines compatibles avec les matériaux exposés phase mobiles.
- 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 ($CHCl_3$), 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. (Ces acides peuvent cependant être utilisés dans le cadre de procédures de nettoyage, à condition que l'exposition soit brève.)
- N'utilisez pas de tubes PEEK écrasés, pliés ou abimés.
- Avant d'interrompre le fonctionnement pendant plusieurs jours ou plus, observez les précautions figurant en page 83.
- Utilisez des pièces de rechange d'origine. Effectuer des remplacements par des pièces ne provenant pas de Thermo Fisher Scientific ou utiliser des accessoires ne provenant pas de Thermo Fisher Scientific peut affecter les performances de l'instrument.
- N'utilisez pas l'instrument de manière autre que celles décrites dans ce manuel.
- Toujours avoir un débit lorsqu'une tension est appliquée. Ne jamais utiliser la cellule électrochimique à sec lorsqu'un potentiel est appliqué sur les électrodes, ceci pourrait endommager les électrodes. Mettre en route le débit de la phase mobile avant d'allumer la cellule.

- Utiliser une phase mobile avec un électrolyte compatible pour contrôler le potentiel électrique. Suivez les recommandations d'utilisation des phases mobiles avec un détecteur électrochimique (→ page 57).
- Avant l'utilisation du détecteur avec une chaîne contenant des pièces métalliques, tel l'acier inoxydable, passiver les appareils et les tubulures, afin de prévenir toute oxydation des composants électroactifs dans la phase mobile. Pour plus d'information sur la passivation , reportez-vous à la page 137.
- Ne pas utiliser le détecteur et ses accessoires autrement que de la manière décrite par Thermo Fisher Scientific. Sinon, les dispositifs de protection fournis par l'équipement pourraient être altérés.

1.3 Intended Use

The detector is designed to perform equally well as a dependable system for routine analyses or as a sophisticated research instrument for use in HPLC and UHPLC (ultra-high performance liquid chromatography) applications, especially as part of the UltiMate 3000 system. However, it can also be used with other HPLC systems if adequate control inputs and outputs are available. A PC with a USB 2.0 is required.

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

- Xcalibur™, Compass™/HyStar™, or Analyst®. Installation of the DCMS^{Link} (Thermo Scientific Dionex Chromatography Mass Spectrometry Link) software is required in addition to the installation of the data system.
- Empower™. Installation of the Thermo Scientific Dionex Instrument Integration Software is required in addition to the installation of the data system.

For information about the availability, contact the Thermo Fisher Scientific sales organization for Dionex HPLC products.

Please note that the detector may be operated only using the accessories and spare parts originally supplied with the unit (→ page 131) and within its technical specifications (→ page 127).

Use only standard solvents of at least HPLC grade or better LC-MS grade (0.2 µm, filtered), and buffers that are compatible with the flow path materials. Note the special properties of the solvents, such as the viscosity, boiling point, and electroactive compounds as well as pH value.

Observe the information about the solvent compatibility, buffer concentrations and mobile phase requirements of the other UltiMate 3000 system modules. For more information, refer to the *Operating Instructions* for the modules.

If there is any question regarding appropriate usage, contact Thermo Fisher Scientific before proceeding.

Thermo Fisher Scientific cannot be held liable for any damage, material or otherwise, resulting from inappropriate or improper use of the instrument.

1.4 Federal Communications Commission (FCC) Note

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the U.S. FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference, in which case the user will be required to correct the interference at his expense.

2 Overview

2.1 Unit Description

The detector is a high-quality instrument designed for the detection of electroactive species and HPLC analysis as part of the UltiMate 3000 system. The module was developed especially for HPLC and UHPLC analysis and can be used in numerous laboratory environments for routine analysis and sophisticated HPLC research tasks:

- Choose from coulometric or amperometric cells for your analysis:
  - ♦ Coulometric detection provides stability, maintenance-free operation and flow rate independence.
  - ♦ Amperometric cells provide the ultimate sensitivity, even with volume-limited samples.
- The detector can measure up to four channels with independent parameters (sensitivity, filter constant) simultaneously.
- The electrochemical cells (or sensors) are equipped with SmartChip™ technology for simple and flexible operation. The potential limits are automatically defined by the SmartChip cell identification. Applied potential to a cell is disabled automatically via active monitoring if the mobile phase flowing through the cell is interrupted.
- Electrochemical detection delivers the high sensitivity needed for the measurement of neurotransmitters, simplicity and robustness for pharmaceutical or clinical diagnostics, and the selectivity for characterization of complex samples such as natural products, biological tissues and fluids.
- Full-scale autoranging protects data integrity by preventing that data is lost due to peak over ranging.
- The integrated controlled column compartment provides accurate column temperature control for reproducible chromatographic results without the need for a separate column oven.
- Controlling the detector by Chromeleon provides a high degree of system integration, as well as maximum analysis efficiency due to comprehensive data analysis and evaluation features in Chromeleon.
- Various safety and monitoring features are provided for optimum system performance and reliability (→ page 25).
- All parts that may be exposed to solvents are made of materials that provide optimum resistance to the most commonly used solvents and buffer solutions in HPLC.

2.2 Operating Principle

Electrochemical detection involves the use of an applied potential to effect a chemical reaction. During this process, the current resulting when a compound undergoes reduction (or oxidation) is measured and related to the amount of compound undergoing electrolysis. In an electrochemical detector, the eluent passes through a cell that provides the appropriate potentials and measures the current.

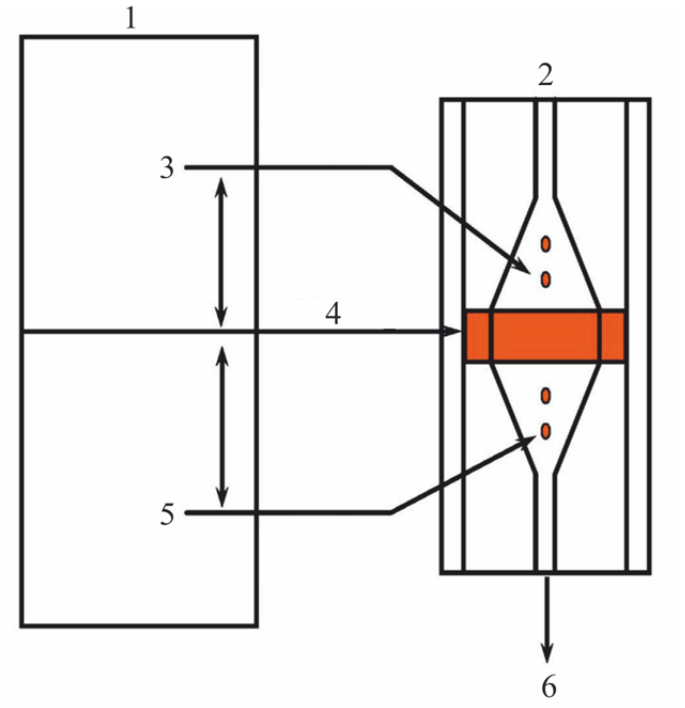


Fig. 1: Schematic of the operating principle

No.	Description
1	Potentiostat module
2	Electrochemical cell
3	Reference electrode (set point for the potential to be applied to the working electrode)
4	Working electrode (electrochemical reaction occurs)
5	Counter electrode (current is measured)
6	Flow out of the cell

There are two methods of electrochemical detection:

- **Amperometric Detection**
  In an amperometric cell, the eluent *flows by* the electrode surface. In this design, a fraction of the electroactive species in the eluent will be oxidized (reduced); but most of the electroactive compound flows by the electrode surface and does not react. The fraction of the electroactive compound that reacts is typically in the order of 5-15%. The current is proportional to the concentration of the compound of interest.

- **Coulometric Detection**
  In a coulometric cell, the eluent *flows through* a porous graphite electrode, rather than flowing by the electrode, as in an amperometric cell. Since the surface area is large, essentially all of the electroactive species will be oxidized or reduced. Since a larger amount of the electroactive compound (10 to 20 times as much) is oxidized (or reduced) without a corresponding increase in noise, this detector can provide enhanced sensitivity. The current produced is directly proportional to the amount of the compound of interest in the sample via Faraday's law.

Both amperometric and coulometric cells can be used with the detector to optimize performance for a given application.

2.3 Configurations

To operate the detector a potentiostat module and an electrochemical cell are required that must be ordered separately.

The following detector is available:

Detector Description	Part no.
ECD-3000RS detector for electrochemical detection	5070.0010

The detector is shipped with an accessory kit and *Operating Instructions*.

The following potentiostat module is available for the detector:

Description	Part no.
Potentiostat Module, dual channel DC, including Installation Instructions	6070.1400

For further information about the potentiostat module, refer to page 20.

The following electrochemical cells are available for the potentiostat module.

Description	Part no.
Coulometric Cell 6011RS, including Cell Waste Line Kit and Installation Instructions	6070.2400
Amperometric Cell 6041RS, including Cell Waste Line Kit, Gasket Kit (25 and 50 nL), and Installation Instructions The cell is shipped without working electrode. The working electrode must be ordered separately.	6070.3000

The following working electrodes are available for the Amperometric Cell 6041RS:

Description	Part no.
Working electrode, glassy carbon (GC)	6070.3200
Working electrode, boron-doped diamond (BDD)	6070.3100

For further information about the electrochemical cells and the working electrodes, refer to page 21.

2.4 Interior Components

The front panel door tilts upward to provide easy access to the interior front panel, for example, for maintenance and repair work.

⚠ **Important:** The open front panel door is not designed to carry weight. Therefore, you should not place any objects on the open door.

⚠ **Important:** Ne placez aucun objet lourd sur la porte ouverte du panneau avant. Ceci pourrait endommager la porte.

Fig. 2: Interior front panel view

No.	Description
1	Bay, assigned A, B, C, D (from left to right) (here with cover)
2	Potentiostat module (→ page 20)
3	Electrochemical cell connection port (→ page 21)
4	Column compartment (here with cover) (→ page 23)

2.5 Front Panel Elements

Fig. 3: Front panel view

No.	Front Panel Element	Function
1	Display	Shows information about the detector: - General information upon power up (→ page 63) - Status screen (→ page 63)
2	**LEDs**	
	Power	The LED is blue when the detector is on.
	Connected	The LED is green when the detector is controlled by Chromeleon.
	Status	The LED is red when an error has been detected, e.g. when an error occurred.

2.6 Rear Panel

Fig. 4: Rear Panel

No.	Description
1	Power switch (→ page 17)
2	Fuse cartridge (→ page 17)
3	Main power receptacle (→ page 32)
4	Protective grounding
5	Digital I/O port (→ page 18) for communication with a pump and other external devices, for example, a mass spectrometer
6	USB port (USB 2.0) for connecting the module to the Chromeleon computer (→ page 18)

2.6.1 Power Switch

The power switch on the rear panel is the main power switch for the detector. Turn on the power switch before initial operation of the detector and leave it on. For routine operation, leave the main power switch on. Turn off the main power switch when instructed to do so, for example, before performing a service procedure or when interrupting operation for longer periods (one week or more). Observe the precautions on page 83.

2.6.2 Fuse Cartridge

The fuse cartridge contains two slow-blow fuses rated at 1 A (5 x 20 mm). For information about how to change the fuses, see page 124.

2.6.3 USB Connector

The Chromeleon Chromatography Management System can use a USB connection to control the detector. Data is transferred digitally via the appropriate USB cable (→ page 32). The PC must be equipped with a USB 2.0 port. Connect the detector directly to the PC. To ensure trouble-free operation, use only the cables shipped with the detector.

For information about how to connect the detector to the Chromeleon computer, see sections 3.4.1 and 3.4.2 (→ page 32).

2.6.4 Digital I/O

The detector has two digital I/O ports, each of which is a 6-pin mini DIN digital I/O port that provides 3 digital inputs and 3 relay outputs. The ports can be used to connect a pump of the UltiMate 3000 series (except the LPG-3400XRS pump*) and to exchange digital signals with other external devices. For details about connecting the digital I/O ports, refer to page 33.

For information about the functions of the connector pins and pin assignment, see page 139.

* The connection to a LPG-3400XRS pump will be supported in a future Chromeleon version.

2.7 Fluid Connections

The detector is designed to provide easy access to the fluid components. Tilt the front cover upward. At dedicated positions in the interior front panel of the enclosure, four slots are provided for the capillaries: on the left and right side of the enclosure (two slots each). In addition, there are capillary slots on the column compartment for easy and direct access of capillaries from the column to the cells.

Capillary guides on the column compartment facilitate routing the capillaries to devices that are located underneath the detector in the UltiMate 3000 system stack.

When closing the front panel door, avoid bending the capillaries and make sure that they are routed to the outside through these slots.

Tip: The volume between the column and the electrochemical cell should be as low as possible to avoid peak broadening effects and the accompanying loss of chromatographic efficiency.

2.8 Bays

Four bays on the interior front panel (→ Fig. 2, page 15) provide the space to insert potentiostat modules and connect electrochemical cells to the detector. The bays are closed with covers when the detector is shipped to prevent dust and other particles from entering them. Keep the bays closed with the covers when not used and only remove the covers for installing potentiostat modules.

2.9 Potentiostat Module

The detector is shipped without a potentiostat module. Install a potentiostat module first (→ page 49). For general information about the installation of the potentiostat module, refer to page 49. For detailed instructions, refer to the Installation Instructions that are provided with the potentiostat.

The potentiostat module contains all the necessary electronics to apply a potential to a cell and measure the resulting current produced when an analyte undergoes an electrochemical reaction.

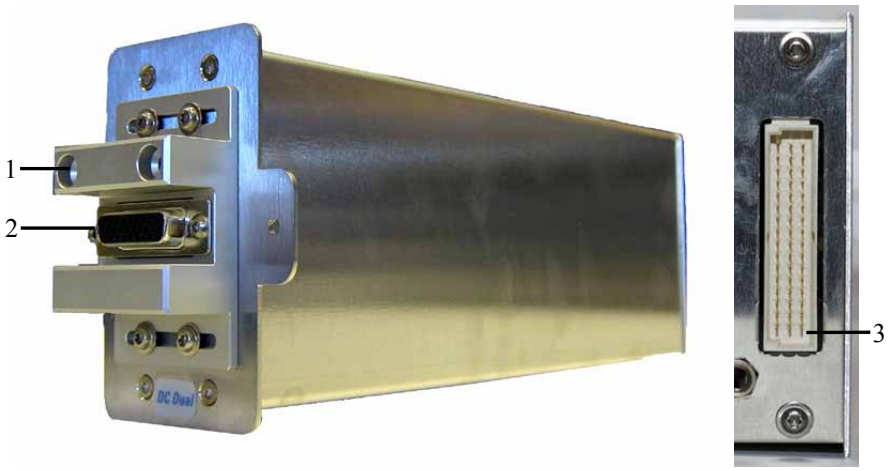


Fig. 5: Side and rear view of a dual DC potentiostat module

No.	Description
1	Slots for guiding pins on the electrochemical cell
2	Connector for the electrochemical cell
3	Connector for the connection to the detector

The following potentiostat module is available for the detector:

Part no.	Description
6070.1400	Potentiostat Module, dual channel DC, including Installation Instructions

2.10 Electrochemical Cells

The detector is shipped without an electrochemical cell. Install a suitable electrochemical cell first as described on the Installation Instructions provided with the cell.
For general information about the installation of the electrochemical cell, refer to page 54.

An electrochemical cell (or sensor) contains electrodes to which a potential (voltage) is applied.

All electrochemical cells are fitted with an identification chip at the factory (SmartChip™ technology). The chip stores unique information about the electrochemical cell, including the cell model and serial number. When the electrochemical cell is installed, the detector connects to the electronics on the cell. Two types of electrochemical cells are available for the detector: amperometric cells and coulometric cells. Both types are used for analytical measurements.

Fig. 6: Left: Coulometric Cell 6011RS
Right: Amperometric Cell 6041RS

The following electrochemical cells are available for the detector:

Cell type	Cell material	Cell inlet volume	Working electrode	Part no.
Coulometric Cell 6011RS, pressure limit: 40 bar	PEEK, porous graphite, palladium, PTFE	7.06 µL	2 working electrodes, micro-porous graphite carbon	6070.2400
Amperometric Cell 6041RS, pressure limit: 13.8 bar	PEEK, palladium, boPET*	25 nL gasket or 50 nL gasket (selectable)	1 working electrode, glassy carbon, or boron-doped diamond (selectable)	6070.3000

* Further cell material depends on the working electrode used.

All cells are shipped with cell accessories. For further information about the content of the respective cell kits, refer to section 9.2 (→ page 133).

Note: The Amperometric Cell 6041RS is shipped without a working electrode installed. The working electrode must be ordered separately and installed as described on the Installation Instructions provided with the cell before first use.

For further information about the operation of electrochemical cells and optimization of the cell performance, refer to section 5.5 (→ page 72) and section 5.6 (→ page 75).

2.11 Column Compartment

A removable panel on the front of the detector provides easy access to the column compartment and the components inside.

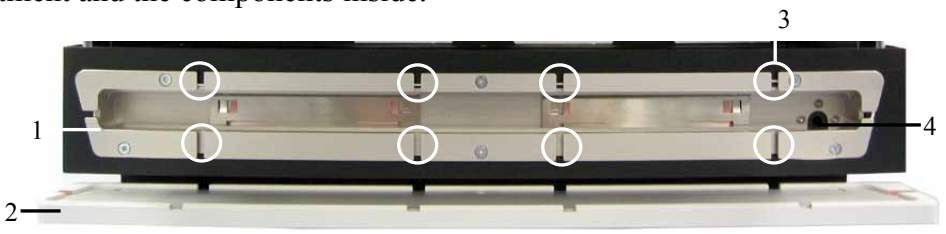


Fig. 7: View into the column chamber

No.	Description
1	Column compartment
2	Removable panel
3	Capillary slots to route the capillary from the column outlet to the outside
4	Passage from the drain port to the column compartment A capillary is routed from the column compartment directly to the drain port of the detector as direct drain passage if liquid has collected in the compartment.

The column compartment can house one column with a maximum length of 30 cm and a maximum outer diameter of 18.5 mm.

For information about how to connect the column, see section 4.6 (→ page 53).

2.12 Leak Sensor

A leak sensor (→ Fig. 2, page 15) is installed inside the detector between Bay B and C for the automatic sensing of fluid leaks. If liquid collects in the drip tray under the fluid connections**,** the leak sensor reports a leak. The **Status** LED on the front panel door changes to red, and a message appears in the Chromeleon Audit Trail and the cells are turned off.

When the leak sensor reports a leak, eliminate the cause for the leakage, dry the leak sensor and perform the appropriate remedy actions from Chromeleon (→ page 123). If the sensor is not dry, the **Status** LED remains red.

2.13 Chromeleon Software

The detector can be controlled by the Chromeleon Chromatography Management System. To control the detector, an appropriate Chromeleon version and a Timebase Class 1 Chromeleon license are required.

Tip: All software details in this manual refer to *Chromeleon 6.80*.

If you want to operate the detector 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 the following:

- Chromeleon 7 terminology is different from the terminology used in Chromeleon 6.80. For details, refer to the 'Glossary -Chromeleon 7.0,' which is available in the Documents folder of your Chromeleon 7 installation.
- Chromeleon 7 may not yet support all functions supported in Chromeleon 6.80.

Two modes of software control are available:

- **Direct Control**
  With direct control, you select operating parameters and commands in the **Commands** (F8) dialog box. Direct commands are executed as soon as they are entered. For routine operation, most parameters and commands are available also on a control panel. For more information about direct control, see page 66.
- **Automated Control**
  With automated control, you create a program (or PGM File). This is a list of control commands, executed in chronological order, for automated operation of the detector. Programs can be created automatically with the help of a software wizard or manually by editing an existing program. For more information about automatic control, see page 68.

2.14 System Wellness and Predictive Performance

System Wellness monitors the health of the detector and the electrochemical cells. Therefore, the detector supports several performance and reliability features that can help you detect small problems before they turn into big ones:

- Internal monitoring of all operations
- Automatic self test upon power up
- SmartChip technology for automatic cell identification and documentation
- Automatic shutdown of cells when pump is connected and flow is stopped during a run
- Leak sensor (→ page 23)

When an error is detected, the **Status** LED on the front panel turns red and a message is displayed in the Chromeleon Audit Trail.

Additional functions for estimating the lifetime of consumables and monitoring and recording service and (re)qualification information (= predictive performance; → page 81) are available.

3 Installation

3.1 Facility Requirements

- Make sure that the installation site meets the power and environmental specifications listed in the Technical Information section (→ page 127).
- Install the detector 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 detector for power connections and ventilation.

3.2 Unpacking

All electrical components of the detector 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 module in case of future transit. Shipping the module in any other packaging automatically voids the product warranty.

1. Open the packaging box of the detector and remove the accessories kit and power cord. Some accessories may be shipped in a separate box.
2. Grasp the detector by the sides. Slowly and carefully, pull the detector out of the shipping container and place it on a stable surface.

Important: To prevent the detector from falling, grasp the detector by the sides, and then lift the detector together with the foam spacers out of the shipping container. Do *not* lift the module by the foam spacers and *not* by the front panel doors.

Important: Afin d'empêcher l'instrument de tomber, saisissez-la par les côtés. Ne soulevez l'instrumente à l'aide du matériau d'emballage ou par les portes des panneaux avants.

3. Remove the foam spacers, and then remove the polythene packaging.

4. Tilt the front panel of the detector upward and remove the foam inserts securing the front panel door during shipment.

5. Before connecting the detector to the power source, wait approximately 4 hours to allow the instrument to come to room temperature and to allow any condensation that might have occurred during shipping to evaporate. After 4 hours, check the detector; if condensation still exists, allow the detector to continue to warm up (without connecting it to the power source) until the condensation is completely gone.

3.3 Positioning the Detector in the UltiMate 3000 System

If the detector is part of an UltiMate 3000 system, for example for analytical HPLC applications, you should stack the individual modules, for example, as shown in Fig. 8 and interconnect them on the rear panel as shown in Fig. 9. A top-down fluidic path from pump to autosampler, with the detector in between provides the best arrangement. However, the arrangement of the system modules depends on the application and may vary if an optical detector is used additionally.

Fig. 8: Module arrangement for an UltiMate 3000 system with ECD-3000RS detector (example)

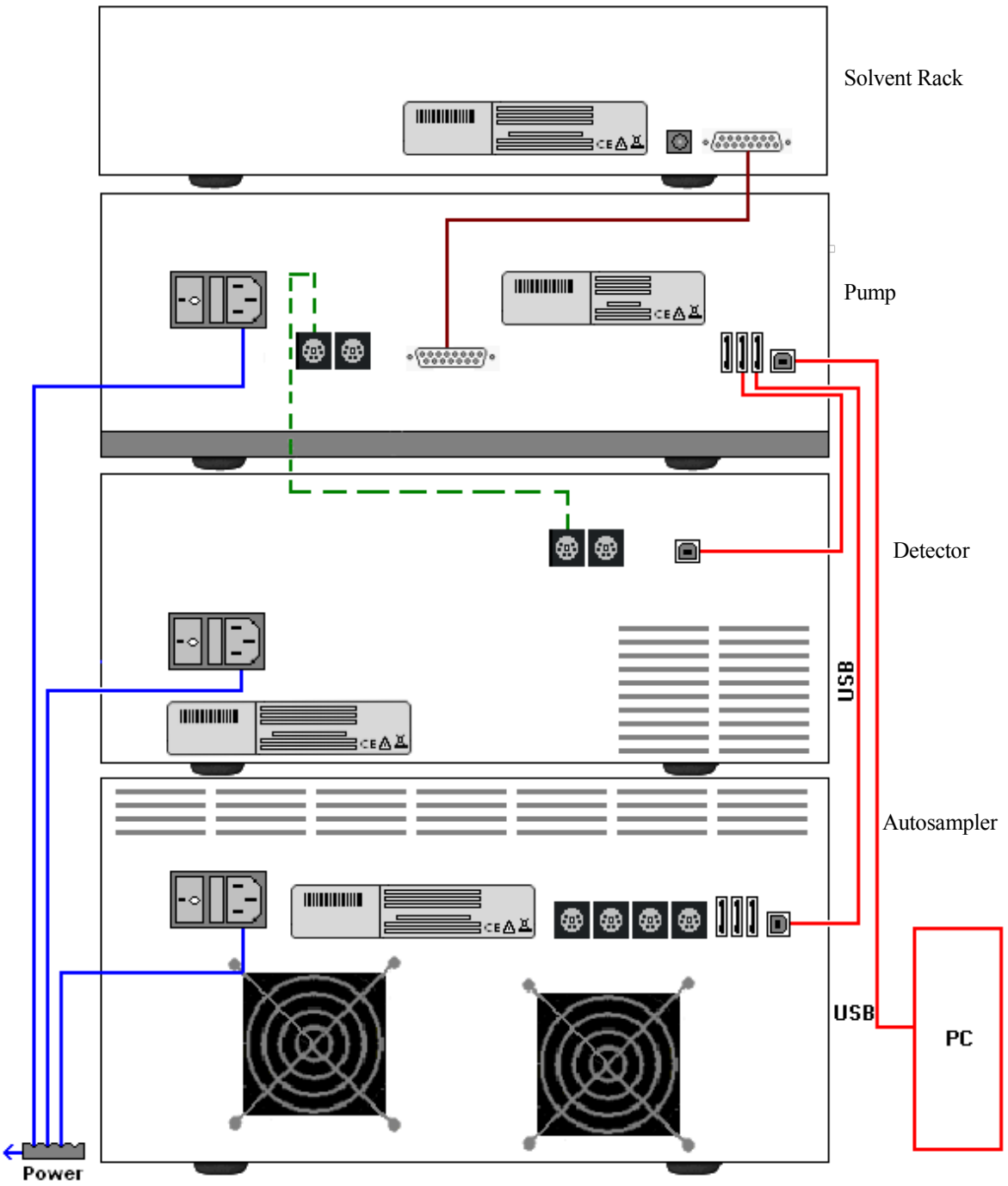


Fig. 9: Example for the rear panel connections on an UltiMate 3000 system with ECD-3000RS detector

When connecting the detector to the UltiMate 3000 system, keep in mind that the detector has no USB hub on its rear panel. Thus, it can only be connected to USB hubs of other instruments in the UltiMate 3000 system or directly to the computer.

Apart from the Solvent Rack, all modules of the UltiMate 3000 system can be connected separately to the Chromeleon computer via the USB port on the rear panel of the instrument. However, Thermo Fisher Scientific recommends interconnecting all modules, and then connecting the system to the Chromeleon computer with only one connection. For systems with a DAD-3000(RS) or MWD-3000(RS), you can use *only* the hub on the DAD or MWD detector for the connection.

Tip: As the electrochemical cell of the detector has a pressure limit of 13.8 bar (amperometric cell) or 40 bar (coulometric cell), the electrochemical detector must always be the last module in the fluidic path.

It is not possible to use the USB hub on the autosampler for connection of the detector to the Chromeleon PC.

For proper operation, connect the detector directly to the UltiMate 3000 pump using the Digital I/O port as shown in Fig. 9. For further information, refer to section 3.4.4 (→ page 33).

For information how to connect the USB port on the rear panel, refer to section 3.4.2.

3.4 Connecting the Detector

3.4.1 General Information

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 if you install Chromeleon first, the USB driver for the detector is automatically loaded and the Windows® operating system can detect the detector when the power is turned on.

3.4.2 Connecting the USB Cable

Directly connect the detector to the Chromeleon computer via the USB port on the rear panel (→ Fig. 4). The PC must be equipped with a USB 2.0 port. Note that it is not possible to connect the detector to a USB hub on the autosampler.

- Connect the detector directly to the USB port on the computer.
- Connect the detector to an internal USB hub on the pump of the UltiMate 3000 series. (except LPG-3400XRS pump*). Thermo Fisher Scientific recommends connecting all modules to the pump, and then connecting the system to the computer via only one connection. If the system includes a UV detector in addition to the electrochemical detector, Thermo Fisher Scientific recommends connecting the UV detector directly to the computer.

  For information about how to connect a pump to the detector, refer to section 3.4.4 (→ page 33).

Tip: The USB standard limits the USB cable length to 5 meters. Each USB device can be separated from the PC or next USB hub by no more than 5 meters

The following cables are available (provided in the accessories kit for the detector):

USB Cable	Part no.
USB cable, type A to type B, high speed USB 2.0 (cable length: 5 m)	6911.0002

* The connection to a LPG-3400XRS pump will be supported in a future Chromeleon version.

3.4.3 Connecting the Power Cord

Use the power cord shipped with the detector to connect the instrument to the main power source. Connect the power cord from the main power receptacle on the rear panel (→ Fig. 4). No manual adjustment is required to adapt the line voltage to local voltage requirements.

3.4.4 Connecting the Digital I/O

To connect external devices to the digital I/O port, use the appropriate cable.

Description	Part No.
Mini DIN signal cable, 6-pin The cable is included in the accessories kit for the detector. For connection to UltiMate 3000 pumps (except the LPG-3400XRS pump*).	---

Use the appropriate mini-DIN cable from the accessory kit for the detector to connect pumps of the UltiMate 3000 series (except the LPG-3400XRS pump*).

To connect the detector to an UltiMate 3000 pump (except LPG-3400XRS pump):

1. Plug the 6-pin connector of the mini-DIN cable into the Digital I/O port A (or B) on the detector.
2. Plug the 6-pin connector of the mini-DIN cable into the Digital I/O port on the pump.

When the detector is properly connected to a pump* of the UltiMate 3000 series with the appropriate mini DIN cable, the cell is turned off (Cells Off function) when an UltiMate 3000 pump* is connected and an error occurs in the pump (for example, a leak occurs). For further information about the Cells Off function, refer to page 82.

For information about the functions of the connector pins and pin assignment, see page 139.

* The connection to a LPG-3400XRS pump will be supported in a future Chromeleon version.

3.5 Setting Up the Detector in Chromeleon

This section provides brief instructions for setting up Chromeleon. For details, also see the Chromeleon Help.

Tip: When the detector is connected to the Chromeleon computer, verify that the Chromeleon software is installed before turning on the detector power for the first time. Only then, the USB driver for the detector is automatically loaded and the Windows operating system detects the detector when the power is turned on.

3.5.1 Loading the USB Driver for the Detector

1. Turn on the computer power, if it is not already on.
2. Under Windows Vista® (Windows® XP, Windows® 7, or Windows® Server 2008) log on as a
   - Local administrator if the computer is a local computer.
   - User with local computer administrator privileges if the computer is a network computer.
3. Open the **Chromeleon Server Monitor** program by double-clicking the Chromeleon Server Monitor icon  on the Windows taskbar.

   If the Server Monitor icon is not on the taskbar, click **Start** on the taskbar, point to **Programs** (or **All Programs**, depending on the operating system), point to **Chromeleon,** and then click **Server Monitor**.
4. Click **Start** to start the server.
5. Click **Close** to close the Server Monitor window. The Server Monitor icon  appears on the taskbar.

   Tip: Clicking the **Quit Monitor** button quits (exits) the **Server Monitor** program, but does not stop the server. To stop the server, click **Stop**.
6. Turn on the main power switch on the rear panel of the detector.

7. Windows Vista, Windows 7, and Windows Server 2008
will automatically detect the new detector and perform the USB installation.

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

   a) Click **Cancel** to exit the wizard.

   b) Turn off the detector.

   c) Install Chromeleon.

   d) Turn on the detector power. Windows will now detect the detector and install the USB software for the detector automatically.

   Windows XP
   will automatically detect the new detector and launch the Found **New Hardware Wizard**, which guides you through the USB installation. Select the following options:

   a) If asked whether Windows can connect to Windows Update to search for software, select **No, not this time**.

   b) Accept the default option (**Install the software automatically**) and click **Next>**.

   c) Click **Finish** when the wizard reports that the software for the detector has been installed.

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

   d) Click **Cancel** in the Windows message box.

   e) Turn off the detector.

   f) Install Chromeleon.

   g) Turn on the detector power. Windows will now automatically detect the detector and launch the **Found New Hardware Wizard**.

3.5.2 Installing the Detector

The detector should complete the self-test (→ page 63) prior to connecting the detector to the computer to prevent possible installation errors. A green front panel LED status light indicates a completed self-test and that the detector is ready for connection.

After the USB software for the detector has been installed (→ page 34), install and configure the detector in Chromeleon:

1. Start the Chromeleon **Server Monitor** (→ page 34) and the Chromeleon server if they are not yet running.
2. Start the Chromeleon **Server Configuration** program by clicking **Start** on the taskbar. Point to **Programs** (or **All Programs**, depending on the operating system), point to **Chromeleon**, and then click **Server Configuration**.
3. If necessary, click the plus sign beside the server icon  to display the items underneath.
4. Select the timebase to which the detector will be assigned, or create a new timebase (on the **Edit** menu, click **Add Timebase**).
5. Open the **Add device to timebase** dialog box. To do so, click **Add Device** on the **Edit** menu or right-click the timebase and click **Add Device** on the menu.
6. On the **Manufacturers** list, click **Dionex HPLC: UltiMate 3000** and on the **Devices** list, click **ECD-3000RS Detector**.
7. The configuration pages are opened. On each page, verify that the settings are correct and select additional settings if needed. For a description of the pages, see section 3.5.3.1 (→ page 37).
8. Click **OK** to complete the configuration of the detector.
9. On the **File** menu, click **Save Installation** and then close the **Server Configuration** program.

3.5.3 Configuring the Detector

3.5.3.1 Initial Installation

During the installation, Chromeleon connects to the detector and transfers the settings from the instrument firmware to Chromeleon, setting the options on the wizard pages accordingly. Verify that the settings are correct and make additional settings if needed. You may reopen the configuration pages later again to change the settings (→ page 44).

Tip: Changing the settings for a specific application in the **Commands** (F8) dialog box, in a program file (PGM), or on a control panel will not change the default settings on the configuration pages.

For additional information about a page, click **Help**.

General Page

Shows the general instrument parameters.

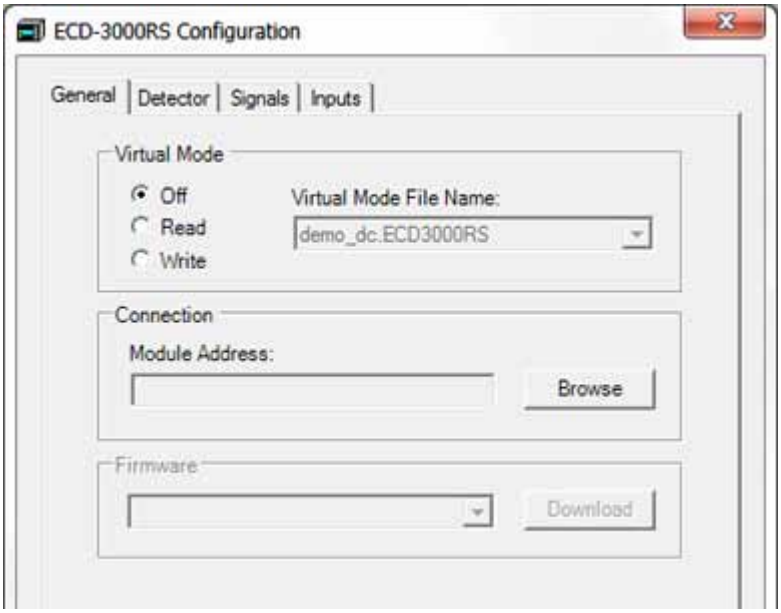


Fig. 10: General page

- **Virtual Mode**

  Verify that the virtual mode is set to off. If the virtual mode is enabled, the **Module Address** box will be unavailable. If you exit this page without having entered a module address, the virtual mode will be enabled automatically.

  In the virtual mode, Chromeleon simulates detector control and data acquisition.

  - Click **Read** to read and display data from an existing demo file instead of real data. Select the file from the **Virtual Mode File Name** list.
  - Click **Write** to save the data currently delivered by the detector as a demo file. Enter the file name in the **Virtual Mode File Name** field or select a name from the list.

- **Module Address**

  Select the module address of the detector if necessary. The module address states the USB port and the serial number of the detector. Click **Browse** and then double-click the detector that you want to use on the **Device List**. The address is automatically entered in the **Module Address** field. Chromeleon connects to the detector and transfers the settings from the instrument firmware to Chromeleon, setting the options on the pages accordingly. Confirm the related message with **OK**.

- **Firmware**

  Click this button to transfer the current detector configuration to Chromeleon. (The button appears dimmed if the virtual mode is enabled.)

  The detector is shipped with the most recent firmware version. If a firmware update is ever required, follow the steps in section 7.7 (→ page 125).

Detector Page

The Detector page shows the detector configuration.

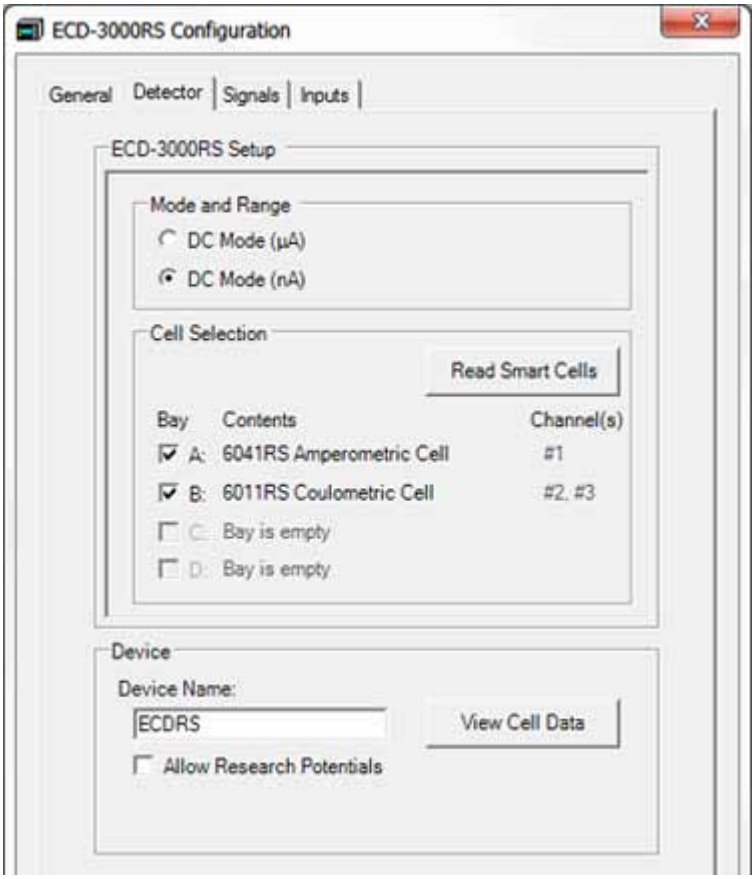


Fig. 11: Detector page

- **Mode and Range**

  Select the desired level of sensitivity. The selected sensitivity affects the maximum signal range and the signal unit:

  - DC Mode (nA)
    This mode is selected by default. Select this mode to measure with high sensitivity and low data ranges (when expected current is below < 2147.4 nA or 2.1 µA).

  - DC Mode (µA)
    Select this mode to measure with low sensitivity and high data ranges (when expected current is above < 2147.4 nA or 2.1 µA).

Tip: When selecting the data range, be aware that in Chromeleon 6.80 high data (current above < 2147.4 nA or 2.1 µA) will be truncated in the nA range and low data will be lost. In Chromeleon 7, both range selections support the full range of current values detectable by the detector, without truncation. Note that the resolution of the data is less when using the µA signal range.

Mode	Signal unit	Signal range
DC Mode	nA	min. -2147.4 nA max. +2147.4 nA
DC Mode	µA	min. -100.00 µA max. +100.00 µA

- **Cell Selection**

  Shows the four bays, the installed potentiostat modules, and the cells connected to the potentiostat modules. Before the first configuration of the bays or if no potentiostat module is installed in a bay, the **Content** of the bay is displayed as "Bay is empty". If no cell is installed in a bay, the **Content** of the bay is displayed as "No cell detected". To display the cells that are installed in the potentiostat modules in the respective bays, click **Read Smart Cells**. Under **Content** the cell model number and type of the cells installed to the potentiostat modules are shown. The channels are assigned automatically. The channel numbers are assigned in the order of the bays, from left to right. Amperometric cells use one data channel; coulometric cells use two data channels. For more information about the data acquisition, refer to page 74.

  To use the respective cell(s) in the bay(s), enable the **Bay** checkbox with the cell(s) which you want to use for detection.

  If you add or remove cells or potentiostat modules from the detector, click **Read Smart Cells** to refresh and display the cell details.

  Tip: When the detector is connected in Chromeleon, the data stored on the cell chips are compared to the **Content** data on the **Detector** page. If the data is not the same, a warning is displayed and the detector cannot be connected.

- **Device**

  - **Device Name**

    Displays the name used to identify the detector the installation environment and in the Chromeleon client program. To control the detector with the existing control panels, 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 device name in the program files. The default **Device Name** is **ECDRS**.

  - **Allow Research Potentials**

    Each cell is operated in its normal potential range which is defined in the cell chip to prevent damage to the cells. You can choose to use an extended potential range, called research potential. To expand the potential range for a cell, select the **Allow Research Potentials** checkbox. For more information, refer to page 72.
    This checkbox is disabled by default.

    ⚠ **Important:** Applying potentials outside the recommended range diminishes the cell performance and can seriously damage the electrodes in the electrochemical cell. Thermo Fisher Scientific recommends not to apply potentials other than that recommended in this manual to avoid damage to the electrodes.

    ⚠ **Important:** L'utilisation de tensions en dehors de la plage recommandée réduit les performances de la cellule et peut endommager sérieusement les électrodes de la cellule électrochimique. Thermo Fisher Scientific déconseille l'utilisation de tensions autres que celles recommandées dans ce manuel, afin d'éviter la détérioration des électrodes.

  - **View Cell Data**

    Click **View Cell Data** to open the **Cell Properties** dialog box, which shows all of the data stored on the cell chip for the cell in the selected bay, such as type, cell serial number, number of electrodes, the operation time and the working electrode material, etc. If the cell installed in the bay is a coulometric cell, the working electrode is displayed and cannot be changed. If the cell in the selected bay is an amperometric cell, the working electrode material that is installed in the amperometric cell must be selected.

The cell properties provide important information for monitoring the performance of the electrochemical cells (→ page 81).

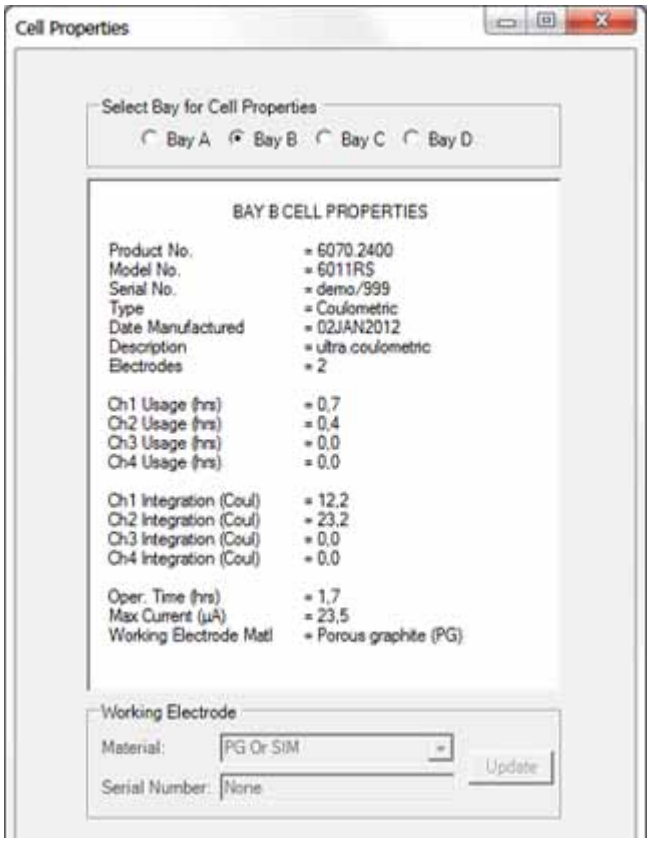


Fig. 12: Cell Properties page

To select the working electrode for an amperometric cell in the **Working Electrode** dialog, click the arrow on the **Material** list and select the working electrode material. Before the first configuration of the working electrode material, the default setting is **Not set**. Open the dropdown menu to select the desired working electrode material for the installed amperometric cell. Be sure to enter the serial number of the working electrode in order to accurately record the cell properties. For more information, refer to the Installation Instructions provided with the working electrode.

Click **Update** to save the selected material. A message is displayed to confirm or cancel the change of the working electrode setting.

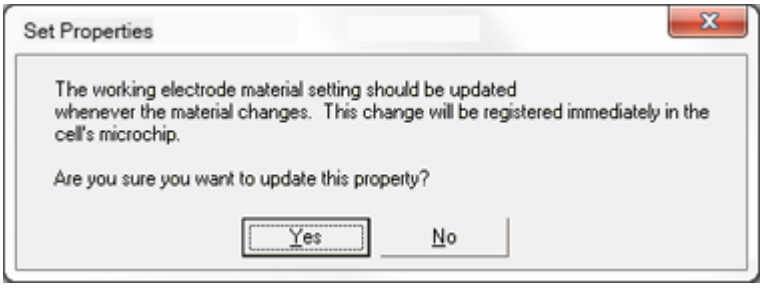


Fig. 13: Saving setting for the working electrode material

The selected working electrode is registered directly to the cell chip. Click **Yes** to save the data to the cell chip and use the working electrode for detection.
If you do not want to save the selected working electrode, click **No**. The data is not registered in the chip and not saved in the detector configuration.

When configuring an amperometric cell and the default setting **Not set** is provided as working electrode, only the lowest range of available potentials can be used in order to avoid damage to the cells.

Signals Page

The page lists all signal channels that the detector can record. The signal type and name of each signal is 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, the detector cannot collect raw data for the signal. To change a signal name, overwrite the existing name directly in the **Name** field.

If the signals are renamed during the configuration, it is also necessary to update the panel tabset.

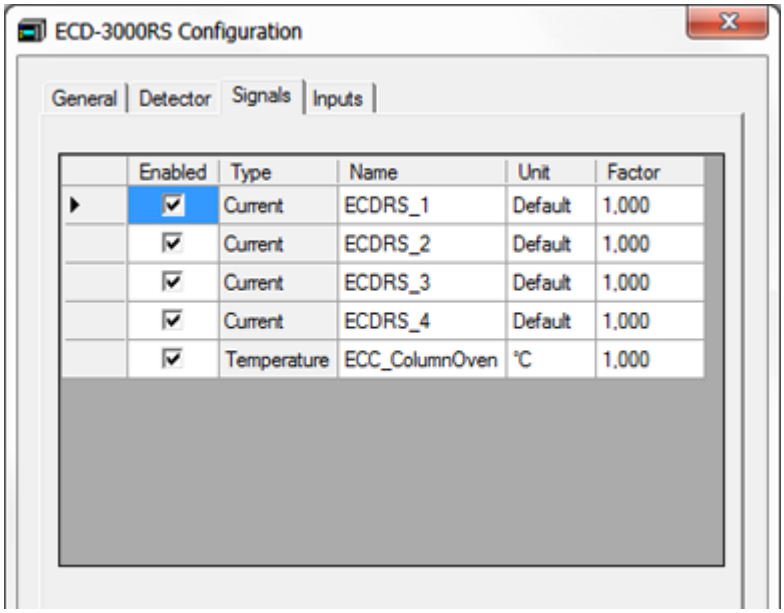


Fig. 14: Signals page

In the column **Unit** of the detector signals ("ECDRS"), the **Default** value stands for either µA or nA, depending on the selected sensitivity on the **Detector** page. You do not need to change the value in the **Unit** field manually for the detector signals.

The **Temperature** signal check box is selected by default. Accept this setting if you want to record the column oven temperature. With this setting, Chromeleon generates the appropriate channel for recording the temperature signal. For more information, see section 5.7.1 (→ page 81).

Inputs Page

The **Inputs** page lists all available remote inputs. Select a check box if you wish to monitor the state of the corresponding input in Chromeleon. If a check box is cleared, the input will not be available in Chromeleon. To change an input name, overwrite the existing name directly in the corresponding line.

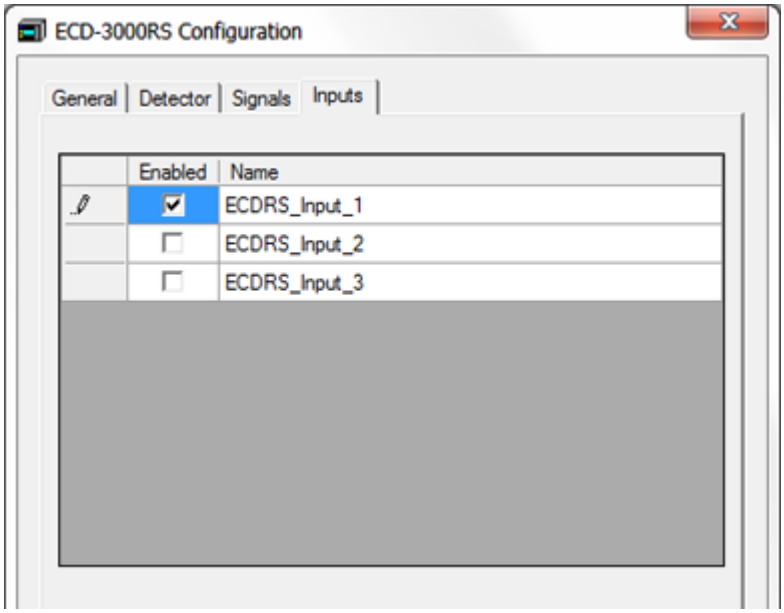


Fig. 15: Inputs page

Input name	Description
ECDRS_Input_1	Inject Start/Stop
ECDRS_Input_2	Autozero
ECDRS_Input_3	Cells Off

If the detector is connected to a pump of the UltiMate 3000 series via the Digital I/O port on the rear panel of the detector and if you wish to monitor this input in the **Commands** (F8) dialog box or with a control panel, you can select **ECDRS_Input_3** on the **Inputs** page. For more information about the cell safety feature, refer to page 82.

3.5.3.2 Changing the Configuration Properties

You may reopen the configuration pages later again to change the settings.

1. Start the **Server Configuration** program (→ page 36).
2. Right-click the **ECD-3000RS Detector** in the timebase and click **Properties** on the menu.
3. Change the settings as needed. For a description of the pages, see section 3.5.3.1 (→ page 37).
4. To save the changed configuration, click **Save** on the **File** menu and then close the **Server Configuration** program.

3.6 Setting Up the Detector in DCMSLink

To set up the detector in DCMSLink, refer to *DCMSLink Installation Guide*, which is provided on the DCMSLink DVD in the *Additional Documents\DCMSLink User Documents* folder.

1. Install and configure the DCMSLink software and access the Chromeleon Server Configuration program by following the instructions in the DCMSLink installation guide.
2. In the Server Configuration program, add the detector to the timebase, by following the appropriate steps in section 3.5.2 on page 36. To configure the detector, see section 3.5.3 on page 37.

For more information about DCMSLink, refer to the *DCMSLink Quick Start Guide*, which is also provided on the DCMSLink DVD and to *DCMSLink Help*.

4 Preparation for Operation (Startup)

4.1 General Precautions for Connecting Capillaries

The following section provides information about how to connect capillaries in your UltiMate 3000 system.

When connecting capillaries to the module, observe the following general precautions:

- Thermo Fisher Scientific recommends using the optional nanoViper capillary connections with the detector to ensure
  - ♦ Metal-free fluidics for optimum electrochemical compatibility
  - ♦ Fingertight connections with zero dead volume
  - ♦ Easy connection of all fluidics
  - ♦ Full compatibility with higher pressure, especially when sub-2 micron columns are used
- When you connect capillaries, make sure that the connectors are free from contaminants. Even minute particles may cause damage to the system.
- Different fitting systems are used in an UltiMate 3000 system. Therefore, install the capillaries and fittings only at the positions for which they are intended.
- Use only the capillaries shipped with the module or original spare capillaries.
- Always make sure that the ID of the replacement capillary corresponds to the ID of the capillary shipped with the system.

Note the following:

- ♦ *Viper and nanoViper fitting connections*

  Loosen or tighten the Viper connection only using the black knurled screw and only with your hand (do not use tools). The knurled screw can be easily removed and reattached to the capillary at any time. If you observe leakage on the connection, tighten the screw a little further. If leakage continues, remove the capillary, clean the capillary ends carefully by using a cloth or tissue wetted with isopropanol, and reinstall the capillary. If the connection continues to leak, replace the Viper capillary.

  When connecting the Viper capillary to the cell inlet, please observe the guidelines in the Installation Instructions shipped with the capillary.

  Capillaries with Viper fitting connections can be reused also for a different connection.

- ♦ *Conventional fitting connections (non-Viper)*

  Do not over-tighten these 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.

For more information about the available capillaries, see section 9.2.

4.2 Overview

After you have unpacked, positioned and connected the detector as described in sections 3.1 through 3.4 (→ page 27 and following), prepare the detector for operation. Follow the sequence of steps below:

1. Connect the drain tubing (→ page 48).
2. Install a potentiostat module to the desired bay (→ page 49). For detailed instructions, refer to the Installation Instructions for the potentiostat module.
3. Install the fluidics for the detector. Make sure that any capillaries used with the detector are made of an electrochemically compatible material.

   a) Connect the in-line filter with graphite filter element (→ page 50).

   b) Connect the column to the column department (→ page 53).

   c) Connect the in-line filter with PEEK filter element (→ page 52).

   When connecting the capillaries, make sure that the connectors are free from contaminants. Even minute particles may cause damage to the system, for example, to the column or the cell.
4. Power up the detector (→ page 63).
   The detector should complete the self-test (→ page 63) prior to connecting the detector to the computer to prevent possible installation errors. A green front panel LED status light indicates a completed self-test and that the detector is ready for connection.
5. No electrochemical cell is installed when the detector is shipped. Install an electrochemical cell to the potentiostat module (→ page 54). For detailed instructions, refer to the Installation Instructions for the respective cell.
6. Set up the detector in Chromeleon as described in section 3.5 (→ page 34).
7. Flush the system and the cells with mobile phase while the cells are turned off. Observe the guidelines for mobile phases (→ page 57).
8. Check the leak sensor setting if necessary (→ page 71).
9. Flush the cell and the system with the mobile phase for the desired application while the cells are turned off. Observe the guidelines for mobile phases (→ page 57).
10. Select a potential and turn on the cells (→ page 72).
11. Before using the module for sample analysis, equilibrate the electrochemical cell and the entire system (→ page 55).

4.3 Connecting the Drain System

To discharge liquid leaks and waste, the detector has a drain port at the bottom right of the instrument.

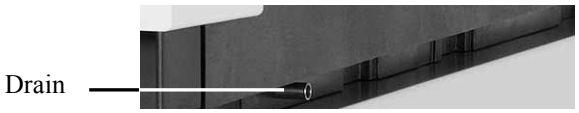


Fig. 16: Drain port

Direct liquid leaks to waste via the drain system of the UltiMate 3000 system, using the components from the drain kit. The kit is shipped with the UltiMate 3000 pumps and can be ordered separately (part no. 6040.0005). The kit includes all required components and detailed installation instructions. If there is more than one detector in your system and you need an additional tee piece, you can find one in the accessories kit of the fluorescence, multiple wavelength, or diode array detector.

4.4 Installing the Potentiostat Module

The installation of the potentiostat module (part no. 6070.1400) is required to connect an electrochemical cell with the detector. For further information about the potentiostat module, refer to page 20.

Detailed instructions for installing the potentiostat module can be obtained from the Installation Instructions for the potentiostat module and also from section 7.2 (→ *Replacing the Potentiostat Module*, page 98). The procedure for first-time installation corresponds to the installation steps when replacing a potentiostat module.

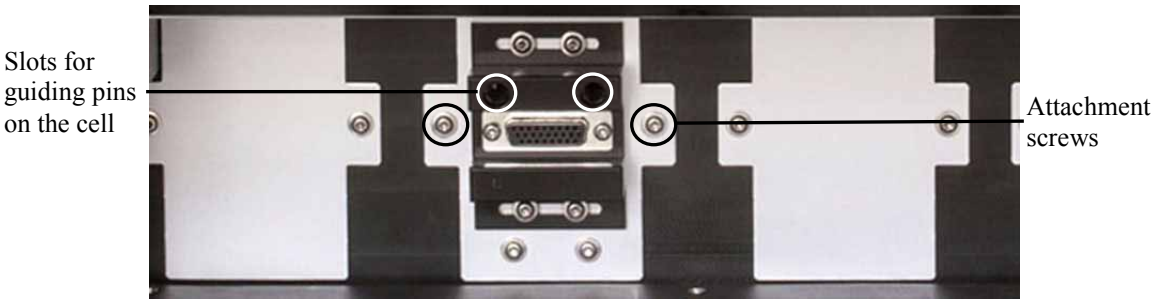


Fig. 17: Potentiostat module installed

4.5 Connecting the In-Line Filters

The inline filters can be used to ensure that particulate matter does not clog the cell, resulting in high backpressure and lowered system performance. Two In-Line Filter kits are included in the accessories kit for the detector:

- In-Line Filter Kit with Graphite Filter Element (part no. 70-0893) including five filter elements
- In-Line Filter Kit with PEEK Filter Element (part no. 70-4093), including five filter elements

The in-line-filter elements should be replaced on a regular basis. For information about the replacement procedure, refer to section 7.4 (→ page 119).

4.5.1 In-Line Filter with Graphite Filter Element

The in-line filter with graphite filter element is to be connected between the pump outlet and the autosampler inlet.

When operating the system, keep in mind that the graphite filter elements are made for a maximum pressure of up to 250 bars. If the application requires higher pressures, do not use the in-line filters.

Fig. 18: In-line filter with graphite filter element

1. Locate the in-line filter kit with graphite filter elements.
2. Open the filter holder by turning the lock nuts counterclockwise and insert the graphite filter element.
3. Close the filter holder by turning the lock nuts clockwise. Do not overtighten the lock nuts with a metal tool.

4. Connect the capillary from the pump outlet (or the Titanium filter) to the inlet of the in-line filter.
5. Connect a capillary to the outlet of the in-line filter. Make sure that the capillary is connected correctly, with the filter holder being installed in the direction of the orientation sign on the filter pointing downstream.
6. Connect the capillary from the filter holder outlet to the inlet of the autosampler.
7. Connect a capillary from the autosampler outlet to the waste.
8. Start the delivery of the mobile phase to remove any contaminants from the system.
9. Monitor the fittings and tighten as appropriate. If a significant increase in pressure is observed, check if an obstruction or clogged component is present.
10. Connect a column to the column compartment (→ page 53).

Fig. 19: In-Line filter installed

4.5.2 In-Line Filter with PEEK Filter Element

The in-line filter with PEEK filter element is to be connected between the column outlet and the electrochemical cell inlet.

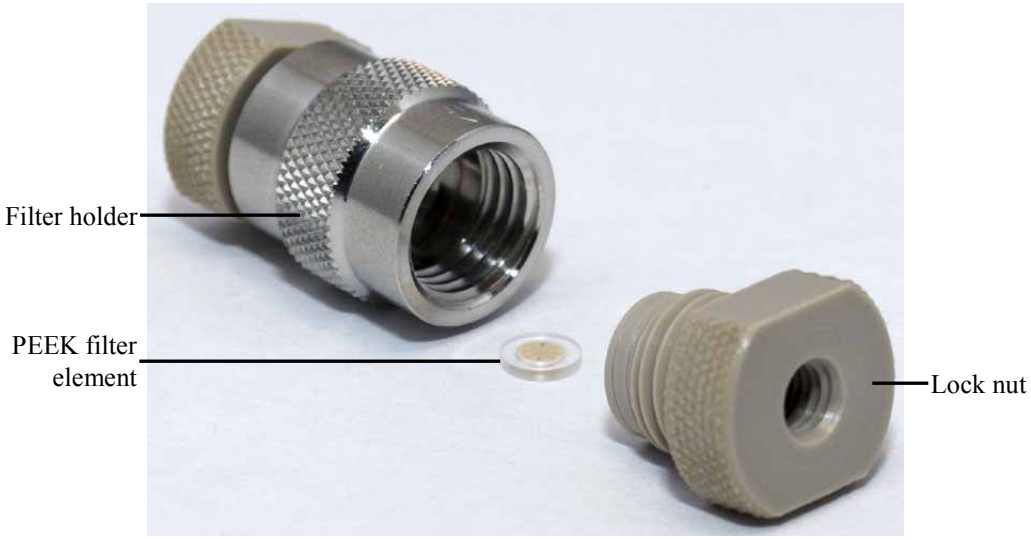


Fig. 20: In-line filter with PEEK filter element

1. Locate the in-line filter kit with PEEK filter elements.
2. Open the filter holder by turning the lock nuts counterclockwise and insert the PEEK filter element.
3. Close the filter holder by turning the lock nuts clockwise. Do not overtighten the lock nuts with a metal tool.
4. Connect capillaries to the inlet and outlet of the in-line filter.
5. Connect the in-line filter with the capillaries from the outlet of the analytical column and to the inlet of electrochemical cell.
6. Make sure that a capillary leads from the outlet of the electrochemical cell to the waste.
7. Flush the system for 3-5 minutes with mobile phase.
8. Monitor the fittings and tighten as appropriate. If a significant increase in pressure is observed, check if an obstruction or clogged component is present.

4.6 Connecting the Column to the Column Compartment

A removable panel on the front of the detector provides easy access to the column compartment.

Fig. 21: View into the column compartment

1. Remove the panel from the column compartment.
2. Make sure that the capillaries before and after the column are connected as described in section 4.5 (→ page 50).
3. Place the column in the column compartment and route the capillary from the column outlet to the outside through one of the capillary slots.
4. Turn on column thermostatting and set the desired temperature (→ page 70).
5. Flush the column with at least 200 mL mobile phase for several hours before connecting the electrochemical cell to the system. Solvents that may be contained in the column should be flushed with the mobile phase before initial use.

4.7 Installing the Electrochemical Cell

The detector is shipped without an electrochemical cell. Install an electrochemical cell to the potentiostat module.

Detailed descriptions on the installation for the respective electrochemical cell are included on the Installation Instructions for the respective cell.

This section provides general guidelines when installing the cell to the detector. For detailed instructions, see section 7.3 (→ page 101). The procedure for first-time installation corresponds to the installation steps when replacing a coulometric cell (→ page 107) or amperometric cell (→ page 111).

Observe the following when installing an electrochemical cell:

- Always hold electrochemical cells by the cell body with the type label. Do not touch the sensitive electronics on the cell rear side.
- Use proper electrostatic discharge (ESD) precautions to avoid performance degradation or loss of functionality.
- Shipping plugs are installed at the electrochemical cell in and out connections. Keep the plugs in a safe place. If the cells are stored for a longer time period, reinstall the plugs.
- Capillary connections between the column and electrochemical cell should be as short as possible to avoid peak broadening effects due to excessive dead volume.
- When connecting coulometric cells, make sure not to invert input and output.
- Amperometric cells are shipped without working electrodes. Working electrodes must be ordered separately and installed before the amperometric cell can be connected to the installed potentiostat module.
- Inspect the connector on the cell body and ensure that there are no bent pins that may cause damage during the installation.
- If the system contains metals, such as stainless steel, make sure that you passivate instruments or components before connecting the cell to the system. For further information about passivation, refer to section 10.1 (→ page 137).

⚠ **Important:** Failure to passivate instruments or components that contain metals, such as stainless steel, may irreversibly damage the electrochemical cells.

⚠ **Important:** La non passivation de la chaîne et des composants qui contiennent du métal, tel l'acier inoxydable, peut irrémédiablement endommager la cellule électrochimique.

4.8 Equilibration

4.8.1 Equilibrating the System

Before using the detector for sample analysis, equilibrate the UltiMate 3000 system:

1. Pump the starting solvent through the entire system until the system is free of any other liquid composition.
2. Heat or cool all temperature-controlled devices, such as the column oven, to the temperature required for the application.
3. Monitor the pump pressure. Verify that the reading is correct for the application and is stable.
4. Perform an equilibration of the electrochemical cell (→ page 55).

To equilibrate the system from Chromeleon

- Select and perform the operating commands and parameters from the **Commands** (F8) dialog box.
- Create and run an equilibration program to automate the process (→ page 68).

The equilibration panel shows the equilibration status of each instrument in the system.

4.8.2 Equilibrating the Electrochemical Cell

The cell should be allowed to stabilize (for the background currents to decay) to a reasonably flat baseline prior to attempting an analysis. This stabilization could take as little as a few minutes for less sensitive analyses to periods of hours for highly sensitive analyses.

The amount of time needed for the cell to equilibrate a steady baseline is dependent on a number of factors such as the nature and purity of the mobile phase, the potential applied to the cell and especially the level of sensitivity needed for the analysis.

1. Make sure that one or more electrochemical cells are connected to the detector (→ page 54).
2. In Chromeleon, set the potential to the value(s) required for the application.
3. Start the data acquisition (→ page 74).
4. On the panel tabset, select **More Options**.
5. Click **Start Equilibration** for each cell that you want to equilibrate. Make sure that the respective cell is selected and configured on the configuration pages (→ page 37).

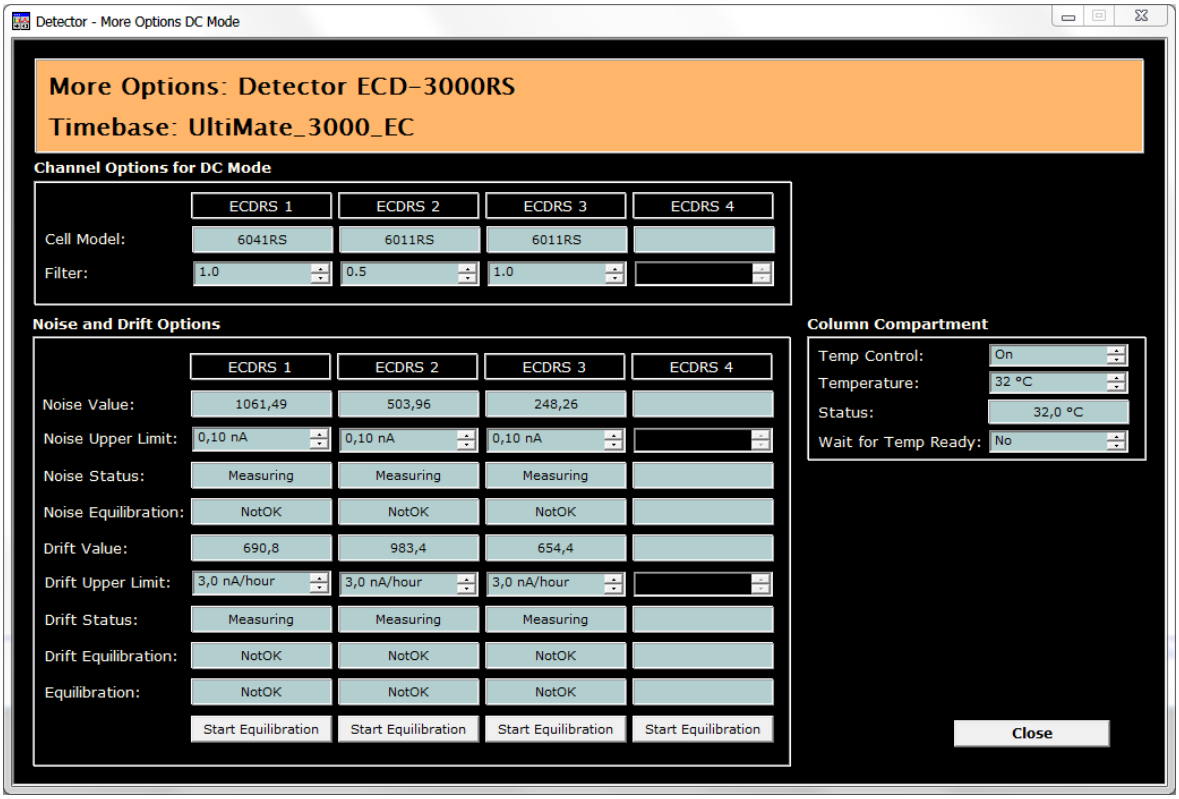


Fig. 22: Equilibration of electrochemical cells

Alternatively, you can execute the **Start Equilibration** command in the **Commands** (F8) dialog box, for each cell separately.

6. Monitor the current for each cell. Initially the current from the working electrode will be high. Over time, this current decays exponentially. Once equilibrated, the baseline signal of the cell should be stable and the noise should be diminished. The period of time required for equilibration depends on the desired sensitivity:

   - Coulometric cells should be equilibrated for at least 1 hour.
   - Amperometric cells should be equilibrated for at least 4 hours after the initial assembly for routine analysis. If the amperometric cell is to be used in very low potential ranges it is better to let the cell equilibrate longer.

7. When the equilibration of the cell(s) has been successful, values are displayed in the corresponding (“Measuring”) boxes in the equilibration window for the cells.

4.9 General Guidelines for Detector Operation

The following sections offer general guidelines for detector operation. For information about how to optimize the detector performance, see section 5.7.1 (→ page 81).

4.9.1 Mobile Phases

Mobile phase quality significantly affects detection limits and instrument performance. A careful consideration in the selection of the components of the mobile phase will be extremely useful in minimizing baseline noise and optimizing the performance during analysis. This section describes general guidelines for the use of the mobile phase with the detector.

To ensure optimal performance of the detector, observe the following guidelines:

- Prepare all mobile phases with HPLC-grade (or better) solvents, reagent-grade chemicals, and filter HPLC-grade water with a 0.2 µm membrane filter. Usually, HPLC-grade solvents will provide good results.
  This is particularly important when using coulometric cells, as particulates can clog the porous electrodes.

- The quality of the water used in electrochemistry is extremely important, as it is typically the most commonly used solvent for reverse-phase applications. Although ultra-pure water typically with a resistivity of 18.2MΩ is preferred, it is also important that the water be free from microbial growth and other organic contaminants that tend to increase the total organic content (TOC). High TOC levels can lead to reduced separation performance of the analytical column, increased background currents and reduced sensitivity of the electrochemical cell, as well as ghost peaks on the chromatogram.

- Use mobile phases containing water, solvents and modifying reagents of the highest purity available. Make sure that the mobile phase does not contain any electroactive impurities to achieve optimum sensitivity. The amount of impurities in the mobile phase has an influence on the amount of time needed for the electrochemical cell to equilibrate a steady baseline and the background current produced.

- Degas all mobile phases before use.

- A supporting electrolyte should be used for potential control. The electrolyte concentration should be in the order of 20-100 mM to provide a suitable level of electrolyte in solution.

- Ensure that the mobile phase is not contaminated by metal system components. For details, see page 59.

- Always use a buffered mobile phase.
  The concentration of buffers should be kept between 50 and 100 mM to minimize the background current and baseline drift while maintaining constant pH value.
  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. Do not allow buffers to remain in the cell without flow for extended periods. Cells should not be allowed to dry containing a mobile phase with buffers. For storage they should be flushed with at least 20 % methanol.

- Know the oxidation and/or reduction potential of each component of all mobile phases that you are using. The components should not be electroactive at the analytical potential. The practical limit is 50 mV below the oxidation potential of the most easily oxidized component in the mobile phase.

- The stability of the mobile phase may decrease over a period of time. Use freshly prepared mobile phase for the analysis.

4.9.2 Mobile Phase Delivery System

The pumping system should deliver continuous flow while ensuring intermixing of the mobile phase (if gradient elution is used). Fluctuations in pump backpressure can cause baseline noise and may result in reduced performance of the electrochemical cells. If the noise is synchronized with the pump stroke, check your HPLC pump.

For electrochemical operation, these guidelines are recommended:

- The mobile phase reservoir should be glass. In some applications, however, it may be necessary to use plastic solvent reservoirs. In carbohydrate applications, for example, PTFE solvent reservoirs should be used to prevent carbon dioxide build up in the mobile phase.
- All tubing connections should be made of materials suitable for HPLC electrochemistry (for example, PTFE, ETFE, PEEK, passivated stainless steel, or titanium), as required for the operating pressures and application.
- Any steel component in the flow path can have corrosion sites that will negatively influence the background currents and facilitate auto-oxidation of electroactive compounds. Therefore, minimize the number of metal components in the HPLC system. When using the detector in a system that contains metals, such as stainless steel, in the flow path, passivate the instruments and components before installation of the electrochemical cells. For further information about passivation, refer to page 137.
- In biocompatible systems with Titanium components, solvents with a pH value >12 that are exposed to this metal can form oxides that influence the background current when using elevated pH conditions. Before starting application work with solvents with a pH value >12, observe the following for handling the pump:
  - Titanium mixer and filters at the outlet of the pump must be removed. Titanium frits must be replaced with PEEK frits.
  - The pump should be passivated before use. For further information about passivation, refer to section 10.1 (→ page 137).
- Always use in-line filters from the In-Line Filter Kits to ensure that particulate matter does not enter the cell, as particulate matter may clog the cell and lower system performance and/or create backpressure (→ page 50).

4.9.3 Electrochemical Cells

- Never run the electrochemical cell dry while applying potential to the electrodes, as this may damage the electrodes. Make sure that the EC compatible mobile phase flow is established before the cell is turned on and that it is maintained whenever potential is applied to avoid permanent damage to the cell.
- The parts of the electrochemical cell that are exposed to solvents may be made of PEEK (polyetheretherketone), porous graphite, palladium, boPET (biaxially-oriented polyethylene terephthalate), boron-doped diamond, glassy carbon or PTFE (polytetra-fluorethylene). The chemical resistance of an electrochemical cell depends on the solvents used for the analysis. This applies particularly to strongly acid solvents with high buffer concentrations and certain solvents, such as THF (tetrahydrofuran) and $CHCl_2$ (dichloromethane).
- Do not expose the electrochemical cells to mobile phases with high molar concentrations of nitric acid (> 5M HNO_3).
- Always flush the instruments in the system upstream of the detector before connecting them to the electrochemical cells.
- Operate electrochemical cells only in the specified range of their operating pressure limits. For information about the pressure range of a cell, see the cell specifications provided with the cell or the information provided in the **Commands** (F8) dialog box in Chromeleon (→ page 72).
- The backpressure on the electrochemical cell must not exceed the specified pressure limits of the cells.
- The contacts for the electrochemical cell identification chip are located on the rear of the cell. To ensure proper functionality of the detector and the cells, use proper electrostatic discharge (ESD) precautions. If liquid comes into contact with the electronics, dry it immediately.
- Construct a hydrodynamic voltammogram for each new cell to determine its optimal potential for the desired application. For further information, see section 5.6.1 (→ page 75).
- Monitor background currents. Changes in the background current may be indicative of a possible problem. For further information about background currents, refer to section 5.6.3 (→ page 78).
- Be sure to turn off the cell and remove it from the detector when chemically cleaning it or any other component of the system.
- Do not apply potentials to the cell if no electrolyte is present in the mobile phase and during organic cleaning or aqueous washing procedures.

- The cells are sensitive to dirt and dust. Therefore:
  - Always keep unused electrochemical cells in their original dust-free packaging.
  - Even during periods of detector inactivity, ensure that the detector front cover is closed and the bays are closed with a cover or a potentiostat module is installed to prevent dust from entering into the cell bay and reaching sensitive electronics.
  - When shipping the instrument, *remove* the electrochemical cell and the potentiostat module and install the special cover to protect the bay opening. Close the cell inlet and outlet with the plugs that were installed when the electrochemical cell was shipped. The cell must be shipped in its original packaging.
- During longer idle times, the following is recommended:
  - *Coulometric cells*
    Flush the cells with 20 % methanol, and plug the cell input and output using the plugs that were installed when the cell was shipped. Using different plugs and tightening them may destroy the cell.
  - *Amperometric cells*
    Flush the cells with 20 % methanol. Disassemble the cell, and clean the flow surfaces with isopropanol. Rinse the working electrode with isopropanol and water and dry it. Store the working electrode in the packaging that it was shipped in.
- If a cell leaks, remove the cell from the detector as quickly as possible and take remedial action:
  - *Coulometric cells*
    Replace the cell (→ page 107).
  - *Amperometric cells*
    Replace the gasket and/or the working electrode (→ page 114 and 117). If the cell is still leaking, replace the cell (→ page 118).
- For information about how to clean and service an electrochemical cell, see page 101.
- Remove the electrochemical cell from the detector when connecting a new column. Allow the column to flush for several hours to remove particles from the column before re-attaching the cell.
- If a problem occurs in the system, first check other components of the system before making the conclusion that the problem is a result of the cell.

For information about the operation of electrochemical cells, refer to section 5.5 (→ page 72).

5 Operation and Maintenance

For information about how to control the detector via the Chromeleon Chromatography Management System, see section 5.3 (→ page 65).

5.1 Power-Up

To start the detector for the first time, turn on the main power switch on the rear panel of the detector. The following sequence of events occurs when the detector is powered up:

- For a short time, general information about the detector appears on the display: device type, firmware version, and serial number.
- The detector runs a series of internal tests. (The test time depends on the number of potentiostat modules installed. With one potentiostat module installed, the self test takes approximately 2 minutes). During these self-diagnostics, all of the main components are checked. As the self test is performed, the display shows the status of the test.
  - ♦ When the self test was successful, the **Status** LED on the front panel changes from red to green.
  - ♦ If an error is detected, the detector is not ready for analysis. The **Status** LED on the front panel turns red. If the detector is operated with Chromeleon, the message appears in the Chromeleon Audit Trail. Remove any cells that are installed. Turn off the detector, take appropriate remedial action (→ page 89), and turn on the detector again. If the detector repeatedly fails the self test, note the exact wording of the error message displayed in the Chromeleon Audit Trail and contact Thermo Fisher Scientific Service for Dionex HPLC Products.
- Make sure that you establish the desired mobile phase flow before turning on the cells.

For routine operation, leave the main power switch on. Turn the main power switch off when instructed to do so, for example, before performing a service procedure.

5.2 Status Screen

When the self test was successful, the initial screen changes to the status screen.

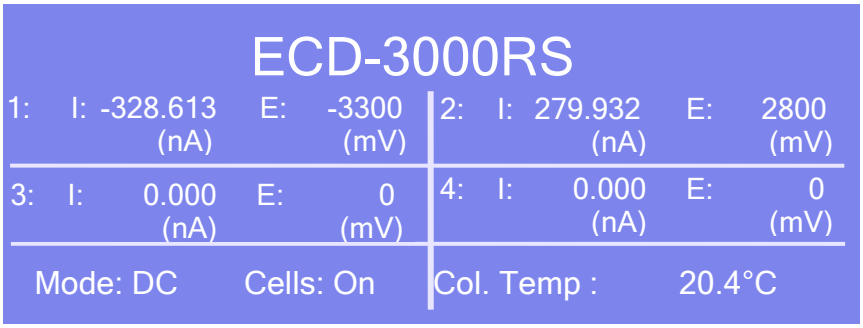


Fig. 23: Status screen (example)

The status screen shows the following information:

- Signal unit (measured sensitivity)
- Applied potential
- Mode
- Cells on/off
- Column compartment temperature

5.3 Chromeleon Software

Before you begin, verify that

1. The Chromeleon software is installed on the computer and the license code is entered. The computer meets the system requirements (→ page 8).
2. The 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 detector is set up in Chromeleon, as described in section 3.5 (→ page 34).

Before you can operate the detector with Chromeleon, you have to connect the timebase in which the detector is installed to the Chromeleon client program (→ page 65).

Two modes of software control are available:

- **Direct control** with the parameters and commands from the **Commands** (F8) dialog box (→ page 66) or from a control panel (→ page 67).
- **Automated control** with a control program (PGM) (→ page 68).

5.3.1 Connecting to Chromeleon

1. Start the Chromeleon **Server Monitor** and the Chromeleon server if they are not yet running (→ page 34).
2. Start the Chromeleon client by clicking the Chromeleon icon  on the desktop. If the Chromeleon icon is not on the desktop, click **Start** on the taskbar, point to **Programs** (or **All Programs**, depending on the operating system), point to **Chromeleon**, and then click **Chromeleon**.
3. Connect the Chromeleon client program to the timebase in which the detector is installed. For details about how to do this from the **Commands** (F8) dialog box, see page 66. For details about how to do this on a control panel, see page 67.

When the detector is correctly connected to Chromeleon

- The **Connected** LED on the front panel is green.
- Functions for estimating the lifetime of consumables and monitoring and recording service and (re)qualification information are provided (→ page 82).

Before turning off the detector by the main power switch, always **disconnect** the module in Chromeleon.

5.3.2 Direct Control

With direct control, you select operating parameters and commands in the **Commands** (F8) dialog box. Direct commands are executed as soon as they are entered. For routine operation, most parameters and commands are available also on a control panel.

To open the Commands dialog box for the detector

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 **ECDRS**.

   The commands and parameters available in the dialog box vary, depending on the
   - Chromeleon version
   - Options selected for the detector in the Properties dialog (→ page 37).
   - Display filter level (**Normal**, **Advanced**, or **Expert**)
5. Change the display filter level if necessary. Right-click in the commands list and select the filter level on the menu.

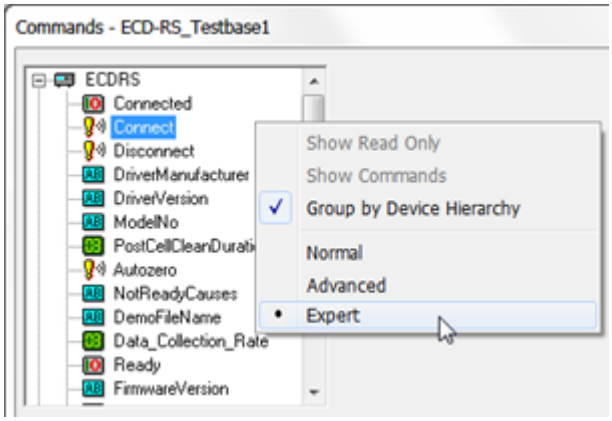


Fig. 24: Commands dialog box

6. Verify that the detector is connected to Chromeleon. If it is not, select **Connect** to connect the detector.

For a list of the commands and properties that are supported for the detector, see the Chromeleon Help. In addition to the detector commands and parameters, the **Commands** (F8) dialog box provides access to all of the commands and parameters available for all devices that are installed in the selected timebase.

To open a control panel

1. On the **View** menu, click **Default Panel Tabset** or click the corresponding icon on the toolbar , 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 detector.

3. Verify that the detector is connected to Chromeleon (the LED next to the Connect button is green). If it is not, click **Connect**.

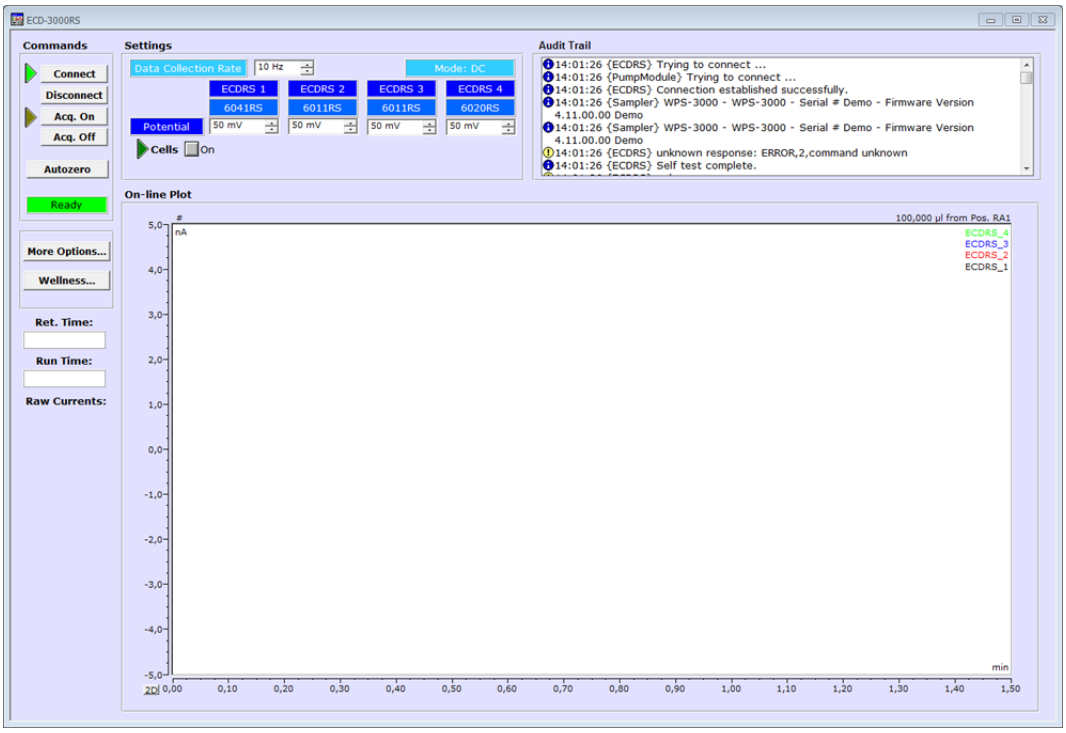


Fig. 25: Detector control panel

The control panel provides access to the operating parameters and commands required for routine operation of the detector. Additional functions are available in the **Commands** (F8) dialog box. To open the **Commands** box from the panel tabset, select **Command** on the **Control** menu.

5.3.3 Automated Control

With automated control, you create a program file (PGM) for automated operation of the detector. Programs can be created automatically with the help of a software wizard or manually by editing an existing program.

In addition to programs for sample analysis, you can also create programs for special purposes, for example, to automate system shutdown (→ page 81) or to ensure that the system automatically restarts operation as desired after a power failure. For details, see the Chromeleon Help.

To create a program with the Program Wizard

1. Open the Program Wizard. On the **File** menu, select **New**, and then select **Program File**.
2. The wizard guides you through program creation. On each wizard page, make the desired settings or accept the default values. For additional information about a page, click **Help**.
3. After you finish the wizard, Chromeleon automatically creates the corresponding program.
4. To start the program, follow the steps below.

To create a program manually

1. Open an existing program.

   Select and double-click the program you want to open.
   - or -
   On the **File** menu, select **Open**. In the dialog box, select **Program** on the **Object of Type** list and select the program.

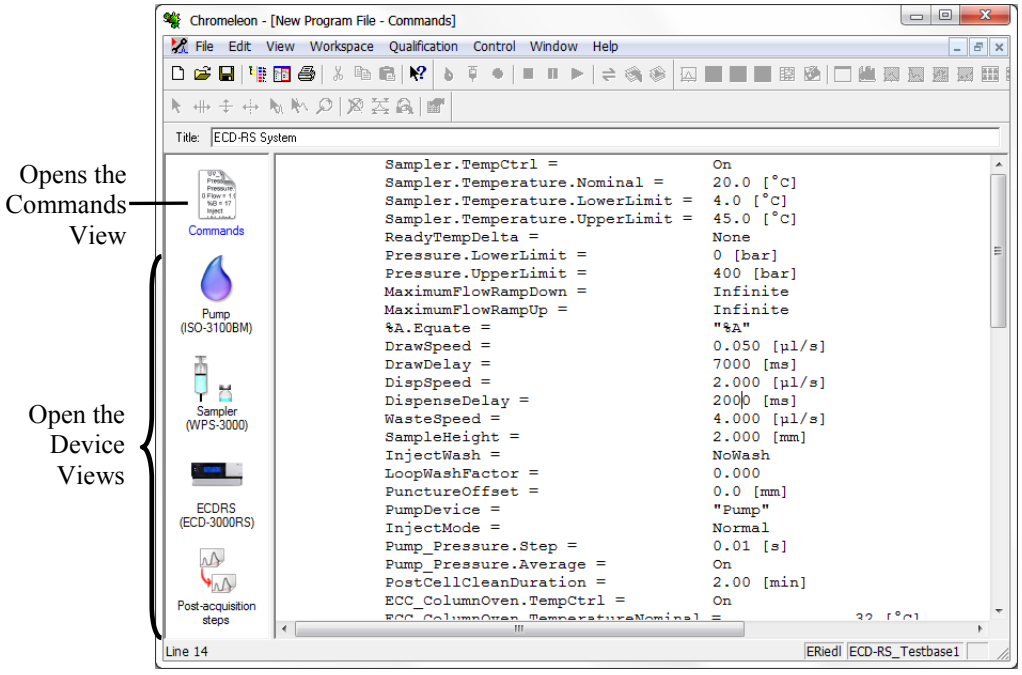


Fig. 26: Chromeleon program file (here program shown in the Commands view)

2. Change the settings in the program as desired.

   The easiest way to edit a program is to do this in the Device Views (→ Fig. 26). Click a device icon and change the settings on the device pages. Editing the program in the Device Views ensures correct command syntax.

   If you cannot edit a certain parameter in the Device View, click **Commands** to open the Commands View. The **Commands** view shows the entire program, listing the control commands in chronological order. For more information, see the Chromeleon Help.

3. To start the program, follow the steps below.

To start a program

Program for sample analysis

1. Create a sample list (sequence). A sequence must include the program and a method for evaluating the sample data (for example, for peak identification, area determination, and amount determination).
2. Assign the program and method to each sample on the list.
3. Add the sequence to the batch and start the batch.

For information about each of the above steps, see the Chromeleon Help.

Other programs

Add the program to the batch and start the batch.

5.4 Operational Settings

This section provides information for operating the detector.

To learn more about ...	See page ...
Turning On Column Thermostatting	See below
Detecting liquid leaks	71

In addition, note the information about special functions that are available for the detector in Chromeleon (→ page 75).

5.4.1 Turning On Column Thermostatting

You can turn column thermostatting on and off and set the desired temperature in Chromeleon.

To turn on column thermostatting from Chromeleon

1. In Chromeleon, open the **Commands** (F8) dialog box for the detector.
2. Select **ColumnOven** and **Temperature**.
3. Under **TemperatureNominal**, enter the desired temperature. Entering a temperature sets **TempCtrl** to **On** if it is not yet running.

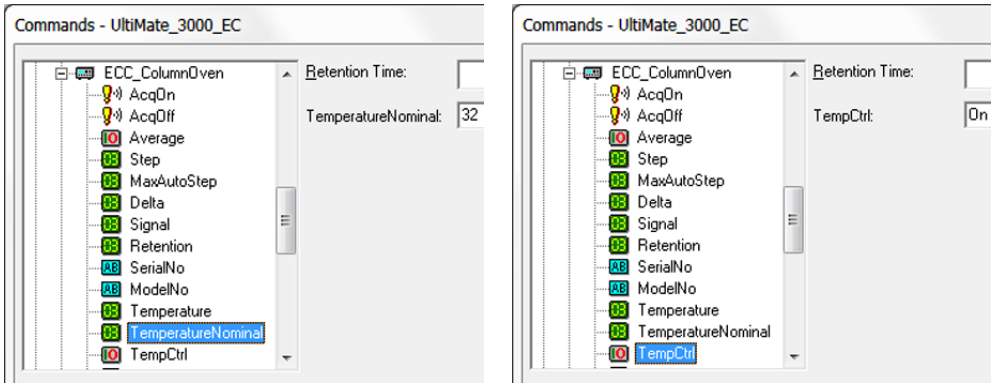


Fig. 27: Turning on column thermostatting

Set **TempCtrl** to **Off** if you do not want to use column thermostatting for a certain application.

If you want to activate column thermostatting later again, set **TempCtrl** to **On**. When you change the temperature setting under **TemperatureNominal**, Chromeleon sets **TempCtrl** automatically to **On**.

5.4.2 Detecting Liquid Leaks in the Detector

Leak detection is enabled as a standard when the detector is shipped. When leak detection is active and the leak sensor reports a leak

- The **Status** LED on the front panel door is red.
- A message appears in Chromeleon in the Audit Trail.
- The cells are turned off.
- The running batch is aborted.

When the leak sensor reports a leak, eliminate the cause for the leakage and dry the leak sensor (→ page 123).

5.5 Operation of Electrochemical Cells

This section provides information about the operation of electrochemical cells. To optimize the detector performance, refer to section 5.6 (→ page 75).

For maintenance and service of electrochemical cells, refer to section 7.3 (→ page 101).

For further information about the available cells, refer to page 21.

5.5.1 Direct Current Mode

The detector is operated in a direct current mode (DC mode). The current resulting from the oxidation or reduction of the analyte is measured at a constant potential.

Autozero Current

Chromeleon supports the **Autozero** function to recalibrate the zero current.

When performing the **Autozero** command from Chromeleon, the detector records the values of all signal channels, and continuously subtracts them from each of the signal(s) values. As a result, the signals on all channels are set to zero. The signals remain at zero until a chromatographic event causes a change in the baseline (for example, an analyte peak eluting from the column or a baseline drift and/or noise). The **Autozero** function does not affect the baseline current that is shown on the detector display.

Typically, the **Autozero** command should performed before an injection.

Perform an **Autozero** only when the baseline is stable and flat. If the baseline is still stabilizing during equilibration, is noisy or in the middle of an event (for example, in the middle of a peak), wait until the baseline is reasonably flat and the signal is low for at least 15 seconds.

For optimum performance, perform the Autozero command with the cells off on a weekly basis.

5.5.2 Setting the Potential

Before setting the analytical potential, make sure that you observed the guidelines (→ page 60) and the potential ranges for the electrochemical cells (→ see below).

⚠ **Important:** The mobile phase flow through the cell must be established whenever potential is applied to avoid permanent damage to the cell.

⚠ **Important:** Pour éviter tout dommage irréversible sur la cellule un débit doit être appliqué à la phase mobile phase dès qu'un potentiel est appliqué sur la cellule.

On the tabset panel for the detector, set the desired potential, or in the Chromeleon **Commands** (F8) dialog box, click the plus sign for the respective electrochemical cell to see the commands available. Enter the desired potential and retention time.

The potential ranges for the cells can be obtained from the **Potential** property of each cell in the **Commands** (F8) dialog box in Chromeleon.

After a potential has been applied to the cell, the background current will reach a maximum value and then rapidly decay before slowly decaying to a stable value. The rapid current decay is predominantly due to the charging current whereas the slower decaying current is associated with the equilibration of the system. The exact steady-state baseline current depends upon the applied potential, type and condition of the mobile phase (age, source of reagents, amount and type of organic modifiers, etc.), temperature and condition of the HPLC system. The background current is application specific. However, if a particular method has been running, the currents of the new cell should be similar to what has been observed in the past.

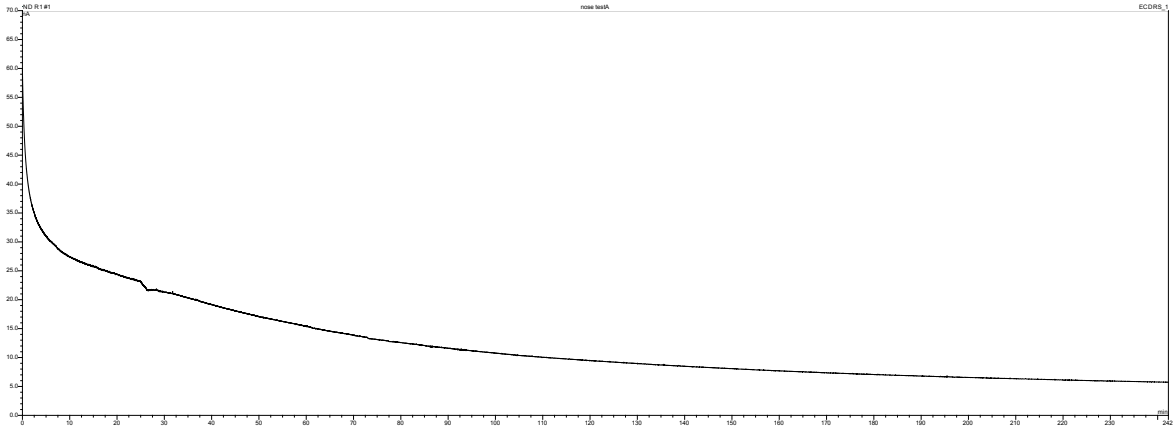


Fig. 28: Typical background current after equilibration
(Here: Amperometric Cell 6041RS with glassy carbon electrode, mobile phase MDTM 0.5 mL/min, applied potential of 300 mV)

Monitor the baseline when the desired method is run. Wait until a quiet/flat baseline has been established. The time period that is required to obtain a quiet/flat baseline depends on the sensitivity desired. A longer time will be required for situations where maximum sensitivity is desired.

Allow Research Potentials

This function allows you to apply potentials to an electrochemical cell beyond the specified normal potential range of that cell. This may be necessary for certain applications. However, consider the following when using this function:

- The cell performance may be diminished.
- The lifetime of the cell may be shortened.
- The electrodes may be damaged.

5.5.3 Starting and Stopping Data Acquisition

You can start and stop data acquisition in Chromeleon. In addition, you can watch the progress of data acquisition on the display.

To start or stop data acquisition in Chromeleon

1. Open the **Commands** (F8) dialog box for the detector.
2. Set the analytical potential (→ page 72).
3. Perform the **AcqOn** command to start data acquisition.
   Perform the **AcqOff** command to stop data acquisition.

In Chromeleon up to 4 data signal channels can be viewed and stored.

Monitoring the progress of data acquisition on the detector display

You can watch the progress of data acquisition on the display. The display shows the applied potential (mV) and the sensitivity (nA or µA).

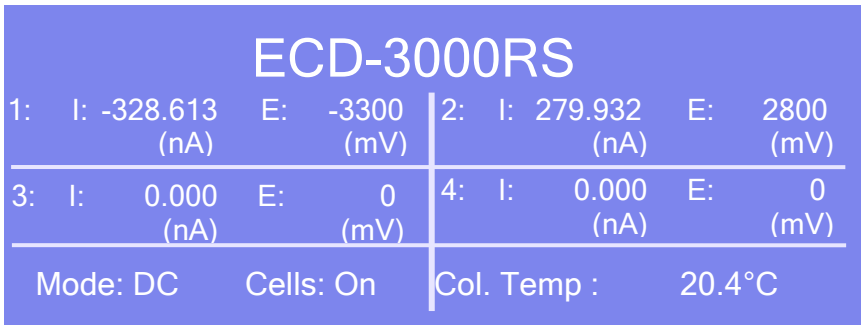


Fig. 29: Data acquisition screen (example)

The status screen shows the following information:

- Measured sensitivity (nA or µA)
- Applied potential (mV)
- Mode (DC)
- Cells on/off
- Column compartment temperature

5.6 Optimizing Detector Performance

The performance of the detector can be optimized by careful selection of key operating parameters. The table summarizes these parameters, indicates the performance characteristics affected, and offers guidelines for selecting the parameters.

Operating Parameter	Performance characteristics affected	Selection guidelines
Analytical Potential	Selectivity, baseline noise, sensitivity	→ see below
Filter Constant	Sensitivity, baseline noise	→ Page 77
Background current	Sensitivity, reproducibility, baseline noise	→ Page 78
Data Collection Rate	Peak resolution, disk space, possibly baseline noise	→ Page 80
Selectivity		→ Page 80

In Chromeleon, you can set operating parameters in the **Commands** (F8) dialog box for the detector (→ page 66) and on the page for the detector of the **Panel Tabset** (→ page 67). Note that you may need to change the Display filter level to see all operating parameters (→ section 5.3.2, page 66).

5.6.1 Analytical Potential

The appropriate potential for an analysis is the potential that provides the largest signal for the oxidation (or reduction) of an analyte while minimizing the signal from interferences (for example, electroactive compounds that co-elute with the analyte or the mobile phase itself).

The use of an electrochemical detector for liquid chromatography requires knowledge of the potential to effect the desired electrochemical reaction (oxidation or reduction of the species of interest). This potential depends on many factors including the type of electrochemical cell used, the working electrode, pH value, composition of the mobile phase and the chemical structure of analyte. For more information about these factors, see further down in this section (→ page 77).

Optimizing an Analytical Potential

Hydrodynamic Voltammogram (HDV)

A hydrodynamic voltammogram (HDV), often referred to as a current-voltage (CV) curve, is a plot of the current (signal or response) produced when an electrochemically active compound undergoes electrolysis at the working electrode as a function of the applied potential. The information contained in an HDV is used in selecting the optimal applied potential to the working electrode for the detection of an analyte under a given set of chromatographic conditions. A primary goal of an HDV is to determine the lowest practical applied potential for detection of your analyte of interest.

When a system is initially set up, a new electrochemical cell is installed, or a new analytical method is set up, run a hydrodynamic voltammogram to determine the optimal applied potential. A well characterized HDV curve provides the best applied potential in order to maximize the signal, and minimize the baseline.

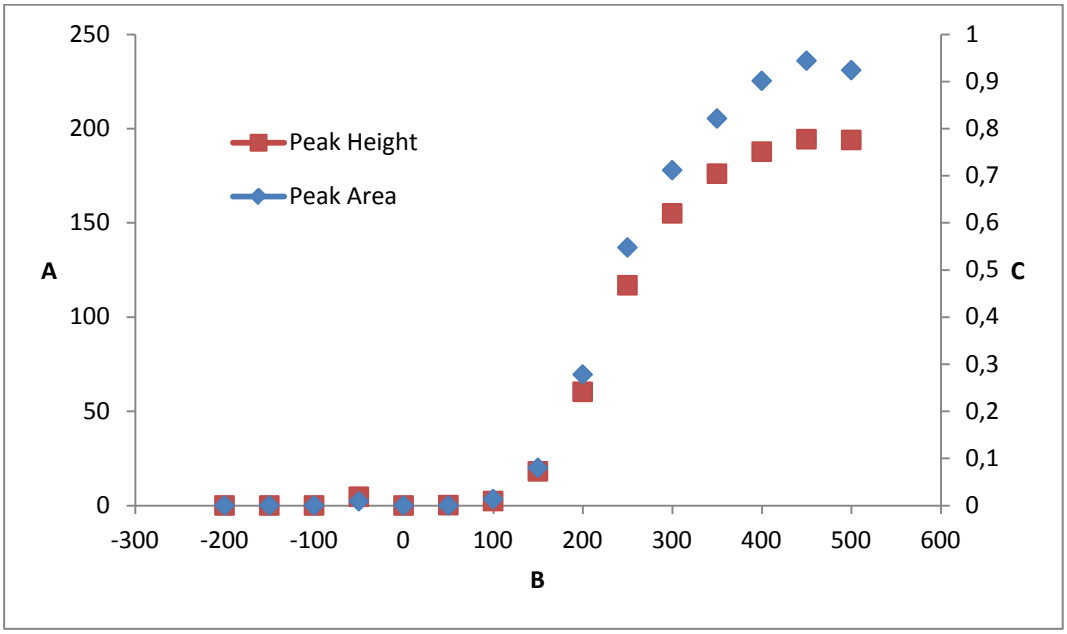


Fig. 30: HDV of 3.4 dihydroxybenzylamine detected with an amperometric cell 6041RS (example)

No.	Description
A	Peak height of DHBA, nA
B	Applied potential, mV
C	Peak area of DHBA, nC

As shown in the example in Fig. 30, the optimal potential would be set just short of the curve plateau. Some adjustments may be required depending on the matrix of the sample. Do not set the applied potential too high otherwise other coeluting compounds with similar redox potentials will also react.

Generate an HDV by injecting the compound of interest at a constant concentration and plotting peak response vs. applied potential. As an example, apply the potential in 100 mV incremental steps, allowing sufficient time for equilibration between the injections.

Guidelines for an Optimum Potential

Consider the following factors for an optimum potential:

- The best applied potential is typically obtained by generating an HDV curve and choosing a potential at which the signal just begins to plateau. The result is a maximum signal response by selecting the lowest applied potential possible.
- Typically the chromatographic conditions need to be finalized before the final detector settings are determined. It should be noted that factors which affect the separation (e.g. the ionic strength and the organic modifies) can alter the electrochemical characteristics of the analyte.
- The mobile phase and buffer solutions should not contain components that are oxidized or reduced at the analytical potential. Maintain a potential difference of 50 mV between the compound of interest and components in the mobile phase, if possible.

5.6.2 Filter Constant

The filter time constant is used to electronically reduce the noise in the chromatogram. A small filter value (e.g., 0.2 sec) removes little noise, while a large value (e.g., 10 sec) will perform a significant amount of smoothing. Although a large value for the filter presents a very smooth chromatogram, it might be possible that small peaks are eliminated in the smoothing process. On the other hand, use of a very small filter setting leads to a very noisy chromatogram.

To set the filter constant, open the Chromeleon **Commands** (F8) dialog box, and click the plus sign for the respective electrochemical cell to see the commands available. Under **FilterConstant** enter the desired filter constant. Alternatively, you can set the filter constant on the detector panels.

As a general rule of thumb, the filter should be set to a value that is 1/4th to 1/16th the base width of the narrowest peak (in seconds) to achieve no peak reduction. Higher filter times can be used. Note that as long as standards and samples are all run under the same conditions, the selection of the filter value will not affect analytical results, so long as the peak is clearly observable. If the peaks of interest are very sharp and occur soon after injection, a short filter time (2 seconds, for example) is advised. A filter of 5 seconds is sufficient for most applications. A large filter time constant may attenuate the peak height, especially if the peak width is small. Use smaller filter times for the first few minutes of a chromatographic run and then larger filter times later in the run.

5.6.3 Background Current and Baseline Noise

Baseline noise is a function of the background current. Background current can be either faradic (obeying Faraday's Law) or non-faradic. Electrolysis of contaminants in the mobile phase is a typical source of faradic current. Non-faradic currents include charging currents (when applied potential is changed), electronic noise, noise from connections, and thermal effects, etc.

The background current will typically be high immediately after the potential is applied to the working electrode. This current is primarily a result of the capacitive effects after a change in the applied potential. The current will fall rapidly as the electrodes stabilize.

If a higher potential is used, the resulting higher background current may shorten the useful lifetime of the working electrode.

Causes

Baseline noise and background current depend on a number of factors:

- The applied potential. The greater the applied potential, the greater the background current and baseline noise. Mobile phases with high organic content will typically have lower background current for a given potential than highly aqueous mobile phases. Those with high concentration of electrolytes will typically have higher background currents. Make sure that all mobile phase components are compatible with the applied potential being used.
- The quality of water and the organic modifier (for example, methanol, acetonitrile) used in the mobile phase (→ page 57). Make sure that the highest quality is used, and that contaminants are kept to a minimum.
- The quality of the salts used in mobile phase production. Use the highest quality with minimal transition metal contamination. Electroactive impurities of as little as 0.001 % may mean that the resulting background current will adversely affect high sensitivity analyses.
- System components. All fluidic components should be inert with minimal metal surfaces. Many typical HPLC components contain metal surfaces that can bleed redox active transition metals into the mobile phase causing high background currents and promotes auto-oxidation of analytes which may damage the cell. Passivate such components routinely. For further information about passivation, refer to section 10.1 (→ page 137).
- The analytical column. New columns must be flushed to waste for several hours or overnight before attaching the electrochemical cell in order to remove metal contaminants resulting from the end frits, column body and remaining catalysts from the synthesis of column particles. Test the contribution of the column by monitoring the background current.

- The cleanliness of glassware used to make the mobile phase. Make sure that the glassware is totally dry. Cover open vessels with aluminum foil to prevent entry of dust, etc. Do not use soap as any residue left on the glassware surface can dissolve into the mobile phase and act as an ion-pairing agent.
- The growth of microbes in mobile phases that contain low levels of organic modifiers. Use lithium salts or if unavailable, use Reagent MB (typically 100 µL/L of mobile phase) when preparing mobile phases with < 3 % organic modifier to prevent microbial growth. For information about the Reagent MB solvent, contact the Thermo Fisher Scientific sales organization for Dionex HPLC products.
- Recycling. Especially when measuring dirty biological samples and/or the use of high concentration standards recycling the mobile phase can cause background current and baseline noise.
- Not degassing the mobile phase sufficiently. This becomes a problem at higher oxidation and reduction potentials.
- For optimum operation, use a biocompatible pump, such as the ISO-3000BM pump of the UltiMate 3000 series.

Marked changes in the background current may be an indication that something is wrong with the HPLC system.

Minimizing Background Current

The background current and baseline noise can be reduced by observing the following recommendations:

- Decreasing the electrochemical potential slightly if the setting is near the plateau of the HDV curve.
- Minimizing the impurities in the mobile phase. For further information, refer to page 57.
- Use a dedicated column for each specific analysis to avoid cross-contamination of the column.
- When operating at extremely high or negative potentials take care to effectively degas the mobile phase.
- Ensure that the HPLC system does not contaminate the mobile phase. Minimize the number of stainless steel components in the HPLC system. Corrosion sites on metallic components of the system may contribute to higher noise and loss of signal.
- Allow sufficient time to achieve a stable baseline before performing an analysis.
- Limit the recycling of mobile phase, especially when working with impure samples at high concentrations.

- The background noise will increase at higher applied potentials due to the oxidation or reduction of components in the mobile phase, such as the electrolysis of water. This is dependent on the working electrode material, composition of the mobile phase and the applied potential. Know the oxidation and/or reduction potential of each component in the mobile phase. These components should not be electroactive at the analytical potential.

5.6.4 Data Collection Rate

The data collection rate is the number of data points per second (Hz) that Chromeleon collects from the detector and stores as raw data. Note the following guidelines:

- You can generate data with a maximum collection rate of 100 Hz or 200 Hz (200 Hz under Chromeleon 7.1).
- In general, define each peak by at least 20 data points. For chromatograms with co-eluting peaks or low signal-to-noise ratios, 40 data points per peak is recommended.
- If all peaks are relatively wide, select a slower data collection rate (for example, 1.0 Hz). This saves disk space and allows for a faster display of data in Chromeleon.
- If the data collection rate is too slow, the start points and end points of peaks will not be determined accurately.
- If any peaks of interest are less than a few seconds, select a faster data collection rate (20.0 Hz, for example). If the collection rate is too high, data files may need more disk space and post-run analyses may require more processing time.

5.6.5 Selectivity

If several electroactive species are present in a sample, the chromatographic separation is normally developed so that only one compound is presented to the detector at a given instant. If this is the case, the potential should be set to the value which provides the maximum response for the compound that is of primary interest.

In general, if more than one compound in the sample is of interest and good chromatographic resolution is obtained, the potential can be set to the optimal potential of the compound requiring the highest potential. The resulting response is relative to the abundance of each analyte of interest, and it may be necessary to optimize the applied potential to take this into account.

If two (or more) electroactive compounds coeluted, use a coulometric cell with dual electrodes and set the potential to optimize the current from the compound of interest while minimizing the current from the interferent. If, for example, the limiting current for a compound of interest is observed at 550 mV, but a trace component that co-elutes with the primary compound had an oxidation potential of 775 mV, it may be necessary to use a slightly lower value for the potential.

5.7 Monitoring System Functions

This section provides a short overview of some special functions for qualification, documentation and monitoring the system that Chromeleon supports for the detector. Some functions are available also on the control panel for the detector. For additional information about a function, see the *Chromeleon Help*.

To learn more about ...	See page ...
Recording the Column Compartment Temperature	See below
Predictive performance	See below
Operational Qualification and Performance Qualification	82

All of these functions are available in the **Commands** (F8) dialog box (unless otherwise noted). In addition, some functions are available also on the control panel for the detector. For additional information about a function, see the *Chromeleon Help*.

5.7.1 Recording the Column Compartment Temperature

On the **Signals** page, the **ECC_ColumnOven** check box is selected by default when the detector is installed and configured in Chromeleon (→ page 42). With this setting, Chromeleon generates the appropriate channel for recording the column compartment temperature. The channel is then available in the **Commands** (F8) dialog box for the detector.

5.7.2 Predictive Performance

Predictive Performance provides various functions for estimating the lifetime of consumables and for monitoring and recording service and (re)qualification information.

SmartChip Technology

SmartChip technology provides automated cell identification and documentation with identification chips that are included in the electrochemical cells. You can view information about the electrochemical cells installed in the detector in the Chromeleon **Server Configuration** under **View Cell Data** (the **Cell Properties** page opens), the following information is available:

- Usage (hours)
- Integration (hours)
- Operating Time (hours, total operating time)
- Maximum current of the respective cell or working electrode
- Working electrode

The cell information on the configuration pages shows the cell properties for the last time the chips were read. Click the **Read Smart Cells** button *before* opening the **Cell Properties** page to update the cell data and obtain the latest cell information.

Cells Off Function

When a pump of the UltiMate 3000 series (except the LPG-3400XRS pump*) is properly connected to the detector using the mini-DIN cable, the cell is automatically turned off and the mobile phase flow is stopped during a run or when an error occurs. Note that the input for the Cells Off function (= **ECDRS_Input_3**) does not have to be selected on the configuration pages for the function to work. The Cells Off function is handled automatically. However, in Chromeleon, you can select the **ECDRS_Input_3** checkbox and monitor the function of this input in the **Commands** (F8) dialog box, or on the detector display.

To select the input:

1. Open the Chromeleon **Server Configuration**.
2. In the **Properties** for the detector, on the **Inputs** page, select the **ECDRS_Input_3** checkbox.
   For further information about setting inputs, refer to page 43.

* The connection to a LPG-3400XRS pump will be supported in a future Chromeleon version.

Commands (F8) Dialog Box

The **Commands** dialog box may contain more predictive performance parameters than described in this manual. For a complete list of available commands and parameters, see the *Chromeleon Help*.

5.7.3 Operational Qualification and Performance Qualification

Operational Qualification and Performance Qualification allow you to check and document the performance of the HPLC system. All materials required for performing qualification and detailed instructions are available on request.

5.8 Shutting Down the Detector

Observe the following precautions before interrupting the operation or before shipping the detector.

5.8.1 General Guidelines

- Make sure that the potential is turned off before stopping the pump flow.
- Rinse out any solvents from the cell. Observe the storage procedures for the cells (→ see below).
- Reinstall the cardboard with the notice about the shipping locks that was shipped with the detector. To do so, push the cardboard under the orange shipping locks on the detector bottom as shown and fold the other end of the cardboard around the front panel door.
- Ship the detector only in the original shipping container and observe the packing instructions. Shipping the unit in any other packaging automatically voids the warranty. If the original shipping container is not available, appropriate shipping containers and packing material can be ordered from Thermo Fisher Scientific sales organization for Dionex HPLC products. The packing instructions are included in the "Installation and Qualification Documents for Chromatography Instruments" binder and are also available on request. For more information, see the warranty statement in the terms of sale.
- *Amperometric cells only*
  Even during periods of detector inactivity, keep the cell assembled.

5.8.2 Storage of the Electrochemical Cells

Coulometric Cells

Short-term storage

For short-term storage of the cell (less than one week), follow these steps:

1. In Chromeleon, turn the cell off.
2. *If traces of salts may be present in the cell*
   Flush the cell with a mobile phase with a water/organic mixture that does not contain salts. The duration of the flushing should be sufficient to remove any trace of the application mobile phase from the cell. After the flushing is completed, stop the mobile phase flow.
3. Flush the cell with organic solvent used in mobile phase to remove water and prevent microbial growth.
4. Remove the cell from the detector.
5. Disconnect the capillaries on the inlet and outlet ports of the cell.

6. Disconnect the detector in Chromeleon.
7. Close the cell input and output using the plugs that were installed when the cell was shipped. Using different plugs and tightening them may damage the cell.

Long-term storage

For long-term storage of the cell (more than one week), follow these steps:

1. In Chromeleon, turn the cell off.
2. Flush the cell with a mobile phase of 20 % methanol and 80 % water. The duration of the flushing should be sufficient to remove any trace of the application mobile phase from the cell. After the flushing is completed, stop the mobile phase flow.
3. Disconnect the detector in Chromeleon.
4. Remove the cell from the detector.
5. Disconnect the capillaries on the inlet and outlet ports of the cell.
6. Close the cell input and output using the plugs that were installed when the cell was shipped. Make sure that the cell interior has dried before installing the cell plugs. Using different plugs and tightening them may damage the cell.

Amperometric Cells

Short-term storage

For short-term storage of the cell (less than one week), follow these steps:

1. In Chromeleon, turn the cell off.
2. Stop the mobile phase flow.
3. Remove the cell from the detector.
4. Disconnect the capillaries on the inlet and outlet ports of the cell.
5. Disconnect the detector in Chromeleon.
6. Keep the cell assembled, and close the cell input and output using the plugs that were installed when the cell was shipped. Make sure that the cell interior has dried before installing the cell plugs. Using different plugs and tightening them may damage the cell.

Long-term storage

For long-term storage of the cell (more than one week), follow these steps:

1. In Chromeleon, turn the cell off.
2. Flush the cell with a mobile phase of 20 % methanol and 80 % water. The duration of the flushing should be sufficient to remove any trace of the application mobile phase from the cell. After the flushing is completed, stop the mobile phase flow.
3. Disconnect the detector in Chromeleon.
4. Remove the cell from the detector.
5. Disconnect the capillaries on the inlet and outlet ports of the cell.
6. Disassemble the cell. The procedure corresponds to the disassembly steps in section 7.3.3.1 (→ page 110).
7. Dry the surfaces of the cell and the working electrode with a lint-free cloth.
8. Store the working electrode in its original shipping box.
9. Close the cell input and output using the plugs that were installed when the cell was shipped. Make sure that the cell interior has dried before installing the cell plugs. Using different plugs and tightening them may damage the cell.

5.9 Routine and Maintenance Intervals

5.9.1 General Information

The detector 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, using lint-free cloth or paper. If surfaces are exposed for longer periods, these liquids can cause damage.

Maintenance between Analyses
When the instrument is powered up, several chromatographic and electrochemical equilibria are established. If the unit is turned off and powered up again, wait until these equilibria have been re-established to obtain maximum performance. To achieve high sensitivity analyses (e.g., 50 nA or below), it may take several hours or longer to re-establish these equilibria factors.

In order to minimize the instrument downtime during the periods of non-use, it is recommended to keep the cells turned on with a decreased mobile phase flow rate and recirculation.

5.9.2 Maintenance Intervals

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

Frequency	What you should do...
Daily	Inspect the fluid connections for indications of leakage or restrictions.
	When using buffer solutions, flush the system thoroughly after use. Use a solvent that does not contain buffers or salts.
	Check that the solvent bottle(s) contain sufficient mobile phase for the expected analysis.

Frequency	What you should do...
Regularly	Check the drain tube connected to the drain port on the bottom right of the detector (→ page 48). 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.
	Inspect the tubing for possible damage, such as cracks, nicks, cuts, or blockage.
	Replace the graphite and PEEK filter elements of the in-line filters at least on a quarterly basis (→ page 119).
	Recalibrate the zero current for the signal channels of the cells by performing an **Autozero** command from Chromeleon (→ page 72).
Annually	Have a service representative check the detector once a year to prevent contamination and excessive wear.

Tip: Chromeleon supports functions for estimating the lifetime of consumables to check the performance of certain detector components (→ page 82).

6 Troubleshooting

6.1 Overview

The following features help you to identify and eliminate the source for problems that may occur during the operation of the detector or UltiMate 3000 system.

Status Screens

The status indicators on the front panel provide a quick visual check of the operational status of the detector. They indicate whether the detector is turned on, connected in Chromeleon, and operating properly (→ page 16).

Detector Behavior in Case of an Error

If a critical fault or error is detected during the operation of the detector, the **Status** LED on the front panel door is red, the cell will turn off and, if applicable, the running analysis will be aborted. If the detector is operated with Chromeleon, a message is displayed in the Chromeleon Audit Trail.

In such a case, see the Chromeleon Audit Trail and take appropriate remedial action. It may help to disconnect and reconnect the detector in Chromeleon and to turn it off and on.

Tip: For information about operating problems that might occur during the operation of an UltiMate 3000 system, see Operating Problems (→ page 90).

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

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
No information appears on the detector display.	The instrument is not connected to the mains.	Connect the power cord.
	The power is turned off.	Turn on the detector power.
	The fuses blow.	Replace the fuses (→ page 124).
	Replacement fuse blows immediately.	Contact Service.
	An error occurred in the electronic system.	Contact Service.
Problems during control under Chromeleon	There is no connection between the detector and the Chromeleon computer.	Check the USB cable and connection to the computer.
	The USB port on the computer is not ready for operation.	Check the USB port on the computer. It must comply with the USB 2.0 standard.
	The Chromeleon PC is very slow.	Verify that the system requirements are met (→ page 24).
No flow	The system is leaking.	Find and eliminate the leak.
	There is a gas bubble in the flow path.	Perform a wash cycle (→ *Autosampler Manual*). Non-degassed wash solution is used. Degas the wash solution (→ *Autosampler Manual*).
	For further causes, refer to the Operating Instructions of your pump.	
The system has very high backpressure.	Fluidic parts in the system (capillaries, filter, and column) are blocked by precipitate, or capillaries are damaged by bending.	Check the capillaries in the system step by step from the detector to the pump, remove the blockage, or replace the capillaries.
	The cell may be clogged.	Remove the cell from the detector and check the backpressure. Clean the cell (→ page 105). Restore the cell performance (→ section 7.3.1, page 101).

Problem	Probable Cause	Remedial Action
The system has very high backpressure *(Cont'd)*	Particulates from the mobile phase, the column, or injected samples have accumulated.	Replace the in-line filter elements (→ page 119). Ensure that the mobile phase and/or samples are filtered sufficiently. Use a mobile phase with a substantial fraction of an organic solvent to prevent growth of microorganisms. Observe the guidelines for mobiles phases (→ page 57). Use freshly prepared mobile phase. Growth of microorganisms in the mobile phase may lead to clogging of the filter.
High baseline drift	The column is contaminated.	Clean or replace the column.
	The system is not sufficiently equilibrated.	Flush the system until equilibration.
	The eluents are dirty or not homogeneous.	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 environmental conditions are unstable.	Make sure that the temperature and the humidity are constant. Avoid draft.
	The mobile phase is delivered in circles.	Direct the mobile phase to waste when acquiring data and/or running analyses.
	The electrochemical cell may be contaminated.	Clean the cell (→ page 101). If necessary, replace the electrochemical cell (→ page 107 or 118).
	The mobile phase is contaminated.	Use fresh solvent. Use HPLC-grade eluents only.
Strong noise, non-periodic baseline fluctuation	There are pressure fluctuations from the pump.	Purge the pump; check general function (→ *Pump Manual*).
	There are air bubbles in the system.	Purge the system (→ *Pump Manual*).
	The eluent is dirty or their purity is insufficient.	Use fresh solvent. Use HPLC-grade eluents only.
	The gas content of the eluent is too high.	Degas the eluent and/or install a restrictor at the cell outlet.
	The detector is defective.	Contact Service.

Problem	Probable Cause	Remedial Action
Strong noise, non-periodic baseline fluctuation *(Cont'd)*	There is a problem with the cell.	Clean the cell (→ page 105). Replace the cell if necessary (→ page 107 or 118).
	The system is not grounded.	Verify that all system components are grounded.
	The mobile phase is contaminated.	Use fresh solvent. Use HPLC-grade eluents only.
Periodic baseline fluctuation, pulsation	There are pressure fluctuations from the pump.	Purge the pump; check general function (→ *Pump Manual*).
	There are air bubbles in the system.	Purge the system (→ *Pump Manual*).
Peak Tailing	Too large extra column volume	Use short capillary connections with a suitable inner diameter.
	There are bad capillary connections.	Use different capillaries, for example, Viper capillaries.
Peak Broadening, increased dead time	The inner diameter of the capillary to the detector is too large.	Change the capillary.
	The filter frits on the solvent lines are clogged.	Check the filter for permeability. Replace the filter frit if necessary (→ *Pump Manual*).
	The capillaries are clogged or capillary connections bad.	Replace the capillaries. Use different capillaries, for example, Viper capillaries.
	Improper gasket installed in the amperometric cell.	Use a gasket with a smaller volume.
	The sample loop is clogged.	Replace the needle (→ *Autosampler Manual*).
	The proportioning valve is defective.	Contact Service.
	The column is overloaded or contaminated.	Clean or replace the column.
	The eluent has changed.	Use fresh solvent.
Reproducible ghost peaks in the chromatogram.	The degassing channels are contaminated.	Rinse the degassing channels *(→ Solvent Rack or Pump Manual).*
	The solvents are degraded or dirty or their purity is insufficient.	Use fresh and appropriate solvents.
	Contamination occurs somewhere in the system.	Flush the system using an appropriate solvent.

Problem	Probable Cause	Remedial Action
Some broad ghost peaks in the chromatogram.	Late eluting peak from previous analysis.	Extend the run time. Increase the elution strength of the gradient (higher organic content). At the end of the run, flush column with strong eluent.
Spikes	There are air bubbles in the electrochemical cell.	Check all fluid connections for tightness. Degas the mobile phase and/or install a restrictor at the cell outlet.
	Electrical interferences from other instruments.	Isolate the electrical circuit from strong current consumers. Consider using an UPS (Uninterruptible Power Supply) to filter current fluctuations.
	The column temperature is significantly above boiling point of the mobile phase.	Install a restrictor at the cell outlet. Install a post-column cooler *(→TCC-3000RS Manual)*.
Negative Peaks	Sample solvent and mobile phase differ in composition.	Dissolve the sample in the mobile phase.
High Background Current	There are electroactive impurities in the mobile phase.	Increase the potential by 50 to 100 mV. The steady state mobile phase current will increase significantly if a component of the mobile phase is being electrolyzed. *Coulometric cells only* If the current from the first is higher than the current from the second electrode, mobile phase impurities may be likely. If possible, reduce the potential and avoid using triethylamine and other organic amines as chromatographic modifiers as organic amines tend to contain electroactive impurities. For further information about background currents, refer to section 5.6.3 (→ page 78).

Problem	Probable Cause	Remedial Action
High Background Current *(Cont'd)*	Electroactive species elutes from the column.	This may occur when a new mobile phase or column is used. Allow the system to equilibrate for an hour with the new mobile phase or until the baseline is stable (overnight if the mobile phase contains an ion-pairing agent) and check the current again Remove the column and re-establish flow. If currents drop, clean or replace the column.
	Adsorption on the electrode	Some electrochemical reactions lead to products that are adsorbed on the surface of the electrode. This may result in decreased response. Reversing the potential may restore the performance of the electrode. Clean the cell (→ page 105).
	Contaminants leach from system components.	Check the mobile phase reservoir filters, column end frits and replace them if necessary. Passivate the system components (→ page 137).
Loss of response	Compounds of interest are not sufficiently stable.	Some compounds will decompose as a function of time. Check the stability regularly, and prepare fresh standards.
	The optimum potential has shifted.	Generate a new HDV to optimize operating potential. (→ page 75). Perform an electrochemical treatment with the cell (→ page 102).
	The pH of the solvent or the mobile phase composition has been changed.	Check the pH of the solvent. Mobile phases should be freshly prepared if the pH is incorrect. Use a fresh mobile phase.
	Electrochemical cell or working electrode is old or damaged	*Coulometric cells* Replace the cell (→ page 107). *Amperometric cells* Replace the working electrode (→ page 117).
	The wrong potential was selected.	A different potential may be required.

Problem	Probable Cause	Remedial Action
Poor peak area precision	The autosampler draws air from the vial.	There is not enough amount of sample in the vial, the needle height setting is incorrect (→ *Autosampler Manual*), or there are too many replicates.
	There are air bubbles in the syringe or the autosampler fluidics.	Flush the syringe (→ *Autosampler Manual*). Non-degassed wash solution is used. Degas the wash solution (→ *Autosampler Manual*).
	There is a gas bubble in the flow path.	Perform a wash cycle (→ *Autosampler Manual*).
	The draw speed is too high.	Reduce the draw speed (→ *Autosampler Manual*).
	The gas content of the sample is too high or saturated.	Reduce the draw speed (→ *Autosampler Manual*). Degas the sample if possible.
	The needle is clogged or the needle tip is deformed.	Replace the needle (→ *Autosampler Manual*).
	The autosampler, the injection valve, or the syringe valve is not tight.	→ *Autosampler Manual*
	Carry-over occurs in the system.	Flush the needle using an appropriate solvent *(→ Autosampler Manual)*.
	The capillary connections are not installed properly or they are not tight.	Check and tighten the capillary connections. Exchange the needle seat if necessary (→ *Autosampler Manual*). Exchange the needle if necessary (→ *Autosampler Manual*).
	There are dead volumes in the capillary connections.	Replace the fittings. Make sure that the capillaries are installed correctly. Thermo Fisher Scientific recommends using Viper capillary connections whenever possible.
	The piston seals are not tight.	Replace the seals (→ *Pump Manual*).
	There is air in the working head.	Purge the pump; check general function (→ *Pump Manual*).
	There is pump pulsation.	Use degassed solvents.

Problem	Probable Cause	Remedial Action
Poor peak area precision *(Cont'd)*	The gradient is irreproducible.	Change the gradient. Check the pump function and degassing. Check the filter frits in the solvent line filters for contamination. Replace the frits if necessary.
	The sample is unstable and decomposes.	Use new sample or change the conditions. Cool the sample in the autosampler.
	Baseline fluctuations	see "Baseline Fluctuations"
	The environmental conditions are unstable.	Make sure that the temperature and air humidity are constant. Use column thermostatting. Avoid draft.
	Contamination occurs somewhere in the system.	Flush the system using an appropriate solvent.
	There is a loss of response in the cell.	The cell performance may need to be restored (→ page 101). *Coulometric cells* Replace the cell (→ page 107). *Amperometric cells* Replace the working electrode (→ page 117).

7 Service

7.1 General Notes and Safety Precautions

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

Observe the following precautions:

- Observe all warning notes when carrying out maintenance or repair work.
- Keep in mind that the fluid components of the system may be filled with toxic solvents. Before starting maintenance, rinse toxic solvents from the instrument and put on protective clothing.
- Use original spare parts only. Substituting third-party spare parts or using third-party accessories may impair the performance of the detector, thereby voiding the product warranty. For more information, see the warranty statement in the terms of sale.
- Before returning the detector 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 packaging and observe the packing instructions (Service Return Form section in the manual binder) when shipping the detector. Shipping the detector in anything other than the original packaging will void the warranty. If the original shipping container is not available, appropriate shipping containers and packing material can be ordered from Thermo Fisher Scientific sales organization for Dionex HPLC products. The packing instructions are included in the "Installation and Qualification Documents for Chromatography Instruments" binder and are also available on request. For more information, see the warranty statement in the terms of sale.

For instructions on shutting down the unit, see page 83.

7.2 Replacing the Potentiostat Module

Part no.	Description
6070.1400	Potentiostat Module, dual channel DC, including Installation Instructions

Observe the following when removing or installing a potentiostat module:

- Torx 10 screwdriver is required to remove and install the module. The screwdriver is included in the accessories kit for the detector.
- Use an anti-static device to install or remove potentiostat modules.
- Use proper electrostatic discharge (ESD) precautions to avoid performance degradation or loss of functionality.

Fig. 31: Potentiostat module

1. Before you begin, make sure that
   - ♦ the detector is turned off.
   - ♦ the electrochemical cell is removed.
2. With the Torx screwdriver, loosen the two screws that attach the potentiostat module to the interior front panel. Keep the screws for the replacement potentiostat module.
3. Remove the potentiostat module from the bay.

4. Insert the replacement potentiostat module into the bay. Be sure to align the tabs of the potentiostat module with the cell bay guides. Make sure that the connector on the potentiostat module is in the correct orientation with the borings for the guiding pins on the electrochemical cells being above the connector (→ Fig. 32), and that the module base plate is flush against the interior front panel. The potentiostat module should be firmly seated in the cell bay.

Fig. 32: Inserting potentiostat module

5. Tighten the two screws that attach the potentiostat module to the interior front panel handtight (→ Fig. 33). Do not use the screws to seat the module in the cell bay.

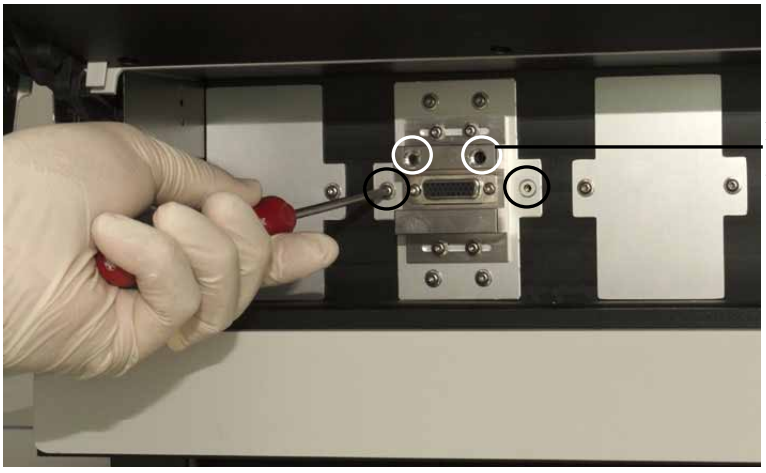


Fig. 33: Tightening the attachment screws

6. Turn on the detector and wait until the self test is complete. If the self test is successful, you can turn off the detector again. If the detector fails the self test, turn off the detector and remove and reinstall the potentiostat module (→ step 1 to 6). If the detector continues to fail the self test, connect to Chromeleon, re-perform the self test, and see the Chromeleon Audit Trail for information about the cause. Note down the exact wording and contact Thermo Fisher Scientific Service Dionex HPLC Products.

7. Reinstall the electrochemical cell. For installation details, refer to the Installation Instructions for the respective cell. For service procedures for the cells, refer to section 7.3 (→ page 101).

8. Turn the detector on and reconnect it in Chromeleon.

7.3 Electrochemical Cells

Observe the following when performing service procedures on an electrochemical cell:

- The contacts for the cell identification chip are located on the rear of the electrochemical cell. To ensure optimum performance of the chip, be careful not to touch the contacts.
- Use proper electrostatic discharge (ESD) precautions to avoid performance degradation or loss of functionality.
- Capillary connections between the column and electrochemical cell should be as short as possible to avoid peak broadening effects due to excessive dead volume.
- No tools are required to remove and install a flow cell.

7.3.1 Restoring the Cell Performance

When a cell does not produce the expected response or is no longer usable, a number of procedures can be tried to restore its performance.

Tip: If a sample analysis is critical, it may be helpful to have a replacement cell available before trying these procedures.

In some situations it is possible that performance will not improve and may even be made worse.

The apparent loss of response in the cell can be a result of many factors such as changes in the HPLC components, degradation of standards and auto-oxidation of the sample on column.

If the effect is isolated to the cell, the loss of response could be due to:

- Poisoning or coating of the electrode
- Shift in the hydrodynamic voltammogram (HDV)
  In many cases the first two effects occur simultaneously.
- Age of the cell
- Physical damage to the cell
- *Coulometric cells only*
  Clogging of porous graphite electrodes

Occasionally, eluent or sample compounds may deposit in the cell or the electrodes, thus increasing the detector noise level and adversely affecting response. In many cases, cleaning the electrochemical cell significantly improves the performance of the detector.

7.3.1.1 Electrochemically Treating the Cell

There is usually a small variation in the HDV characteristics between cells. This can be minimized by a simple electrochemical pretreatment. Electrochemical pretreatment is used when the applied potential is raised to a higher value (approximately +1000mV).When using a cell for analysis, response can often be restored by shifting the HDV curve back to its initial position. A shift in HDV can be verified by initially setting the potential to +300 mV higher than normally used. If the response is restored, the problem is probably a shift in the HDV.

To treat the cell electrochemically and sharpen the hydrodynamic voltammogram (HDV):

1. Turn off the potential to the cell.
2. Replace the mobile phase with fresh phase. The mobile phase should be of low organic solvent composition (less than 15 %) flowing at about 1 mL/min. Do not recycle the mobile phase during the treatment.
3. Apply a potential of +1000 mV to the electrode(s) for 10 minutes with mobile phase flowing. The currents will more likely over-range, this is normal.
4. Reset the potentials to the working potentials for the analysis.
5. Establish a stable baseline and test the response.

If there is no observed shift in the HDV, oftentimes, cell response can be recovered by performing the electrochemical pretreatment along with an additional negative applied potential. The resulting high magnitude current (either positive or negative) can remove unwanted materials on the electrode.

If the response is not back to normal at this point, a combination of potentials should be tried:

a) With fresh mobile phase flowing, set the potential to -350 to -450 mV for 10 minutes. Follow this treatment by setting the potentials to +1000 mV for additional 10-15 minutes.

b) Return to operating conditions. Once a stable baseline is achieved, evaluate the response.

If the response has not been restored, the electrochemical cell should be replaced (→ page 107 or 118).

Automated Electrochemical Treatment for Coulometric Cells

Chromeleon supports the automated electrochemical treatment of the cell after an injection. This setting is designed to clean the surface of porous graphite electrodes of coulometric cells. This setting should only be used when biological samples are injected as part of the analysis. These samples may contain material(s) that can deposit on the porous graphite electrodes resulting in reduced response over time. To activate this setting, open the **Commands** (F8) dialog box and double-click the plus sign next to the cell for which you want to activate the setting. With the **CellClean** command you can activate the setting. To determine the potential (**CleanCellPotential**) and the duration (**CleanCellDuration**), adjust the settings under the corresponding commands.

To determine the period of time that the detector should wait until the next batch after cleaning, make the desired setting under **PostCellCleanDuration** for the detector.

7.3.1.2 Reducing High Backpressure (Coulometric Cells)

Occasionally a coulometric cell will exhibit normal performance but have unusually high backpressure. The cell may continue to be used even with the increased backpressure if it exhibits normal analytical behavior.

⚠ **Important:** Perform the following procedure for coulometric cells only at reduced mobile phase flow rates to avoid cell damage.

⚠ **Important:** Procédez comme suit pour les cellules coulométriques seulement à des vitesses d'écoulement de phase mobile réduit afin d'éviter des dommages aux cellules.

The backpressure in a coulometric cell may be reduced with this mobile-phase backflush procedure:

1. Turn off the cell potential and stop the pump flow.
2. Reverse the fluid flow direction to the cell by inverting the capillaries on the inlet and outlet of the cell. Ensure that the PEEK prefilter remains plumbed to the incoming fluid.
3. Allow mobile phase to flow through the cell at 0.25 mL/min for 10 minutes.
4. Turn off the pump flow, return the cell to its correct orientation and return to operating conditions. Evaluate the back flushing effect. If the backpressure remains high, clean the cell (→ page 105).

To reduce the backpressure with amperometric cells, follow disassemble the cell (→ section 7.3.3.1, page 110) and clean the working electrode (→ section 7.3.3.4, page 115).

7.3.1.3 Cleaning the Cell

In some cases, a exposure of the cells to acidic or base chemicals or alternating aqueous or organic mobile phase reduces the build-up of lipophilic materials.

Tip: Cleaning the cells can have the added benefit of recovering response from dirty and/or fouled electrodes.

Cleaning Coulometric Cells

1. Turn off the cell potential and stop the pump flow.
2. Remove the analytical column from the fluidics.
3. Remove the mobile-phase by flushing the cell with 100% water for 10 minutes at 1.0mL/min.
4. Flush the cell with 100% methanol, acetonitrile, acetone, acetonitrile, methanol and back to water. All sequential flush routines should be done with a flow rate of 1.0mL/min for 10-15 minutes each.
5. Stop the pump flow and change the PEEK inline filer element and re-attach the analytical column.
6. Start mobile phase flow and flush the system with the application mobile phase for 10 minutes.
7. Turn the cell on at the working potentials of the application and test response while monitoring backpressure.

Cleaning Amperometric Cells

1. Turn off the cell potential and stop the pump flow.
2. Remove the analytical column from the fluidics.
3. Remove the mobile-phase by flushing the cell with 100% water for 10 minutes at 0.5mL/min.
4. Flush the cell with 80% water and 20% methanol for 10 minutes at 0.5mL/min.
5. Turn off the pump flow and remove any capillary connections to the cell.
6. Disassemble the cell (→ section 7.3.3.1, page 110).
7. Inspect the working electrode and rinse with water in order to remove any material that may be present on the working electrode.

   Tip: The working electrode can be placed in an ultrasonic bath and cleaned with a lint-free cloth using acetone as a solvent to remove any remaining debris.

8. Inspect the cell body and clean the surface with a lint-free microcloth. The microcloth can be wet with acetone in order to clean the surface.
9. Dry the working electrode and cell body with a lint-free cloth.
10. Re-assemble and reconnect the cell as described in section 7.3.3.2 (→ page 111).
11. Start mobile phase flow and flush the system with the application mobile phase for 10 minutes.
12. Turn the cell on at the working potentials of the application and test response while monitoring backpressure.

If the backpressure of the cell is unusually high and is adversely affecting the assay, the cell will need to be replaced (→ page 118). However, if the system pressure is not approaching unsafe limits and the cell exhibits otherwise normal performance characteristics, the cell may be used with the high backpressure.

7.3.2 Replacing the Coulometric Cell

Part no.	Description
6070.2400	Coulometric Cell 6011RS ultra (cell volume 7.06 µL; cell material: PEEK, porous graphite, palladium, PTFE; pressure limit: 40 bar; working electrode material: micro-porous graphite carbon)

Fig. 34: Rear view of the Coulometric Cell 6011RS

1. Before you begin, verify that the cell is turned off and the mobile phase flow through the cell is stopped.
2. Disconnect the cell from the connector on the potentiostat module and then remove the cell.
3. Remove the capillaries from the cell inlet and outlet.
4. Unpack the new coulometric cell.
5. Re-connect the capillary from the cell inlet to the separation column and the waste capillary from the cell outlet to the waste.
   Route the capillaries to the outside through the slots provided in the detector enclosure to prevent them from being pinched when the front panel door is closed.
   Make certain that the IN and OUT connections are correctly made.
6. Connect the replacement cell to the potentiostat module. Make sure that the cell is in the correct orientation and the guiding pins on the cell match the borings in the potentiostat module (→ Fig. 35).

Fig. 35: Installing the coulometric cell to the potentiostat module

7. Open the Chromeleon **Server Configuration**. On the **Detector** page, click **Read Smart Cells** to update the cell information.

8. Flush the cell for 20 minutes with mobile phase while no potential is applied to the electrodes. Use the same mobile phase and flow rate that will be used for the application. Wait until the pump pressure has stabilized before turning on the cells.

   Tip: Thermo Fisher Scientific recommends that the analytical column is not installed during the flushing.

9. Perform an equilibration of the electrochemical cell (→ page 55).

Fig. 36: Coulometric cell installed to the potentiostat module

Tip: When using a new cell always perform an HDV to optimize cell performance (→ page 75).

7.3.3 Amperometric Cells

Amperometric cells consist of several parts (→ Fig. 37) that can be serviced.

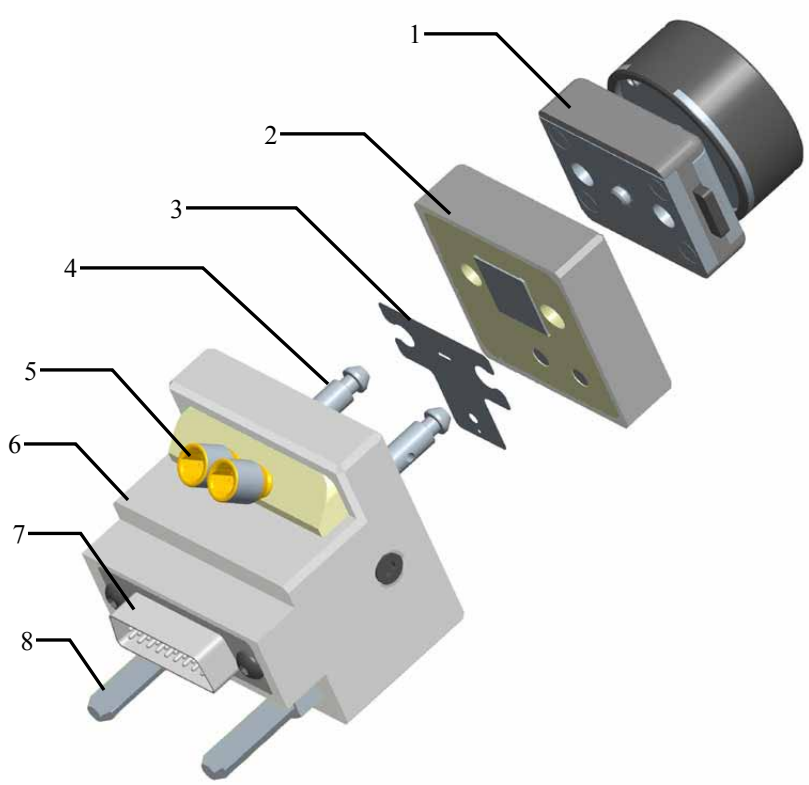


Fig. 37: Explosion view of the Amperometric Cell 6041RS (view from below)

No.	Description
1	Torque knob
2	Working electrode
3	Gasket
4	Alignment pins To align and attach gasket, working electrode and torque knob.
5	Capillary connections (inlet and outlet)
6	Cell body
7	Connector for potentiostat module
8	Guiding pins To install the cell to the potentiostat module.

When performing service procedures on an amperometric cell, place the cell components and working electrode on a clean surface. Even minute particles may cause damage to the cell and the working electrode.

7.3.3.1 Removing and Disassembling the Cell

For the service procedures on the amperometric cell, remove and disassemble the cell:

1. Before you begin, verify that the cell is turned off and the mobile phase flow through the cell is stopped.
2. Disconnect the cell from the connector on the potentiostat module and then remove the cell carefully.
3. Remove the capillaries from the cell.
4. Remove the torque knob from the cell body:

   a) Turn the torque knob counterclockwise to loosen it.

   b) Press the two tabs on the knob and carefully slide it from the alignment pins on the cell body.

Fig. 38: Removing torque knob from the cell

5. Carefully slide off the working electrode from the alignment pins on the cell body.
6. With the tweezers provided in the cell accessories kit, carefully remove the gasket from the cell body, taking care not to scratch the surface of the cell body. Dispose of the used gasket. Do not reuse gaskets.

   Tip: Thermo Fisher Scientific recommends using a new gasket whenever the cell is disassembled.

7.3.3.2 Assembling and Installing the Cell

After cleaning or replacing the working electrode or after replacing the gasket, assemble and install the cell:

1. Carefully place the replacement gasket over the alignment pins of the cell body with the tweezers. Make sure that the two recesses in the gasket match the alignment pins. The gasket should be flat against the cell body. Make sure that
   - ♦ no wrinkles are visible on the gasket.
   - ♦ there are no air bubbles between the gasket and the cell body.

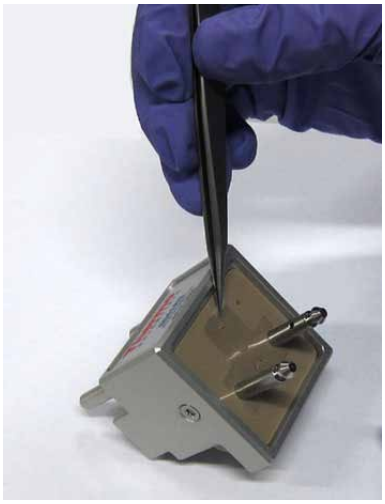


Fig. 39: Installing the gasket

2. Place the working electrode on the gasket. Make sure that
   - ♦ the gold contact pads on the working electrode body match the two gold contact pins on the cell body (→ Fig. 40).
   - ♦ the working electrode is in contact with the gasket (→ Fig. 41).

Fig. 40: Left: Contact pads on a BDD working electrode
Right: Contact pins on the cell body

Fig. 41: Installing the working electrode (here: Boron-doped diamond electrode)

3. Install the torque knob on the cell body:

   a) Take the torque knob by its side tabs and carefully slide it on the alignment pins. Make sure that the tabs are engaged in the slot on both alignment pins.

Fig. 42: Holding the side tabs and installing the torque knob

   b) Turn the torque knob clockwise until it clicks several times to ensure that it is properly seated and seals tightly with the gasket and the working electrode.

4. Connect the capillary from the cell inlet to the separation column and the waste capillary from the cell outlet to the waste.
   To avoid that the capillaries are pinched when the door is closed, route the capillaries to the outside through the slots in the detector enclosure.

   Tip: The cell does not have a directional flow for the fluidic connections. You can choose which port you want to use as inlet port and which one as outlet port. Choose the ports in a way that best optimizes the fluidics of the system.

5. Connect the cell to the connector on the potentiostat module. Make sure that the cell is in the correct orientation and the guiding pins on the cell match the borings in the potentiostat module.

6. Open the Chromeleon **Server Configuration**. On the **Detector** page, click **Read Smart Cells** to update the cell information.
7. Click the **View Cell Data** button to open the configuration page for the working electrode.
8. On the **Cell Properties** page, select the cell bay to which you installed the cell and enter the serial number of the working electrode. Click the arrow on the **Material** list to select the working electrode material.

   Tip: The serial number of the working electrode can be found on the label of the working electrode.

9. Save the configuration and close the Chromeleon **Server Configuration**.
10. Turn on the pump and start the delivery of the mobile phase flow. Flush the cell for a minimum of 20 minutes while the cell is turned off. Use the same mobile phase and flow rate that will be used for the application. Wait until the pump pressure has stabilized before turning the cell on.

    Important: Do not apply flow rates above 1.0 mL/min with the cell to avoid a leak at the gasket which may damage the working electrode.

    Important: Ne pas appliquer débits supérieurs à 1,0 mL / min avec la cellule afin d'éviter une fuite au niveau du joint qui peut endommager l'électrode de travail.

    Tip: Thermo Fisher Scientific recommends that the analytical column is not installed during the flushing.

11. Equilibrate the cell before using it for sample analysis (→ page 55).

7.3.3.3 Replacing the Gasket

The gasket defines the cell volume and flow path of an amperometric cell. Gaskets should be replaced regularly to ensure optimum performance of the amperometric cell.

The following gaskets are available:

Part no.	Description
6070.2528	Cell gaskets, 25 nL volume, boPET, 5 gaskets For Amperometric Cell 6041RS
6070.2529	Cell gaskets, 50 nL volume, boPET, 5 gaskets For Amperometric Cell 6041RS

1. Remove the amperometric cell from the detector and disassemble it as described in section 7.3.3.1 (→ page 110).
2. Locate the replacement gasket and unpack it.
3. Re-assemble the amperometric cell and connect it to the detector as described in section 7.3.3.2 (→ page 111).

7.3.3.4 Polishing the Glassy Carbon Working Electrode

The need for fine polishing the glassy carbon working electrode is generally indicated by a gradual loss in cell performance, tailing peaks, an increase in background current and/or an increase in baseline noise. However, polishing the working electrode should be regarded as a last resort when attempting to recover the cell response by means of physically altering the surface of the working electrode. Before considering electrode polishing, clean the cell as described in section 7.3.1.3 (→ page 105). Also see the Operating Problems section (→ section 6.2, page 90) first when problems with the cell or a loss in cell performance occurs.

⚠ **Important:** Use fine polishing of the working electrode only as a last resort.

⚠ **Important:** Utilisez polissage fin de l'électrode de travail que comme un dernier recours.

Fine polishing is usually sufficient in order to restore prior performance.

The following kit is available to polish glassy carbon working electrodes for amperometric cells:

Part no.	Description
6070.3110	Polishing Kit for Glassy Carbon Working Electrodes, including 1 polishing disc on glass plate 1 bottle alumina suspension, 25 mL

In order to avoid the possibility of cross-contamination, do not use the same disc for polishing different types of working electrodes. Instead, use a separate disc for each working electrode type and label each disc clearly for the type of working electrode that be used on it.

⚠ **Important:** Wear eye protection and take any other needed safety precautions when polishing the working electrode.

All internal parts of the cell should be handled carefully to prevent scratches and other damage, which may cause leaks.

⚠ **Important:** Lors du polissage de l’électrode, porter des lunettes de protection et tout autre équipement de protection individuel (EPI) nécessaire.

Manipuler avec précaution toutes les pièces internes de la cellule afin de ne pas la rayer ou l’abîmer. Ceci pourrait causer des fuites.

1. Remove the cell from the detector and disassemble it as described in section 7.3.3.1 (→ page 110).
2. Rinse the working electrode with purified water in order to remove any mobile phase that is adhering to the working electrode surface.
3. Wet the polishing disk with deionized water.
4. Shake the bottle with the Alumina before each use and add several drops of the polishing compound from the bottle to the polishing disk.
5. Place the part of the working electrode assembly that contains the working electrode material flat on the disc and the polishing compound.
6. Spread the polishing compound around on the disc and then polish the working electrode by moving it around in a figure eight pattern on the disc for approximately 1 minute. Use only a moderate amount of pressure, otherwise the target will wear too quickly or unevenly.
   The working electrode is polished sufficiently when any dark color on the electrode is removed and a smooth surface appears.

Tip: The polishing disc is reusable and does not need to be rinsed after each polishing. After it is used for the first time, add only the needed amount of polishing compound and purified water to re-wet the disc.

7. After the working electrode has been polished sufficiently, rinse it with purified water until all traces of the polishing compound have been removed. In addition, the working electrode can be cleaned by placing it in water in an ultrasonic bath for 30-60 seconds.
8. Dry the working electrode with a clean laboratory tissue. Be careful not to leave any fingerprints on the electrode.
9. Carefully dry the cell body with a tissue. The cell body must be completely dry before reassembly.
10. Re-assemble the cell and connect it to the detector as described in section 7.3.3.2 (→ page 111).

7.3.3.5 Replacing the Working Electrode

The following working electrodes are available:

Part no.	Description
6070.3200	Working electrode, glassy carbon, for Amperometric Cell 6041RS
6070.3100	Working electrode, boron-doped diamond, for Amperometric Cell 6041RS

1. Remove the cell from the detector and disassemble it as described in section 7.3.3.1 (→ page 110).
2. Locate the new working electrode and unpack it.
3. Re-assemble the cell and connect it to the detector as described in section 7.3.3.2 (→ page 111).

7.3.3.6 Replacing the Amperometric Cell

Part no.	Description
6070.3000	Amperometric Cell 6041RS (cell volume depending on gasket; cell material: PEEK, palladium, boPET; pressure limit: 13.8 bar; shipped without working electrode) With cell accessory kit including 2 gasket sets (boPET, 25 nL and 50 nL volume) 1 waste line The working electrode must be ordered separately (→ page 133).

1. Remove the cell from the detector and disassemble it as described in section 7.3.3.1 (→ page 110).

2. Locate the cell and the cell components:

   - The cell body and torque knob.
   - The gasket. Gaskets are shipped with the cell.
   - The working electrode. Working electrodes can be ordered separately (→ section 9.2, page 133).

3. Assemble the cell and connect it to the detector as described in section 7.3.3.2 (→ page 111).

Tip: When using a new cell, always perform an HDV to optimize cell performance (→ page 75).

7.4 In-Line Filters

7.4.1 Replacing the Filter Element

The following filter elements are available:

Part no.	Description
70-0898	Filter Elements, graphite 5 filter elements For in-line filter connection between the pump outlet and the autosampler inlet.
70-3824	Filter Elements, PEEK 5 filter elements For in-line filter connection between the column outlet and the electrochemical cell inlet.

In typical use, the filter elements become clogged and must be replaced on a periodic basis (→ section 5.9.2, page 86). The frequency of replacement is dependent on the level of particulate matter present in the mobile phase and the sample, as well as the production of fine particles from the analytical column. Microbial growth, for example, may occur in mobile phases with low levels (< 3%) of organic solvents, and contribute to high background current and reduced cell performance.

Tip: The filter disks do not have any flow direction sign. They can be placed using either side within the inline filter holder.

To change a filter element:

1. *If not yet done*
   Turn the cells off and stop the mobile phase flow. Allow system pressure to drop to zero before disconnecting any components.

   Important: Do not open a fitting on the high-pressure side of the column before system pressure has dropped to zero. The rapid pressure drop can damage various components in the overall system. Always allow the system to drop to zero before breaking any connections.

   Important: Ne pas ouvrir les raccords situés dans la partie pressurisée précédant la colonne avant que la pression ne soit retombée à zéro. Un rapide changement de pression peut endommager les différents composants de la chaîne. Toujours attendre que la pression redescende à zéro avant d'ouvrir les connexions.

2. Remove the filter holder containing the filter element from the system by removing the nuts on either end of the holder.

Fig. 43: In-line filter with graphite filter element

3. Remove the used filter element from the filter holder. If necessary, use a small plastic rod or dowel to carefully dislodge the filter element.

4. Rinse the filter holder with deionized water.

5. Replace one end nut. Insert a new filter element into the filter holder. Ensure that the element is properly centered and seated against the surface of the end nut.

6. Replace the second end nut and tighten carefully until contact between the cap and the filter is felt. The filter is properly installed if both end nuts are approximately an equal distance from the center of the filter holder. The fitting should be fingertight, do not use a wrench or other tools and do not overtighten the fitting, as this can crush the filter.

7. Re-install the filter holder in the system. Ensure that the direction of flow is as indicated on the filter holder.
   Initially, only the upstream end of the filter should be attached to the HPLC system. Pump about 5 mL of the mobile phase through the filter to waste before attaching the downstream end of the filter to the cell. This will serve to wash the filter and ensure that particulate matter does not enter the cell.

7.4.2 Replacing the In-Line Filter

Part no.	Description
70-0893	In-Line Filter Kit with Graphite Filter Elements, including 1 Filter holder, PEEK 5 Filter elements, graphite 2 Nuts, 1/16”, PEEK For connection between the pump outlet and the autosampler inlet.
70-4093	In-Line Filter Kit with PEEK Filter Elements, including 1 Filter holder, PEEK 5 Filter elements, PEEK 2 Nuts, 1/16”, PEEK For connection between the column outlet and the electrochemical cell inlet.

To replace one or both in-line filters, replace the filters as described in the following order.

1. *If not yet done*
   Turn the cells off and stop the mobile phase flow. Allow system pressure to drop to zero before disconnecting any components.
2. Remove the filter holder containing the filter element from the system by removing the nuts on either end of the holder.

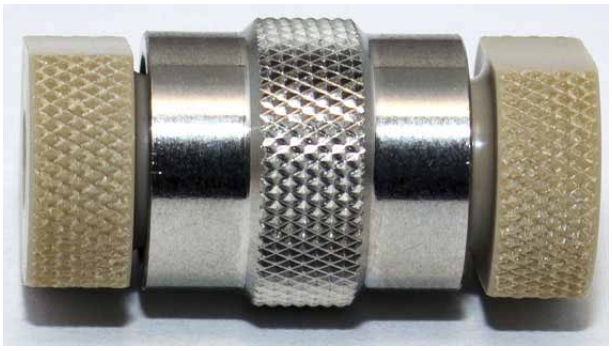


Fig. 44: Filter holder with lock nuts installed

3. Disconnect the capillaries from the filter inlet and outlet.
4. Locate the replacement filter element for the filter holder that you want to replace. Filter elements are included in the respective In-Line Filter Kits and can be ordered separately.
5. Open the filter holder by turning the lock nuts counterclockwise and insert the filter element.
6. Close the filter holder by turning the lock nuts clockwise. Do not overtighten the lock nuts with a metal tool.

7. Re-connect the capillaries to the new in-line filter.

   In-line filter with graphite filter element:

   a) Connect the capillary from the pump outlet (or the Titanium filter) to the inlet of the in-line filter.

   b) Connect a capillary from the outlet of the filter holder to the inlet of the autosampler. Make sure that the capillary is connected correctly, with the filter holder being installed in the direction of the orientation sign on the filter pointing downstream.

   c) Connect a capillary to the autosampler outlet:

      - *If the in-line filter with graphite filter elements is replaced*
        Connect the capillary from the autosampler outlet to the column inlet.
        Continue with step 8.

      - *If both in-line filters are replaced*
        Connect a capillary from the autosampler outlet to the waste.
        Flush the system with mobile phase, for example with a methanol/water solution for 3-5 minutes at a flow rate of 1.0 mL/min to remove any contaminants from the system. Monitor the fittings and tighten as appropriate. If a significant increase in pressure is observed, check if an obstruction or clogged component is present.
        Only then continue with the instructions on installing the in-line filter with PEEK filter elements (→ see below).

   In-line filter with PEEK filter element:

   a) Connect the capillary from the autosampler outlet to the column inlet.

   b) Flush the analytical column with the mobile phase for several hours or overnight before connecting the electrochemical cell to the system.

   c) Stop the mobile phase flow.

   d) Connect a capillary to the outlet of the analytical column and to the inlet of the in-line filter.

   e) Connect a capillary from the outlet of the in-line filter to the inlet of the electrochemical cell.

   f) Make sure that a capillary leads from the outlet of the electrochemical cell to the waste.

8. Start the delivery of the mobile phase, for example with a methanol/water solution, to flow for 3-5 minutes at a flow rate of 1.0 mL/min to remove any contaminants from the system.

9. Monitor the fittings and tighten as appropriate. If a significant increase in pressure is observed, check if an obstruction or clogged component is present. Remove the obstruction, if available.

7.5 Drying the Leak Sensor

The leak sensor is installed inside the detector and reports a leak when liquid collects in the drip tray under the fluid connections. Eliminate the cause for the leakage and dry the leak sensor.

1. In Chromeleon, turn off the cells, stop the pump flow, and disconnect the detector and turn it off.
2. Inspect the electrochemical cell(s) for signs of leakage. If a cell is leaking, tighten the connections to the electrochemical cell. If necessary, replace the cell (→ page 107 or 118).
3. With a cloth or tissue, absorb all liquid that has collected in the tray.

   ⚠ **Important:** Make sure that you do not bend or damage the sensor.

   ⚠ **Important:** Assurez-vous que vous ne tordez, ni n'endommagez le capteur.

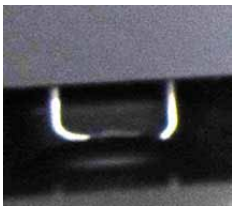


Fig. 45: Leak sensor

4. Allow the sensor to adjust to the ambient temperature for a few minutes.
5. Turn the detector on and reconnect it.
6. If no error is reported after turning on the detector, operation can be resumed.

   Tip: If the sensor is not dry, the **Status** LED remains red. The operation can be continued only when the cause for leakage has been completely removed.

7.6 Replacing the Main Power Fuses

Warning: Turn off the main power switch and disconnect the power cord from its source.

Avertissement: Avant de remplacer les fusibles, arrêtez le détecteur. Assurez-vous de bien débrancher le cordon d'alimentation de la source secteur.

1. Remove the fuse cartridge, using a small screwdriver.

Fig. 46: Fuse cartridge

2. Replace the fuse.

Important: Always install two new fuses.
Use only the fuses indicated in the following table.

Important: Installez toujours deux nouveaux fusibles.
Utilisez uniquement les fusibles indiqués ci-dessous.

Description	Part no.
Fuse, 1A, slow-blow, (5 x 20 mm)	Included in Fuses Kit (part no. 70-6666) For information about the kit, see section 9.3 (→ page 134).

3. Reinstall the fuse cartridge.

4. Reconnect the power cord to its source and turn on the detector.

7.7 Updating the Detector Firmware

The detector is shipped with the most recent firmware version. To check which firmware version is installed in the detector and which version is included in Chromeleon:

- *In Chromeleon*
  In the **Server Configuration** program, open the configuration pages for the detector (→ page 37). On the **General** page, the firmware version is displayed.
- In the Windows Explorer, locate the **IQReport.log** file in the **IQ** folder of your Chromeleon installation. In the file, search for ECDRS FW.

Tip: When updating the firmware via Chromeleon, this information will also be provided during the download (see below).

Whenever a new firmware version is released for the detector, the new version will be provided with the next Chromeleon Service Release and described in the related release notes.

The new firmware will not be downloaded automatically to the detector when you install a Chromeleon Service Release. To update the firmware in the module, follow these steps:

Important: To ensure that the download is successful, make sure that the communication between the detector and Chromeleon is *not* interrupted during the download and do *not* turn off the detector.

Important: Au cours du téléchargement, assurez-vous que la communication entre la pompe et Chromeleon n'est pas interrompue et n'arrêtez pas l'instrument. Ceci peut entraîner des dysfonctionnements de l'instrument.

1. Before you begin, verify that
   - The detector is connected with Chromeleon.
   - The Chromeleon server is in *running idle* mode. This means that all processes on the Chromeleon server PC and in Chromeleon have been stopped.
2. Start the **Server Configuration** program (→ page 36).

3. Right-click the detector in the timebase and select **Properties** on the menu.

4. On the **General** page (→ page 37), the firmware version provided by Chromeleon for the detector is displayed in the **Firmware** field. If more than one firmware version is available for the detector in Chromeleon, select the version from the **Firmware** list.

5. Click **Download**. A message displays the firmware version that is currently installed in the detector and the version that will be downloaded from Chromeleon.

   Tip: If the detector comes with a newer firmware than the version included in Chromeleon, do *not* downgrade the firmware. Older firmware may be incompatible with new hardware revisions.

6. Click **Yes** to start the download. (Click **No** to cancel the action.)

The download may take several minutes. The download is complete when **Download finished successfully** appears in the **Messages Server** window in the Chromeleon Server Configuration program. The message appears also in the Chromeleon Audit Trail.

If the download is not successful, the related messages appear in the Audit Trail. In this case, turn off the detector. Turn on the detector again and repeat the download as described above. If the download fails again, contact Service.

8 Technical Information

8.1 Detector

Detection Type:	Electrochemical detection
Operating Mode:	Fixed potential, direct current mode (with amperometric or coulometric cells)
Potential Control:	Up to four independently controlled channels (with two dual channel DC potentiostat modules)
Potential Range:	max. ± 3.300 mV in 1 mV steps (acceptable range determined by cell type)
Signal Range:	10 pA to 100 µA
Automatic Gain Ranging:	Seamless gain switching over entire signal range
Noise:	< 750 fA (0.75 pA) with 500 MΩ/0.47 µF cell simulator (DC mode), filter constant: 5 seconds
Noise Filtering:	Multi-level digital filtering, adjustable filter constant
Data Collection Rate:	0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 4 Hz, 5 Hz, 10 Hz, 20 Hz, 50 Hz, 100 Hz; 200 Hz (only under Chromeleon 7.1)
Column Compartment:	Integrated temperature control with liquid leak detection
Column Temperature Range:	5 °C above ambient to 40 °C
Control:	All parameters and functions software controlled, USB 2.0, 3 LEDs (Power, Connected, Status) for status monitoring
GLP Features:	In Chromeleon: Full support of automatic equipment qualification (AutoQ™) and system performance monitoring All system parameters are logged in the Audit Trail. Qualification Monitoring with Chromeleon software and cell ID chips.
Display:	LCD display indicating system parameters; 3 LEDs (Power, Connected, and Status) for status monitoring
I/O Interfaces:	3 TTL inputs (Cells Off, Autozero, External Start), 3 relay outputs (contact closures to external devices)
Rear Interface:	AC connector, power switch, USB port, Digital I/O (3 TTL inputs, 3 relay outputs)
Safety features:	Leak sensor, SmartChip technology for limitation of electrochemical cell potential and cell monitoring, Cell potential shutdown mechanism
Power requirements:	100-120 V and 200-240 V, 50/60 Hz; max. 100 VA

Environmental conditions:	Range of use: Indoor use Temperature: 10 °C to 35 °C (50 to 95°F) Air humidity: 80% relative humidity, non-condensing Overvoltage category: II Pollution degree: 2
Dimensions (h × w × d):	19 × 42 × 51 cm (6.3 × 16.5 × 20 in.)
Weight:	Approx. 12.5 kg (approx. 27.5 lbs) with 1 dual potentiostat module

Technical information: December 2012
All technical specifications are subject to change without notice.

8.2 Electrochemical Cells

Specification	Coulometric Cell 6011RS	Amperometric Cell 6041RS
Cell design:	Flow-through, micro-volume, dual-series electrodes	Thin-layer, micro-volume, single-channel cell
Working electrode(s):	Micro-porous graphite carbon	Interchangeable: Glassy carbon (GC) or Boron-doped diamond (BDD)
Cell volume:	7.06 µL	Depending on exchangeable gasket: 25 nL or 50 nL
Upper pressure limit:	40 bar	13.8 bar
Potential range:	-300 to +1100 mV (vs. Palladium)	± 3.300 mV (vs. Palladium), acceptable range determined by working electrode type
Flow rate:	> 0.3 mL/min (Optimum flow rate determined by application and mobile phase composition used)	Up to 1.0 mL/min (Optimum flow rate determined by application and mobile phase composition used)
Fluidic connections:	Inlet and Outlet: 10-32 thread female port (compatible for nanoViper fingertight fitting)	
Cell control:	SmartChip cell technology: For identification of cell type and serial number by mean of a built-in ID chip, and automatic detection parameters configuration by Chromeleon software through cell recognition.	
Environmental conditions:	Operating temperature: 10-45 °C (50-113 °F) Air humidity: 18 to 80 % relative humidity, non-condensing	
Wetted parts:	PEEK, porous graphite, palladium, PTFE	PEEK, palladium, boPET, and glassy carbon or boron-doped diamond
Dimensions (h × w × d):	38 × 44 × 53 mm (1.5 × 1.7 × 2.1 in.)	41 × 45 × 86 mm (1.5 × 1.7 × 2.1 in.)
Weight	Approx. 190 g (6.7 oz)	Approx. 162 g (5.7 oz)

Technical information: December 2012
All technical specifications are subject to change without notice.

9 Accessories, Consumables, and Spare Parts

Accessories, spare parts, and consumables for the detector 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.

For more information about accessories, consumeables and spare parts, contact the Thermo Fisher Scientific sales organization for Dionex HPLC Products.

9.1 Standard Accessories

The following standard accessories are shipped with the detector.

The part number always refers to the packing unit. Unless otherwise stated, the packing unit is 1 unit. For more information, contact the Thermo Fisher Scientific sales organization for Dionex HPLC Products.

Description	**Part no.**	**Quantity in the accessories kit**
Accessories for the ECD-3000RS		
Fuses Kit for ECD (2 fuses, 1A, slow-blow, 5 x 20 mm)	70-6666	1
Additional parts for connecting the drain system: Tee piece for drainage system L piece for drainage system Flexible hose (11.4 mm O.D. x 8.4 mm I.D.)		 1 2 1.5 m
6-pin mini DIN signal cable For connection to UltiMate 3000 pumps (except the LPG-3400XRS pump). For further information, see page 33.	----	1
Attachment screws for cell bay covers 8 screws, Torx M 3 x 6, SST	----	8
In-Line Filter Kit with Graphite Filter Elements, including 1 in-line filter holder, PEEK 5 filter elements, graphite 2 lock nuts for filter holder, with ferrule (PEEK, 1/16”)	70-0893	1
In-Line Filter Kit with PEEK Filter Elements, including 1 in-line filter holder, PEEK 5 filter elements, PEEK 2 lock nuts for filter holder, with ferrule (PEEK, 1/16”)	70-4093	1
Screwdriver, Torx 10 for cell bay cover screws	----	1

Description	**Part no.**	**Quantity in the accessories kit**
Tubings and fittings, including 1 capillary, PEEK (I.D. x O.D. 0.005” x 1/16”), red 1 capillary, PEEK (I.D. x O.D. 0.015” x 1/16”), gray 1 capillary, PEEK (I.D. x O.D. 0.007” x 1/16”) Lock nut, seal tight, with ferrule (PEEK, O.D. 1/16”) Lock nut, seal tight (PEEK, O.D. 1/16”) Fingertight fitting (PEEK, O.D. x L 1/16” x 33 mm), two-piece Ferrule, seal tight (PEEK, O.D. 1/16”) Tubing cutter for PEEK capillaries Note: The tubings and fittings are also available in the Tubing and Fitting Kit for ECD-3000RS (part no. 6070.4800).	 6081.1410 6081.1420 6070.5001 70-4859 70-4746 6000.0011 70-3677 6300.0401	1 2 m 1.5 m 1.5 m 2 8 4 12 1
USB cable type A to type B, 5m	6911.0002	1

9.2 Optional Accessories

Description	Part no.	Remarks
Amperometric Cell 6041RS ultra (cell volume: 25 nL or 50 nL (selectable), working electrode material: glassy carbon or boron-doped diamond (selectable), pressure limit: 13.8 bar)	6070.3000	Includes a Cell Waste Line Kit, 5 gaskets of 25 nL volume, 5 gaskets of 50 nL volume, anti-static tweezers and Installation Instructions for detailed instructions. For general instructions, refer to page 54. For instructions on service procedures, refer to page 101.
Cell Simulator	6070.4100	Includes Installation Instructions for the Cell Simulator.
Coulometric Cell 6011RS ultra (cell volume: 7.06 µL, working electrode material: micro-porous graphite carbon, pressure limit: 40 bar)	6070.2400	Includes a Cell Waste Line Kit and Installation Instructions for detailed instructions. For general instructions, refer to page 54. For instructions on service procedures, refer to page 101.
Drain kit for UltiMate 3000 systems	6040.0005	The kit includes all required components and detailed installation instructions.
nanoViper capillary kit for UltiMate 3000 systems with ECD-3000RS detector	6041.5105	Includes the following capillaries I.D. x L: 2 capillaries 0.1 mm x 75 mm, 1 capillary 0.1 mm x 150 mm, 2 capillaries 0.1 mm x 250 mm, 1 capillary 0.1 mm x 450 mm, 1 capillary 0.15 mm x 250 mm, 1 capillary 0.15 mm x 450 mm, 1 capillary 0.15 mm x 550 mm, 1 capillary 0.15 mm x 750 mm The kit is shipped with installation instructions.
Polishing Kit for Glassy Carbon Working Electrode	6070.3110	Includes 1 polishing disc on glass plate and 1 Alumina suspension, 25 mL. For instructions on use, refer to page 115.
Potentiostat Module, dual channel DC	6070.1400	For detailed instructions, refer to the Installation Instructions provided with the potentiostat module. For general instructions, refer to page 49. For instructions on service procedures, refer to page 98.
Working Electrode, Boron-Doped Diamond (BDD), for Amperometric Cell 6041RS	6070.3100	Includes Installation Instructions.
Working Electrode, Glassy Carbon (GC), for Amperometric Cell 6041RS	6070.3200	Includes Installation Instructions.

9.3 Consumables and Spare Parts

The part number always refers to the packing unit. Unless otherwise stated, the packing unit is 1 unit.

Description	Part no.
Amperometric Cell 6041RS ultra (cell volume: 25 nL or 50 nL (selectable), working electrode material: glassy carbon or boron-doped diamond (selectable), pressure limit: 13.8 bar) Including Cell Waste Line Kit, 5 gaskets 25 nL, 5 gaskets 50 nL, Tweezers, anti-static, Installation Instructions	6070.3000
Bay cover, including 2 attachment screws	6070.1411
Capillaries and Fittings for UltiMate 3000 systems with ECD-3000RS detector	
A─Capillaries	
Capillary (PEEK, 0.007" x 1/16" (I.D. x O.D.), L 1.5 m)	6070.5001
Capillary (PEEK, 0.005" x 1/16" (I.D. x O.D.), L 2.0 m), red	6081.1410
Capillary (PEEK, 0.015" x 1/16 " (I.D. x O.D.), L 1.5 m), gray	6081.1420
B─Fittings, ferrules and lock nuts	
Lock nut, seal tight, with ferrule (PEEK, O.D. 1/16")	70-4859
Lock nut, seal tight (PEEK, O.D. 1/16")	70-4746
Fingertight fitting (PEEK, O.D. x L 1/16" x 33 mm), two-piece	6000.0011
Ferrule, seal tight (PEEK, O.D. 1/16")	70-3677
C─Capillary kit	
Tubing and Fitting Kit The kit includes the capillaries with part numbers 6070.5001, 6081.1410, and 6081.1420 (for details, see 'A─Capillaries'), and fittings with part numbers 70-4859, 70-4746, 6000.0011, and 70-3677 (for details, see 'B─Fittings, ferrules and lock nuts'). It also includes 1 tubing cutter for PEEK capillaries.	6070.4800
Capillaries, nanoViper, for UltiMate 3000 systems with ECD-3000RS detector	
A─nanoViper capillaries	
Capillary (0.1 x 150 mm (I.D. x L), PEEK, nanoViper), for the connection between coulometric cell 6011RS and amperometric cell 6041RS.	6041.5811
Capillary (0.1 x 250 mm (I.D. x L), PEEK, nanoViper), 2 capillaries, for the connection between column and cell.	6041.5812
Capillary (0.1 x 450 mm (I.D. x L), PEEK, nanoViper), for the connection between autosampler and column.	6041.5814
Capillary (0.15 x 250 mm (I.D. x L), PEEK, nanoViper), for the connection between pump and in-line filter with graphite filter element.	6041.5819
Capillary (0.15 x 450 mm (I.D. x L), PEEK, nanoViper), for the connection between in-line filter with PEEK filter element and autosampler.	6041.5821
Capillary (0.15 x 550 mm (I.D. x L), PEEK, nanoViper), for the connection between guard cell and autosampler.	6041.5822
Capillary (0.15 x 750 mm (I.D. x L), PEEK, nanoViper), e.g. for the connection between an LPG-3400 pump to in-line filter with graphite filter element.	6041.5823

Description	Part no.
B─nanoViper capillary kit nanoViper capillary kit for UltiMate 3000 systems with ECD-3000RS detector The kit includes the capillaries with part numbers 6041.5811, 6041.5812, 6041.5814, 6041.5819, 6041.5821, 6041.5822, and 6041.5823 (for details, see 'A─nanoViper Capillaries'). It also includes 2 capillaries (0.1 x 75 mm (I.D. x L), PEEK, nanoViper), e.g. for the connection between two adjacent coulometric cells.	6041.5105
Cell simulator, including Installation Instructions	6070.4100
Cell waste line kit, including Capillary (PEEK, 0.015" x 1/16 " (I.D. x O.D.), L 1.5 m), gray Fitting screws (PEEK, 1/16"), 2 screws	6070.4900
Coulometric Cell 6011RS ultra (cell volume: 7.06 µL, working electrode material: micro-porous graphite carbon, pressure limit: 40 bar) Including Cell Waste Line Kit and Installation Instructions	6070.2400
Filter Element, graphite 5 filter elements	70-0898
Filter Element, PEEK 5 filter elements	70-3824
Fuses kit for ECD-RS (2 fuses, 1A, slow-blow, 5 x 20mm)	70-6666
Gasket, 25 nL, for Amperometric Cell 6041RS, including 5 gaskets, boPET, volume 12 nL	6070.2528
Gasket, 50 nL, for Amperometric Cell 6041RS, including 5 gaskets, boPET, volume 25 nL	6070.2529
In-Line Filter Kit with Graphite Filter Elements, including 1 in-line filter holder, PEEK 5 filter elements, graphite 2 lock nuts for filter holder, with ferrule (PEEK, 1/16")	70-0893
In-Line Filter Kit with PEEK Filter Elements, including 1 in-line filter holder, PEEK 5 filter elements, PEEK 2 lock nuts for filter holder, with ferrule (PEEK, 1/16")	70-4093
Polishing Kit for Glassy Carbon Working Electrodes, including 1 polishing disc on glass plate 1 bottle alumina suspension, 25 mL For polishing of the glassy carbon working electrodes for the Amperometric Cell 6041RS.	6070.3110
Potentiostat Module, dual channel DC, including Installation Instructions	6070.1400
USB cable, type A to type B, high speed USB 2.0 (cable length: 1 m)	6035.9035
USB cable, type A to type B, high speed USB 2.0 (cable length: 5 m)	6911.0002
Working Electrode, Boron-Doped Diamond (BDD), for Amperometric Cell 6041RS, including Installation Instructions	6070.3100
Working Electrode, Glassy Carbon (GC), for Amperometric Cell 6041RS, including Installation Instructions	6070.3200

10 Appendix

10.1 Passivation

A system can include a significant number of components that are made from metals such as polished stainless steel (pump components, tubing, fittings, pulse dampers, filters, etc.). The surface of these components includes oxides of iron that may slowly be dissolved or otherwise removed by the mobile phase. Since these oxides may be electrochemically active and may create high background currents and/or drifting baselines, it may be necessary to thoroughly clean (passivate) certain components of the system to maximize the performance of the detector and avoid unwanted oxidation in the system.

If components of the system are fabricated from metals, such as stainless steel, passivate the components or the system before use. Refer to the specifications documentation for the wetted parts of each component to be placed in the system.

⚠ **Important:** Before passivating an instrument, check that the procedure described will not adversely affect any component.

If you are passivating a solvent delivery module or any other component that is incorporated into an existing system, remove the column and any electrochemical cells before starting the passivation process.

⚠ **Important:** Avant la passivation d'un appareil, vérifier que la procédure de passivation ne va pas nuire aux différents composants.
En cas de passivation du module de pompage des solvants, ou tout autre composant incorporé dans la chaîne existante; retirer la colonne et la cellule électrochimique avant de commencer le processus de passivation.

To passivate an instrument:

1. Disconnect the electrochemical cells, in-line filters and analytical columns from the system fluidics.
2. Flush the instrument or system with solvents in the following order, each at a flow rate of 1 mL/min for approximately 30 minutes:
   a) Solvent mixture of 50 % isopropyl and 50 % water
   b) HPLC-grade water

c) 6N nitric acid

Warning: To avoid damage to the skin and eyes, wear appropriate protective clothing and goggles when using nitric 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 nitrique.

3. Flush the instrument or system with HPLC-grade water at a flow rate of 1.0 mL/min until the pH value of the eluent is approximately 5.0.
4. Flush the instrument or system with solvents in the following order:

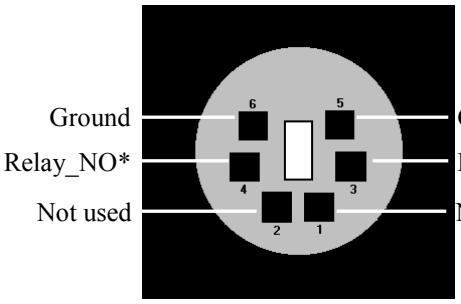
   a) 2 % ethylenediaminetetraacetic acid (EDTA)

   b) HPLC-grade water
5. If the system contains residual levels of organic materials that are not water soluble, flush the system with an organic solvent such as methanol to remove them. After flushing the system with the organic solvent, flush the system with HPLC-grade water. If the mobile phase that is presently in the system is not miscible with water, gradually change its composition so that it will become miscible with water.
6. Flush the instrument or system with HPLC-grade water at a flow rate of 1.0 mL/min until the pH value of the eluent is approximately 5.0.

Tip: Make sure that you passivate small metal components such as ferrules, metal tubing and fittings, use a syringe to flush the components in the manner described above.

10.2 Digital I/O (Pin Assignment)

The two digital I/O ports provide 3 digital inputs and 3 relay outputs that can be used to exchange digital signals with external devices.



* NO = Normally open contact
COM = Common contact

If the relay is not activated or if the detector is turned off, there is no connection between COM and NO. If the relay is activated, the connection between COM and NO is established.

Fig. 47: Mini-DIN Digital I/O port

To connect an UltiMate 3000 pump (except the LPG-3400XRS pump*) to a digital I/O port on the rear panel of the detector, use the appropriate mini-DIN signal cable that is included in the accessories kit for the detector. The table lists the functions assigned to the connector pins and the label of the cable wire connected to each pin.

Pin	Signal Name	Signal Level	Remark
1	Not used	---	---
2	Not used	---	---
3	Relay_COM	Potential free	Common contact (Digital I/O port 2)
4	Relay_NO	Potential free	Closing contact (Digital I/O port 2)
5	Cells Off	TTL	Digital input (Digital I/O port 1)
6	GND	Ground	Reference potential (Digital I/O port 1)

Fig. 48: Pin assignment (port and cable)

Tip: The input has a pull-up resistor.

* The connection to a LPG-3400XRS pump will be supported in a future Chromeleon version.

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