Thermo Scientific
UltiMate 3000 Series

Electrochemical Detector
ECD-3000RS

Operating Instructions
(Original Operating Instructions)

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1 Introduction

1.1 How to Use This Manual

The layout of this manual is designed to provide quick reference to the sections of interest to the reader when operating the Thermo Scientific™ UltiMate™ 3000 detector. However, in order to obtain a full understanding of the detector, Thermo Fisher Scientific recommends that you review the manual thoroughly before beginning operation.

The descriptions in this manual apply to the UltiMate 3000 ECD-3000RS Electrochemical Detector. The following conventions apply to the descriptions throughout this manual:

- The detector is referred to as *detector* or *ECD-3000RS Electrochemical Detector* in this manual. If other detector types are referenced, they are identified by name.
- The detector configuration may vary. Therefore, not all descriptions necessarily apply to your particular detector configuration.
- Illustrations in this manual are provided for basic understanding. They can vary from the actual model of the detector or component. However, this does not influence the descriptions. No claims can be derived from the illustrations in this manual.
- If not otherwise stated, the descriptions for the Viper™ capillary connections apply also to the nanoViper™ and possible other Viper capillary connections.
- The descriptions in this manual refer to firmware version 1.10 and Chromeleon™ 6.80. If you want to operate the module from Chromeleon 7, note the information on page 26.

  This manual assumes that the module is used with a suitable Chromeleon software version with a valid license. For further information on operation of the detector from Chromeleon, see section 2.13, page 26.

This manual is provided "as is". Every effort has been made to supply complete and accurate information and all technical specifications have been developed with the utmost care. 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

1.2.1 Symbols on the Detector and in the Manual

Symbols on the Detector

The table shows the symbols used on the detector:

Symbol	Description
~	Alternating current Courant alternatif
	Power supply is on (-) / Power supply is off (O) L'instrument est mis sous tension (-) / 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 DEEE (Déchets d'Equipements Electriques et Electroniques)—Pour plus d'informations, référez-vous au chapitre WEEE Information dans le classeur "Installation and Qualification Documents for Chromatography Instruments"

Safety Symbols and Signal Words in This Manual

At various points throughout the manual, the following symbols indicate messages of particular importance:

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

Important: Indicates that failure to take note of the accompanying information could cause wrong results or may result in damage to the device.

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 Safety Precautions

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

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

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

Tip: Before initial operation of the detector, make yourself familiar with the contents of this manual.

For the safety precautions in French, see page 7.

- **Protective equipment**

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

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

- **Hazardous substances**

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

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

  Handle all hazardous substances, such as acids, carefully. If procedures such as particular maintenance procedures for electrochemical cells, require using acids such as phosphoric acid, verify that your wear personal protective equipment and take special care.

- **Hazardous gases**

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

- **Electrostatic discharge**

  Discharge of electrostatic energy may lead to sparking and can constitute a fire hazard. This effect is particularly pronounced in insulating capillaries und with non-conductive solvents (for example, pure acetonitrile).

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

- **Self-ignition of solvents**

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

- **Capillaries, capillary connections, open connections**
  - ♦ Capillaries, especially non-metallic capillaries may burst, slip out of their fittings or may not be screwed in. This may result in substances spraying out of the open connections.
  - ♦ In an UltiMate 3000 system, some components are made of PEEK. This polymer has superb chemical resistance to most organic solvents. However, it tends to swell when in contact with trichlormethane (CHCl3), dimethyl sulfoxide (DMSO), or tetrahydrofuran (THF). In addition, it is attacked by concentrated acids, such as, sulfuric acid and nitric acid or a mixture of hexane, ethyl acetate, and methanol. In both cases, capillaries may start leaking or they can burst. Swelling or attack by concentrated acids is not a problem with brief flushing procedures.
  - ♦ Do not use tubing that is stressed, bent, kinked, or damaged.
  - ♦ Capillary connections can be contaminated by harmful substances or harmful substances can escape from open connections.
  - ♦ Always wear safety glasses when handling fused silica tubing, for example, during installation or when cutting capillaries to the length.
- Disconnect the detector from all power sources before removing the panels. When the panels are removed, dangerous electrical connections will be exposed. The enclosure must be opened only by Thermo Fisher Scientific service personnel.
- Always replace blown fuses with original spare part fuses authorized by Thermo Fisher Scientific.
- Replace faulty communication cables.
- Replace faulty power cords. Never use a power cord other than the power cords provided for the device.
- Use only the original spare parts and accessories authorized for the device by Thermo Fisher Scientific.
- When operating the HPLC system, always set a lower pressure limit for the pump. This prevents damage resulting from leakage or from running the pump dry.
- To prevent damage to the detector when lifting or moving, always lift the unit by the bottom sides or sides. Do not lift the detector by the bottom front or front panel door. This may damage the door.
- The open front panel door is not designed to carry weight. Do not place any heavy objects on the open front panel door; this may damage the door.
- After operation, rinse out buffers and solutions that form peroxides.
- Before switching from buffer to organic solution, rinse the analytical system thoroughly with de-ionized or HPLC-grade water.

- When switching to another solvent, ensure that the new solvent is miscible with the one contained in the HPLC system. If the solvents are not miscible, the system can be damaged, for example, by flocculation.
- If a leak occurs, turn off the detector immediately, stop the pump flow, and remedy the situation.
- Use only standard solvents (HPLC-grade) and buffers that are compatible with all parts that may be exposed to solvents.
- Before interrupting operation for several days or more or when preparing the detector for transport, observe the precautions for shutting down the detector (→ page 98).
- **Sensitive Electrochemical Cells**

  Electrochemical cells are sensitive. To prevent damage to the detector and detector components, observe the following safety guidelines:

  - 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 EC-compatible mobile phase flow is established before the cell is turned on and that flow remains turned on whenever potential is applied to avoid permanent damage to the cell.
  - If the system flow path contains ferrous metals, such as stainless steel, this can disrupt operation of electrochemical cells and electrodes. Passivate the instruments and components before connecting the electrochemical cell in the flow path.

    For passivation instructions, see section 10.1, page 131.
  - Do not use nitric acid (concentrated HNO_3) to clean an electrochemical cell. Exposure may damage susceptible internal components. Refer to the *User Guide* for your respective electrochemical cell for recommended cleaning procedures.
  - Make sure that you operate the cell within the specifications for backpressure and applied potential. Observe the specifications and the mobile phase guidelines for the electrochemical cells.

    For mobile phase guidelines, see section 4.6.1, page 64.
- Do not use the detector in ways other than those described in these *Operating Instructions*.
- Keep the operating instructions near the device to be available for quick reference.

1.2.3 Consignes de Sécurité

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

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

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

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

- **Equipment de protection**

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

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

- **Substances dangereuses**

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

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

Manipulez les substances dangereuses, tels que les acides avec prudence. Si des procédures, tels que des procédures de maintenance pour les cellules électrochimiques, requirent l'utilisation des acides, tel que l'acide phosphorique, portez l'équipement de protection personnel et soyez particulièrement prudent.

- **Gaz dangereux**

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

- **Décharge électrostatique**

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

  Prenez des mesures appropriées pour éviter les charges électrostatiques à proximité du système HPLC. Par exemple, assurez-vous qu'il y a une humidité de l'air suffisante et une ventilation adéquate dans le laboratoire. Portez des vêtements ou équipements de protection antistatique. Évitez l'accumulation de bulles d'air dans les lignes de déchets et utilisez des réservoirs à déchets mis à la terre. Utilisez uniquement des capillaires non-conducteurs pour diriger les solvants au réservoir de déchets. Les capillaires électriquement conducteurs doivent être mis à la terre.

- **Inflammation spontanée des solvants**

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

- **Capillaires, connecteurs capillaires, connexions ouvertes**

  - Des capillaires, en particulier les capillaires non-métalliques, pourraient fendre ou glisser des connecteurs ou ne peuvent pas être vissés. Ceci peut en résulter aussi que des substances pourraient jaillir des connexions ouvertes.
  - Dans un système UltiMate 3000, certaines composantes sont en PEEK. Bien que ce polymère présente une excellente résistance chimique à la plupart des solvants organiques, il a tendance à gonfler lorsqu'il est en contact prolongé avec du chloroforme (CHCl3), du diméthyle sulfoxide (DMSO) ou du tetrahydrofuran (THF). De plus, il est attaqué par des acides concentrés tels que l'acide sulfurique et l'acide nitrique ou d'un composé du hexane, éthyle acétate et méthanol. Ceci peut causer des fuites ou des éclatements des capillaires. Ces acides peuvent cependant être utilisés dans le cadre de procédures de nettoyage, à condition que l'exposition soit brève.
  - N'utilisez pas de capillaires écrasés, pliés, abimés ou endommagés.

  - ♦ Les connecteurs capillaires pourraient être contaminés par des substances dangereuses ou des substances dangereuses pourraient sortir des connexions ouvertes.
  - ♦ Portez des lunettes de protection lorsque vous manipulez des capillaires en silice fondue (pendant l'installation, découpe, etc.).

- Débranchez l'instrument de toute source d'alimentation électrique avant de retirer les capots. Quand les capots de protection de l'appareil sont démontés, des connexions électriques sous haute tension deviennent accessibles. Les capots de protection devraient être démontés uniquement par le personnel de service de Thermo Fisher Scientific.
- Remplacez toujours les fusibles grillés par des fusibles de rechange autorisés par Thermo Fisher Scientific.
- Remplacez les câbles de communication défectueux.
- Remplacez les cordons d'alimentation électrique défectueux. Utilisez uniquement les cordons d'alimentation électrique spécifique à l'instrument.
- Utilisez seulement des pièces de rechange originales et des accessoires autorisés par Thermo Fisher Scientific.
- Réglez toujours une limite de pression minimum pour la pompe HPLC. Ceci prévient les dommages résultant des fuites ou du fonctionnement à sec de la pompe sur le long terme.
- 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 substances susceptibles de former des peroxydes.
- Lorsque vous passez d'une solution saline à un solvant organique, effectuez un rinçage intermédiaire du système HPLC à l'eau dé-ionisée ou qualité HPLC.
- Lorsque vous passez à un autre solvant, assurez-vous que le nouveau solvant soit miscible avec celui qui se trouve dans la pompe. Dans le cas contraire, la pompe peut être endommagée; par exemple, par des floculations !
- Si une fuite se produit, arrêtez immédiatement l'instrument, stoppez le débit de la pompe et remédiez au problème.
- Utilisez uniquement des solvants (qualité HPLC) et des solutions salines compatibles avec les matériaux exposés aux phases mobiles.
- Avant d'interrompre le fonctionnement pendant plusieurs jours ou lorsque vous préparez le détecteur pour le transport, observez les précautions figurant en page 98.

- **Cellules électrochimiques sensibles**

  Les cellules électrochimiques sont sensibles. Afin de prévenir tout dommage au détecteur et aux composants du détecteur, observez les consignes de sécurité suivantes :

  - ♦ Assurez-vous de toujours avoir un débit lorsqu'une tension est appliquée. N'utilisez jamais la cellule électrochimique à sec lorsqu'un potentiel est appliqué sur les électrodes, ceci pourrait endommager les électrodes. Mettez en route un débit de phase mobile compatible avec les cellule electrochimiques avant d'allumer la cellule et assurez-vous que lé débit reste en route lorsqu'un potentiel est appliqué afin d'éviter d'endommager la cellule.
  - ♦ Si le trajectoire d'écoulement du système contient des métaux ferreux, tel que l'acier inoxydable, cela peut perturber le fonctionnement des cellules électrochimiques et des électrodes. Passivez les instruments et les composants, avant de raccorder la cellule électrochimique au trajectoire d'écoulement. Pour plus d'informations sur la passivation, référez-vous à la page 131.

- N'utilisez de l'acide nitrique (du HNO_3 concentré) pour nettoyer la cellule électrochimique. Une exposition peut endommager les composants internes sensibles. Consultez le *guide utilisateur* pour la cellule électrochimique respective afin d'obtenir des informations sur les procédures de nettoyage recommandées.

- Utilisez la cellule électrochimique selon les spécifications pour la contre-pression et le potentiel appliqué. Observez les spécifications et les consignes concernant la phase mobile pour les cellules électrochimiques. Pour obtenir les consignes sur la phase mobile, référez-vous à la section 4.6.1, page 64.

- N'utilisez pas l'instrument de manière autre que celles décrites dans ce manuel.

- Conservez ce manuel à proximité de l'instrument pour pouvoir le consulter facilement.

1.3 Intended Use

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

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

The detector is designed for laboratory research use in high-performance liquid chromatography (HPLC) and ultra-high performance liquid chromatography (UHPLC) applications. It is part of the UltiMate 3000 system, but 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™ or Empower™. In this case, installation of additional software is required in addition to the data system software.

For more information, contact the Thermo Fisher Scientific sales organization.

Observe the following when using the detector:

- The detector must be operated only with original accessories and spare parts as recommended by Thermo Fisher Scientific (→ page 125) and within its technical specifications (→ page 123).
- 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.

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

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

1.4 Compliance Information

1.4.1 Declarations of Conformity

The CE Mark label and cTUVus Mark safety label on the rear panel indicate that the detector is compliant with the related standards.

1.4.2 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 electrochemical detector is a modern high-quality instrument designed for the detection of electroactive species, HPLC and UHPLC analysis, especially as part of the UltiMate 3000 system.

- The detector 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.
- Two detection types are available for your analysis:
  - ♦ Coulometric detection
    Provides stability, maintenance-free operation and flow rate independence
  - ♦ Amperometric detection
    Provides ultimate sensitivity, even with volume-limited samples
- The detector can measure up to 4 data channels (depending on the configuration of potentiostat module and electrochemical cells and operating mode) with independent parameters, such as sensitivity or filter constant, simultaneously.
- Different modes of operation are available:
  - ♦ DC Mode: The applied potential is held constant during the analytical measurement.
    For details on the DC Mode, see section 5.5, page 76.
  - ♦ Pulse Mode: The potential is changed at predetermined times during the analysis to recondition the electrode.
    For details on the Pulse Mode, see section 5.6, page 79.
- The integrated controlled column compartment provides accurate column temperature control for reproducible chromatographic results without the need for a separate column oven.
- Full-scale autoranging protects data integrity by preventing that data is lost due to peak over-ranging.
- 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 27).
- 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 (EC) detection involves chemical reactions in which one or more electrons are transferred from one compound (element or ion) to another. In an oxidation, the compound loses one or more electrons; while in a reduction, the compound gains one or more electrons. In order for an oxidation or a reduction to occur, energy in the form of an electric potential is required. In an electrochemical detector, the eluent passes through an electrochemical cell that is equipped with a pair of inert electrodes, and a potential is applied across these electrodes. If the potential is great enough, the reaction will occur and current will flow. The current is measured and related to the amount of compound undergoing electrolysis.

The electrode where the desired electrochemical process occurs is termed the working electrode, while the electrode where the complementary electrolytic reaction takes place is called the auxiliary or counter electrode. A reference electrode is included to provide a stable potential between the working electrode and the counter electrode.

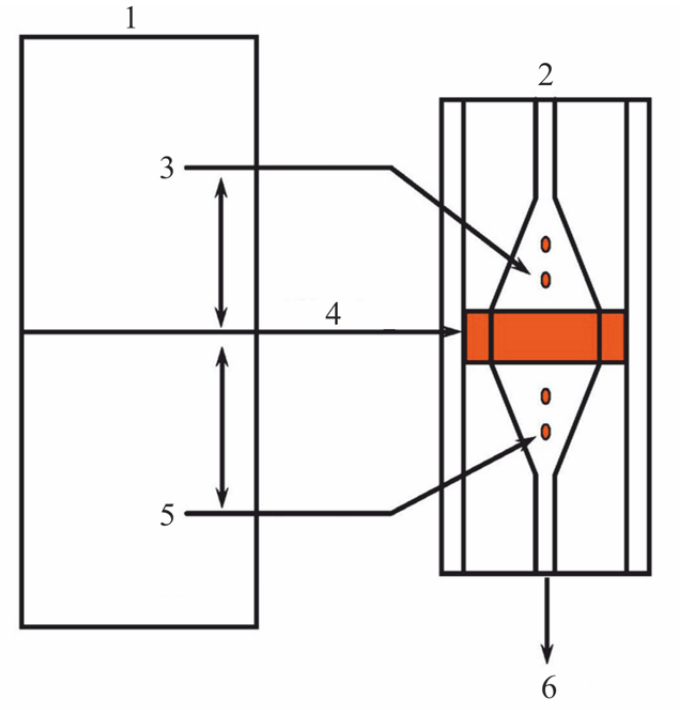


Fig. 1: Schematic of the operating principle

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

There are two approaches to cell design used for electrochemical detection:

- Amperometric Detection
  In amperometric detection, the eluent flows by the electrode surface in the cell. 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 coulometric detection, the eluent flows through a porous graphite electrode contained within the flow path, rather than flowing over the surface of 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 are available for use with the detector to optimize performance for a given application.

2.3 Configurations

To operate the detector, one or more potentiostat modules and analytical electrochemical cells are required that must be ordered separately. The following configurations of the detector components are available:

Description	**Part no.**
ECD-3000RS detector for electrochemical detection The detector is shipped with an accessory kit and operating instructions. For operation of the detector, the following components are required:	5070.0010
Potentiostat module, available as	
Potentiostat module for DC Mode **Note:** Up to four potentiostat modules may be installed.	6070.1400
Potentiostat module for Pulse Mode	6070.1420
For information about potentiostat modules, see section 2.9, page 23.	
Electrochemical cell, available as	
6020RS omni Coulometric Cell Single-electrode coulometric cell, recommended for single-analyte detection, interference screening or analyte conversion	6070.2100
6011RS ultra Coulometric Analytical Cell Dual-electrode coulometric cell for multiple analyte detection	6070.2400
6041RS ultra Amperometric Analytical Cell, for analytical purposes Including • Thin-layer amperometric cell for single-channel detection • Gaskets • Anti-static tweezers *Note:* The 6041RS amperometric cell is shipped without working electrode. Select the required working electrode for the cell.	6070.3000
For use with the following available working electrodes:	
Working electrode (plate type), boron-doped diamond (BDD)	6070.3100
Working electrode (plate type), glassy carbon (GC), high efficiency	6070.3200
Working electrode (plate type), gold (Au)	6070.3300
Thin-film working electrode kit, platinum (Pt), including • Thin-film working electrode, platinum (Pt) • Adapter for thin-film electrode The kit components are also available separately: • Thin-film working electrode, platinum (Pt) (without adapter) • Adapter for thin-film electrode (without electrode)	6070.3510 6070.3500 6070.3005
For further information about electrochemical cells, see section 2.10, page 24.	

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. Do not place any heavy objects on the open front panel door; this may damage the 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	Bays
2	Leak sensor
3	Column compartment (here with cover) (→ page 25)

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 67) - Status screen (→ page 67)
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 a leak has been detected. The LED is orange, for example, during the detector startup. Otherwise, the LED is green.

2.6 Rear Panel

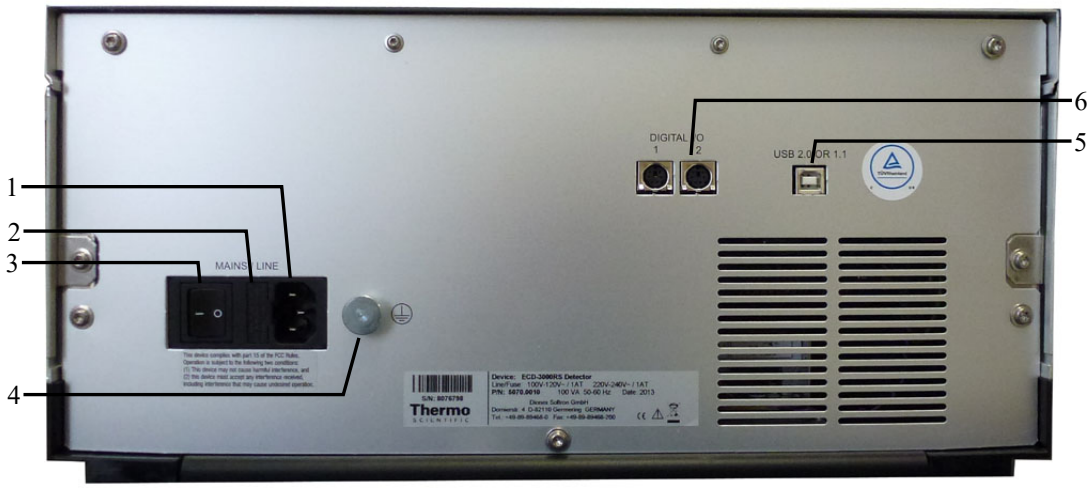


Fig. 4: Rear Panel

No.	Description
1	Main power receptacle (→ page 32)
2	Fuse cartridge (→ page 21)
3	Power switch (→ page 21)
4	Protective grounding
5	USB port (USB 2.0) for connecting the module to the Chromeleon computer (→ page 22)
6	Digital I/O port (→ page 22) for communication with a pump and other external devices, for example, a mass spectrometer

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 98.

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 120.

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. 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 section 3.3 (→ 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 for the LPG-3400XRS pump) or other external devices to exchange digital signals. 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 133.

2.7 Flow 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 analytical 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 provide the space to insert potentiostat modules and connect electrochemical cells to the detector.

When the detector is shipped, the bays are closed with covers 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.

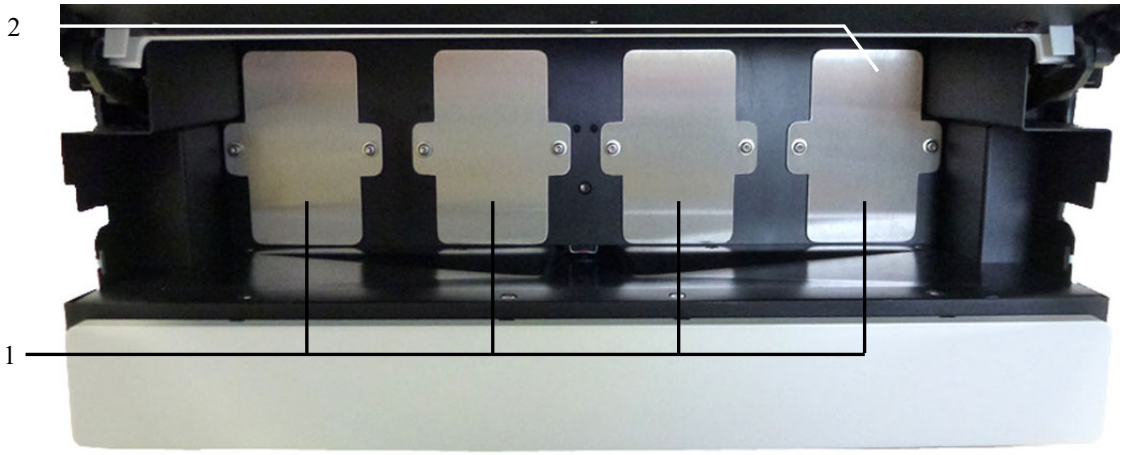


Fig. 5: Bays in the detector

No.	Description
1	Bays Assigned from left to right: A, B, C, D
2	Bay cover, attached to each bay

2.9 Potentiostat Modules

Potentiostat modules are available to be installed in the bays of the detector in order to connect an electrochemical cell. A potentiostat module contains all the necessary electronics to apply a potential to an electrochemical cell and measure the resulting current produced when an analyte undergoes an electrochemical reaction.

Different potentiostat modules are available for different operational modes of the detector. For an overview of the potentiostat modules, see section 2.3, page 18.

The detector is shipped without a potentiostat module. Install the appropriate potentiostat module first.

- For installation guidelines for potentiostat modules, see section 4.3 (→ page 47).
- For installation instructions, refer to the Installation Instructions for potentiostat modules.

Fig. 6: Side and rear view of a potentiostat module for DC Mode

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

2.10 Electrochemical Cells

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

The detector is shipped without an electrochemical cell. For installation, set up, operation, shut down and maintenance of the cells, refer to the respective *User Guide* that is shipped with each electrochemical cell.

Different electrochemical cells are available for different purposes and analyses:

Cell type	Pressure limit	Used for…	Electrode details…
6020RS omni coulometric cell	620 bar	Analytical purposes or screening	Containing one electrode made of micro-porous graphitic carbon
6011RS ultra coulometric analytical cell	40 bar	Analytical purposes or screening	Containing two working electrodes made of micro-porous graphitic carbon
6041RS ultra amperometric analytical cell	13.8 bar	Analytical purposes	Using one working electrode Different types of working electrodes separately available and must be installed before using the cell.

For general guidelines for electrochemical cells, see section 4.3.4 (→ page 50).

Fig. 7: Examples of electrochemical cells
(left: 6011RS ultra coulometric cell; right: 6041RS ultra amperometric cell)

SmartChip Technology

The design of the electrochemical cells incorporates SmartChip™ technology for automatic recognition by the ECD-3000RS electrochemical detector.

For details on this function, see section 5.11 Monitoring System Functions, page 96. For information on the cable connections, see section 3.3.3 Connecting the Digital I/O, page 33.

2.11 Column Compartment

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

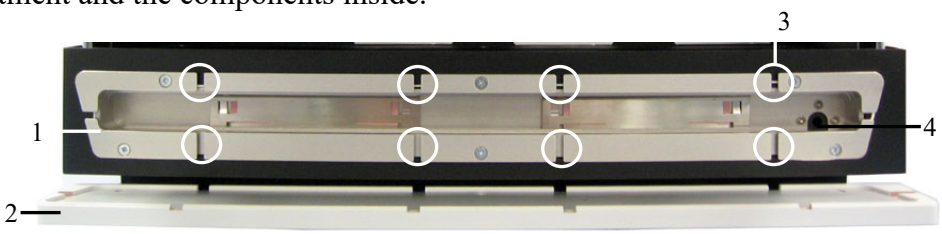


Fig. 8: View into the column chamber

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 4.4.4 (→ page 62).

2.12 Leak Sensor

A leak sensor (→ Fig. 2, page 19) 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, a message appears in the Chromeleon Audit Trail and the electrochemical 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 118). If the sensor is not dry, the Status LED remains red.

2.13 Chromeleon Software

The detector can be controlled by chromatography data systems, such as the Chromeleon Chromatography Management System. To control the detector with Chromeleon, an appropriate Chromeleon version and license are required.

For information about other chromatography data systems, refer to the user documentation and Help of the software.

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

This manual describes the basic steps for operating the detector with the Chromeleon 6.8 software. The steps are identical for the Chromeleon 7 software, but the terminology may differ. For additional information, refer to the *Help* and documents provided with the software.

Detector operation under Chromeleon 7

If you want to operate the module from *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*.

Software control

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 70.

- *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 72.

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 25)

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; → section 5.11, page 96) are available.

3 Installation

3.1 Facility Requirements

The installation site must meet the following requirements:

- The main power switch and the main power receptacle are on the rear panel. Make sure that
  - ♦ Free and unrestricted access to the main power switch is ensured at all times.
  - ♦ The power cord of the device can be easily reached and disconnected from the power line at all times. Provide sufficient space behind the device to unplug the cable.
- Make sure that the installation site meets the power and environmental specifications listed in the *Technical Information* section (→ page 123).
- 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 and Positioning

3.2.1 Unpacking the Detector

All electrical components of the detector are carefully tested before the module is shipped from the factory. After unpacking, inspect the module 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-le par les côtés. Ne soulevez l'instrument qu'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.

3.2.2 Scope of Delivery

The following items are included in the delivery:

- Detector
- Standard accessory kit
  For details about the content of the kit, see section 9.1, page 125.
- Operating instructions
- Power cord

Tip: The detector is shipped without potentiostat module and without analytical electrochemical cell. Potentiostat modules and electrochemical cells are available separately.

For a complete installation of the detector, make sure that the required potentiostat modules and electrochemical cells are available.

3.2.3 Positioning the Detector in the UltiMate 3000 System

If the detector is part of an UltiMate 3000 system, for example for analytical HPLC applications, stack the individual modules and interconnect them on the rear panel, for example as shown below (→ 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.

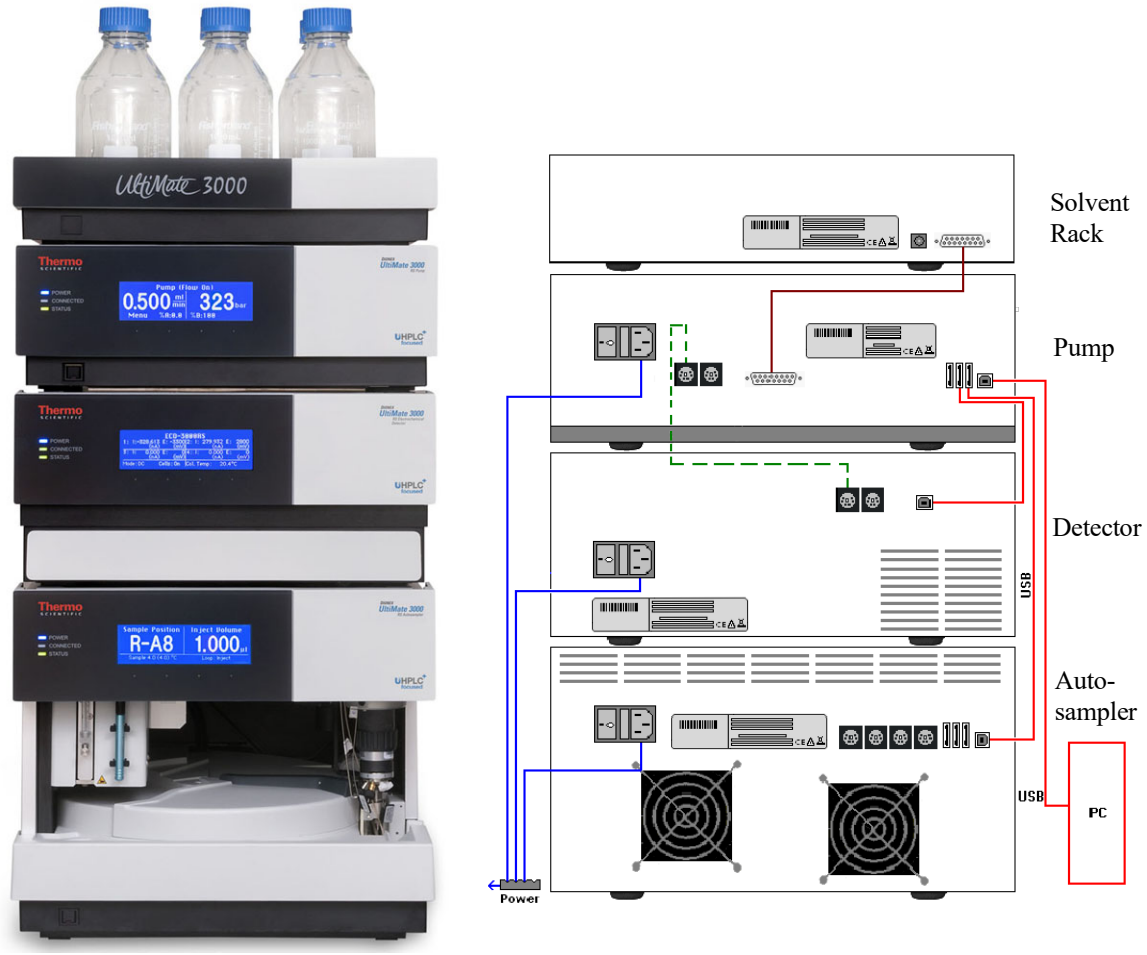


Fig. 9: Module arrangement and rear panel connections for an UltiMate 3000 system with ECD-3000RS detector (example)

Note the following for cable connections in an UltiMate 3000 system with the 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 modules in the UltiMate 3000 system or directly to the computer.

- Thermo Fisher Scientific recommends interconnecting all modules of the system, 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.
- Apart from the Solvent Rack, all modules of the UltiMate 3000 system can be connected also separately to the Chromeleon computer by using the USB port on the rear panel of the module.
- 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 3.3.3 (→ page 33).

For information how to connect the USB port on the rear panel, refer to section 3.3.1.

3.3 Connecting the Detector

3.3.1 Connecting the Power Cord

Parts required

Power cord from the accessories kit for the detector to connect the detector to the main power source

Follow these steps

Connect the power cord from the main power receptacle on the rear panel (→ Fig. 4, page 21) to a grounded power source. No manual adjustment is required to adapt the line voltage to local voltage requirements.

Warning: Never use a power cord other than the power cords provided for the device.

Do not use multiple sockets or extension cords. Using defective multiple sockets or extension cords may cause personal injury or damage to the device.

Avertissement : Utilisez uniquement les cordons d'alimentation électrique spécifique à l'instrument.

N'utilisez pas des blocs multiprise ou des câbles prolongateurs. Cela pourrait entraîner des blessures corporelles ou endommager l'instrument.

3.3.2 Connecting the USB Cable

Connect the detector to the data system computer via the USB port on the rear panel (→ Fig. 4, page 21).

Parts required

USB cable, type A to type B, high speed USB 2.0, from the accessories kit for the detector

Tip: To ensure trouble-free operation, use only the cables shipped with the detector. The PC must be equipped with a USB 2.0 port.

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.

Preparations

If you want to operate the detector from Chromeleon chromatography software

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 upon power-up.

Follow these steps

Connect the USB port to the computer using one of the following connection options:

- 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. 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 3.3.3 (→ page 33).

  Tip: It is not possible to use the USB hub on the UltiMate 3000 autosampler for connection of the detector to the computer.

3.3.3 Connecting the Digital I/O

The detector is equipped with two Digital I/O ports which allow connection to external devices, such as an UltiMate 3000 pump (except the LPG-3400XRS pump) for communication between pump and detector.

If the mini-DIN signal cable is connected between an UltiMate 3000 pump (except the LPG-3400XRS pump) and Digital I/O port 2 on the detector, the cell chip is also able to sense pump flow errors from the connected pump, such as flow interruptions, and as a result turns off potential to the cell.

For information about the **Cells Off** function, see section 5.11, page 96. For information about the functions of the connector pins and pin assignment, see section 10.2, page 133.

Parts required

Mini-DIN signal cable from the accessory kit for the detector to connect pumps of the UltiMate 3000 series (except the LPG-3400XRS pump)

Follow these steps

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

1. Plug the 6-pin connector of the mini-DIN cable into the Digital I/O port **2** on the detector to use the **Cells Off** function for protection of the electrochemical cells in case of a pump flow interruption.
2. Plug the 6-pin connector of the mini-DIN cable into the Digital I/O port on the pump.

3.4 Setting Up the Detector in Chromeleon

This section provides brief instructions for setting up the detector in Chromeleon. For details, 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.

3.4.1 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. *Depends on the operating system*

   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 power to the detector. 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:

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

   b) Turn off the power to the detector.

c) Install Chromeleon.

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

3.4.2 Installing the Detector

Tip: Before you install and connect the detector in Chromeleon, have the detector complete the self-test (→ page 67) 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 3.4.3 (→ 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.

3.4.3 Configuring the Detector

3.4.3.1 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 37).

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**.

3.4.3.2 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 3.4.3.3 (→ page 37).
4. To save the changed configuration, click **Save** on the **File** menu and then close the **Server Configuration** program.

3.4.3.3 Configuration Pages

The detector configuration comprises the following dialog pages and settings:

- **General** dialog page (→ page 38), including connection and firmware settings
- **Detector** dialog page (→ page 39), including operational mode settings, bay selection and cell properties
- **Signals** dialog page (→ page 43), including available signal channels
- **Inputs** dialog page (→ page 44), including available inputs

General Page

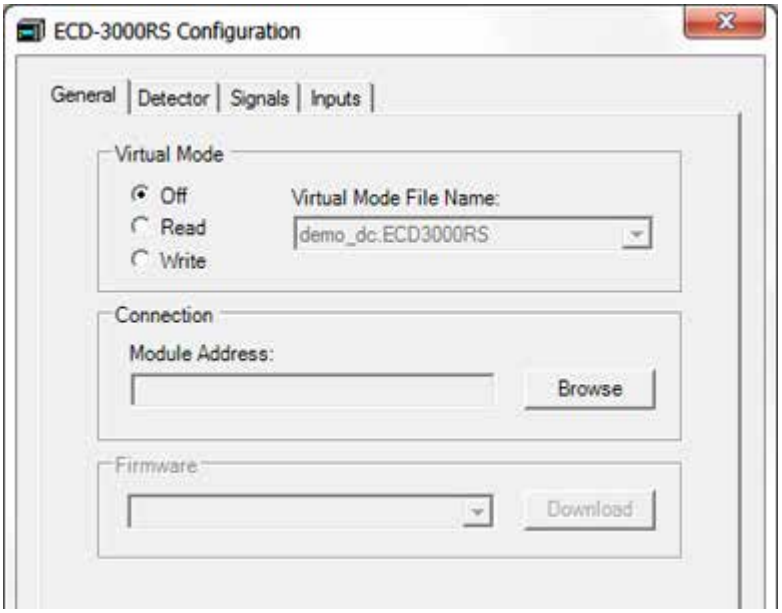


Fig. 10: General page

Setting	Description
Virtual Mode	In the virtual mode, Chromeleon simulates detector control and data acquisition. 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.
	To use the virtual mode: • 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	1. Select the module address of the detector if necessary. The module address states the USB port and the serial number of the detector. 2. 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 detector firmware to Chromeleon, setting the options on the pages accordingly. 3. 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 121).

Detector Page

Shows the configuration of the potentiostat module and cells currently installed on the detector.

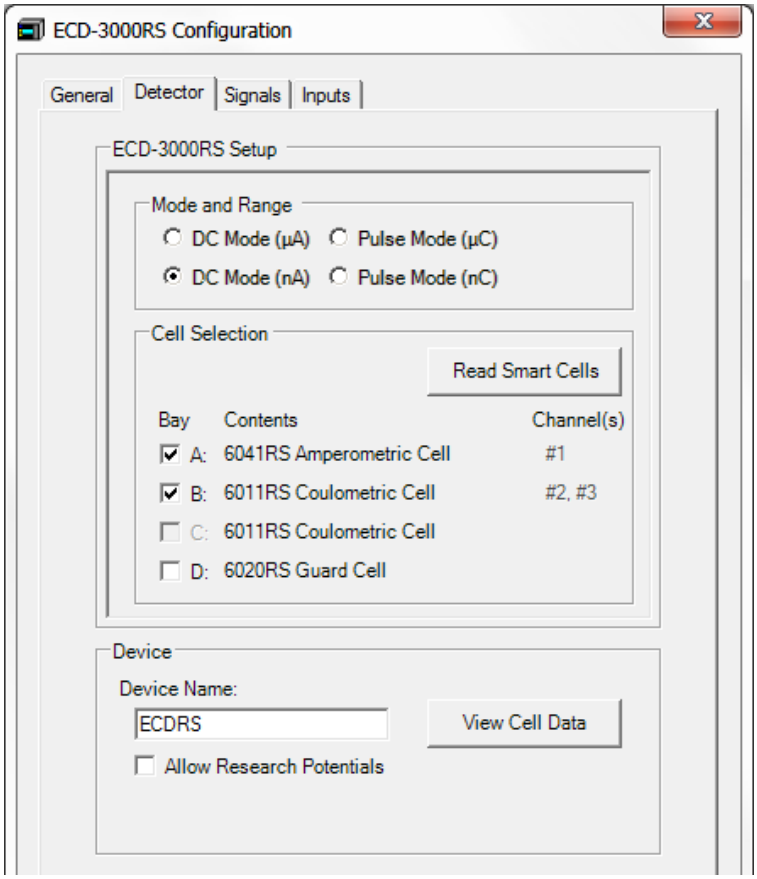


Fig. 11: Detector page

Setting		Description	
Mode and Range		Select the desired level of sensitivity for the operational mode that is used. The selected sensitivity affects the maximum signal range and the signal unit. The selections available depend on the potentiostat module that is installed for the required operational mode. For details on the DC Mode, see section 5.5 (→ page 76). For details on the Pulse Mode, see section 5.6 (→ page 79).	
	DC Mode (nA)	*Requires a potentiostat module for DC Mode* During DC Mode operation, this mode is selected by default. Select this mode to measure with high sensitivity in a narrow signal range (when the expected magnitude of the background current is less than 2147.4 nA or 2.1 µA).	
		Signal unit: nA	Signal range: min. -2147.4 nA / max. +2147.4 nA

Setting		Description	
	DC Mode (µA)	*Requires a potentiostat module for DC Mode* Select this mode to measure with low sensitivity in a wider signal range (when the expected magnitude of the current is greater than 2147.4 nA or 2.1 µA). **Tip:** When selecting the data range, be aware that in Chromeleon 6.80 high data (current greater than 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.	
		Signal unit: µA	Signal range: min. -100.00 µA / max. +100.00 µA
	Pulse Mode (nC)	*Requires a potentiostat module for Pulse Mode and an amperometric cell with noble-metal-based working electrode* During Pulse Mode operation, this mode is selected by default. Select this mode to measure with high sensitivity in a narrow signal range (when the expected magnitude of the background current is less than 2000 nC or 2 µC).	
	Pulse Mode (µC)	*Requires a potentiostat module for Pulse Mode and an amperometric cell with noble-metal-based working electrode* Select this mode to measure with low sensitivity in a wider signal range (when the expected magnitude of the background current is greater than 2000 nC or 2 µC).	
Cell Selection		Shows the four bays, the installed potentiostat modules, and the cells connected to the potentiostat modules. • To display the cells that are installed in the potentiostat modules in the respective bays, click **Read Smart Cells**. • If you add or remove cells or potentiostat modules from the detector, click **Read Smart Cells** to refresh and display the cell details.	
	Bays	Select the **Bay** checkbox for each cell which you want to use for detection. **Note:** The selectable bays depend on the configuration of the potentiostat modules and electrochemical cells and the maximum number of data signal channels that is possible with this configuration. For details, see section 4.3.1, page 47.	
	Contents	• Before the first configuration of the bays or if no potentiostat module is installed in a bay, the **Contents** of the bay is displayed as **Bay is empty**. • If no cell is installed in a bay, the **Contents** of the bay is displayed as **No cell detected**. • Under **Contents** the cell model number and type of the cells installed to the potentiostat modules are shown. • When the detector is connected in Chromeleon, the data stored on the cell chips are compared to the **Contents** data. If the data is not the same, a warning is displayed and the detector cannot be connected.	
	Channels	• The signal channels are assigned automatically. The signal channel numbers are assigned in the order of the bays, from left to right. • Amperometric cells use one data signal channel; coulometric cells use two data signal channels. For more information about the data acquisition, see page 82.	

Setting	Description
Device	Shows details on the connected device.
Device Name	• Displays the name used to identify the detector the installation environment and in the Chromeleon client program. • The default Device Name is **ECDRS**. • 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.
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. For details on the **Cell Properties** dialog box, see below.
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 76. • 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 applying 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.

Cell Properties Page

Click **View Cell Data** on the **Detector** page to open the **Cell Properties** page.

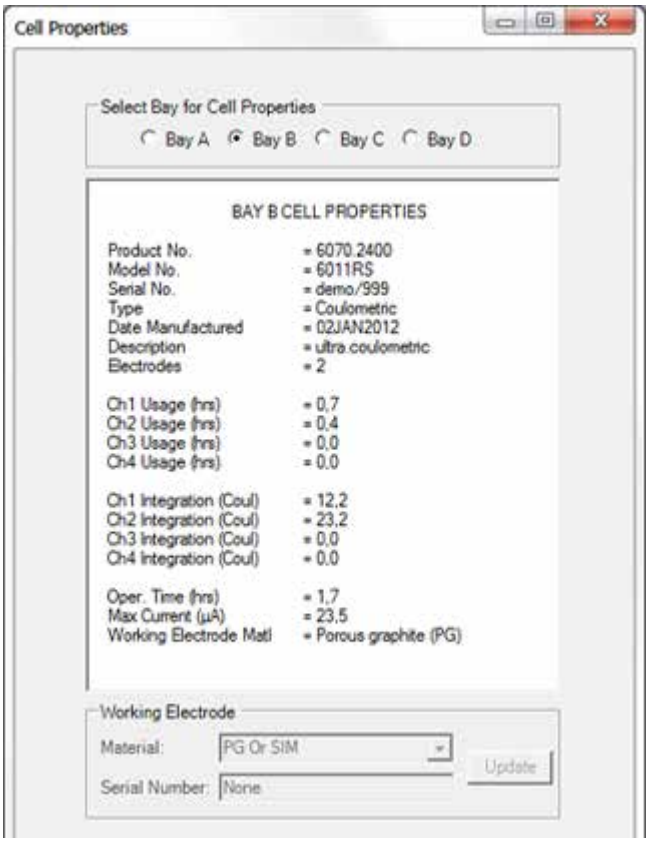


Fig. 12: Cell Properties page

Setting		Description
Cell Properties		Shows all of the data stored on the cell chip for the electrochemical cell in the selected bay, such as type, cell serial number, number of electrodes, channel usage time, operation time and working electrode material, etc. The cell properties provide important information for monitoring the performance of the electrochemical cells (→ section 5.11, page 96).
	Bay A (B, C, D)	Select the respective bay to view the properties of the cell attached to the bay.
	Working Electrode	The required settings depend on the installed cell:
		Coulometric cell: The working electrode is read from the cell chip and cannot be changed.

Setting		Description
	Working Electrode (continued)	*Amperometric cell:* The material and serial number/lot number of the working electrode that is installed in the amperometric cell must be selected: 1. 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**. 2. Enter the serial number of the plate working electrode or lot number of the thin-film electrode in order to accurately record the cell properties. For more information, refer to the *User Guide* for the amperometric cell and working electrodes. 3. Click **Update** to save the selected material. A message is displayed to confirm or cancel the change of the working electrode setting. 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. **Tip:** 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

Setting		Description
Signals		Lists all signal channels that the detector can record, independent of the potentiostat module and cell configuration. 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. The channel is then available in Chromeleon, for example, in the **Commands** (F8) dialog box for the detector. 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.
	Type	Indicates the type of the signal. In a **Current** signal channel, the detector records background current signals when the autozero is performed. The **Temperature** signal channel records the column oven temperature. With this setting, Chromeleon generates the appropriate channel for recording the column compartment temperature signal.
	Unit	In the column **Unit** of the detector signals ("ECDRS"), the value **Default** corresponds to the unit of measurement of the selected operating mode, as was defined on the **Detector** page (either µA or nA). You do not need to change the value in the **Unit** field manually for the detector signals.

Inputs Page

Setting		Description
Inputs		The **Inputs** page lists all available remote inputs. • If you wish to monitor the state of an input in the **Commands** (F8) dialog box or with a control panel in Chromeleon, select the checkbox of the input. • 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.
	ECDRS_Input_1	Designated as Inject Start/Stop
	ECDRS_Input_2	Designated as Autozero
	ECDRS_Input_3	Designated as Cells Off For communication with an UltiMate 3000 pump and pumps of other manufacturers and to monitor the Cells Off safety feature. For more information about the cell safety feature, see section 5.11 (→ page 96).

3.5 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 (→ *DCMSLink Installation Guide*).
2. Open the Chromeleon **Server Configuration** program (→ *DCMSLink Installation Guide*).
3. In the **Server Configuration** program, add the detector to the timebase. Follow the appropriate steps in section 3.4.2 (→ page 36).
4. Configure the detector as described in section 3.4.3 (→ 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 Overview

After you have unpacked, positioned and connected the detector as described in sections 3.1 through 3.3 (→ page 29 and following), prepare the detector for operation and follow the sequence of steps below:

1. Connect the drain tubing (→ section 4.2, page 46).
2. Install one or more potentiostat modules to the desired bays (→ follow the instructions in the *Installation Instructions* for the respective potentiostat module).
3. Power up the detector (→ section 5.1, page 67).

   Tip: The detector should perform the self-test *before* you connect the detector to the computer to prevent possible installation errors.
   The green **Status** LED on the front panel indicates that the self-test is complete. The detector can be connected to the computer.

4. Set up the detector in Chromeleon (→ section 3.4 (→ page 34).
5. Perform a relay test with each installed potentiostat module using the SimulatorRS cell (→ section 4.3.3, page 48).
6. Set up the system flow connections in the flow path before the detector (→ section 4.4, page 52). The electrochemical cell or cells will be connected in the flow path later.
7. Flush the system modules in the flow path before the detector to waste using an appropriate mobile phase (→ see *Operating Instructions* for the pump) for at least 1 hour at a flow rate of 1 mL/min to remove any potential contaminants.
8. Install one or more electrochemical cells: Connect each cell to a potentiostat module, set up flow connections, and flush the cell (→ follow the instructions in the *User Guide* of the respective cell).

   Observe the guidelines for mobile phases (→ section 4.6, page 64).

   Tip: For general guidelines on electrochemical cells, see section 4.3.4, page 50.

9. Configure the electrochemical cell or cells (→ follow the instructions in the *User Guide* of the respective cell).
10. Perform an equilibration of the system and each electrochemical cell:
    a) Equilibrate the system (→ section 4.5, page 63).
    b) Equilibrate each electrochemical cell (→ follow the instructions in the *User Guide* of the respective cell).

After a successful equilibration, the detector and any installed cells are ready for operation (→ section 5 Operation and Maintenance, page 67). Before starting an analysis, check the operating parameters, such as the leak sensor setting if necessary (→ section 5.4, page 74).

4.2 Connecting the Drain System

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

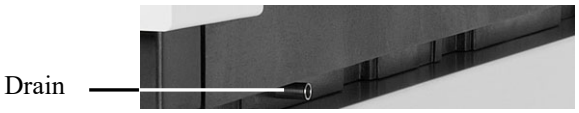


Fig. 13: Drain port

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.

4.3 Potentiostat Modules and Electrochemical Cells

4.3.1 Configurations of Electrochemical Cell and Potentiostat Module

The following tables provide an overview of the cell combinations, the correspondingly available signal channels and configurations of potentiostat modules.

DC Mode Operation

Cell type & combination	Electrodes: number & type	Data channel	Working electrode material	Potentiostat module: number & type
6011RS cell	2 electrodes, coulometric	2	Porous graphite	1 dual-channel DC Mode potentiostat module
6011RS cells, 2x	4 electrodes, coulometric	4		2 dual-channel DC Mode potentiostat modules
6011RS cells, 3x	6 electrodes, coulometric	6		3 dual-channel DC Mode potentiostat modules
6020RS cell	1 electrode, coulometric	1 of 2		1 dual-channel DC Mode potentiostat module
6020RS cell & 6011RS cell	3 electrodes: 1 screening, 2 coulometric	3 of 4		2 dual-channel DC Mode potentiostat modules
6020RS cell & 2x 6011RS cell	5 electrodes: 1 screening, 4 coulometric	4 of 5		3 dual-channel DC Mode potentiostat modules
6020RS cell & 3x 6011RS cell	7 electrodes: 1 screening, 6 coulometric	6 of 7		4 dual-channel DC Mode potentiostat modules
6020RS cell & 6041RS cell	2 electrodes: 1 screening, 1 amperometric	2 of 2	Porous graphite & glassy carbon or Boron-doped diamond	2 dual-channel DC Mode potentiostat modules

Tip: Due to its wetted parts, the UltiMate 3000 ISO-3100BM pump is recommended for high-sensitivity DC Mode operation to obtain best results.

Pulse Mode Operation

Cell type & combination	Electrodes: number & type	Data channel	Working electrode material	Potentiostat module: number & type
6041RS cell	1 electrode, amperometric	1 of 1	Gold (Au) or Platinum (Pt)	1 Pulse Mode potentiostat module

Tip: Due to its wetted parts, the UltiMate 3000 ISO-3100SD and LPG-3400SD pumps are recommended for Pulse Mode operation in carbohydrate analysis to obtain best results.

4.3.2 Potentiostat Module Guidelines

The detector is shipped without a potentiostat module. Install a potentiostat module to operate the detector with electrochemical cells. Refer to the *Installation Instructions* for potentiostat modules.

Note the following:

- Up to four individual potentiostat modules with any combination of type can be installed to the detector.
- A relay test is required after installation or replacement of a potentiostat module. The relay test to be performed depends on the installed potentiostat module type.
- *When using a potentiostat module for DC Mode*
  Up to 4 dual-channel DC potentiostat modules may be installed. DC mode detection may be used with either coulometric cells or amperometric cell.
- *When using a potentiostat module for Pulse Mode*
  With a potentiostat module for Pulse Mode, installation of an amperometric cell with a noble metal-based working electrode such as gold is required.

Fig. 14: Potentiostat module installed

4.3.3 Performing a Relay Test for a Potentiostat Module

When

After installation and replacement of the potentiostat module, before you install an electrochemical cell or a Qualifier RS cell to the potentiostat module, perform a relay test (Ohm's law test) with the module.

The relay test ensures the proper functionality of the potentiostat board before you connect an electrochemical cell, such as the 6041RS ultra amperometric cell or the 6011RS omni coulometric cell.

Parts required

SimulatorRS cell (included in the accessories kit of the detector)

Preparations

1. Install the potentiostat module to the bay to which you want to install the cell. Refer to the *Installation Instructions* for the potentiostat module.
2. *If not yet done:* With only the potentiostat module installed (no cell attached), turn on the detector and wait until the self-test is completed.

   ⚠ **Important:** The detector must perform the self-test *before* you install an electrochemical cell. If the detector attempts to perform a self-test after you have installed the cell, the self-test may fail. In this case, uninstall the cell and re-perform a self-test.

   ⚠ **Important :** *Avant* d'installer la cellule électrochimique, le détecteur doit effectuer le test automatique. Si le détecteur essaye d'effectuer un test automatique après l'installation de la cellule, le test automatique peut échouer. Dans ce cas, déinstallez la cellule et recommencez le test automatique.
3. Install the SimulatorRS cell to the potentiostat module. Refer to the *Installation Instructions for Simulation Cells*.

Follow these steps

The relay test depends on the potentiostat module for which the test is to be performed.

1. Depending on the installed potentiostat module, perform the required relay test:
   - *Performing a Relay Test for DC Potentiostat Modules*

     –or–
   - *Performing a Relay Test for Pulse Potentiostat Modules*

   Refer to the respective sections in the *Installation Instructions for Simulation Cells*.

   Tips:
   - If the relay test was performed after a cold start of the detector, let the detector warm up for 30 minutes prior to the relay test.
   - Before running the relay test, it may also help to perform an Autozero in Chromeleon to re-calibrate the zero current. For details on the Autozero, refer to section 5.4 (→ page 74).
2. If the relay test has been successful, the potentiostat module is ready for installation of an electrochemical analytical cell or QualifierRS cell. Continue the steps in section 4.1, page 45.

   If the relay test repeatedly fails, the potentiostat module and/or the SimulatorRS cell may be damaged. In this case, try the following:

- Replace the SimulatorRS cell (→ *Installation Instructions for Simulation Cells*).
- Replace the potentiostat module (→ section 7.2, page 114).

Re-perform the relay test. If the test is successful, the first SimulatorRS cell or potentiostat module was damaged.

4.3.4 Electrochemical Cell General Guidelines

Cells are sensitive parts and have to be handled with care. Make sure to follow the guidelines provided in the *User Guide* of the respective electrochemical cell.

For details on installation, operation and maintenance for the electrochemical cells, refer to the *User Guide* for the respective electrochemical cell.

⚠ **Important:** **Pressure limits for 6041RS and 6011RS electrochemical cells**
Mind the following maximum operating pressure limits:

- *Amperometric Cell*
  The maximum operating pressure limit for the 6041RS ultra amperometric cell is 13.8 bar (200 psi, 1.38 MPa). Exceeding the pressure may cause leakage at the cell gasket.
  To avoid damage to the cell and restriction to flow, an analytical electrochemical cell must always be the last component in the system flow path.
- *Coulometric Cells*
  Mind the maximum operating pressure limits for coulometric cells:
  6011RS ultra coulometric cell: 40 bar (580 psi, 4.0 MPa)
  6020RS omni coulometric cell: 620 bar (9000 psi, 62 MPa)
  Exceeding this pressure may lead to leaks in the cell.
  To avoid effects of pressure damage, the 6011RS ultra analytical coulometric cell must always be positioned after the analytical column in the system flow path.

⚠ **Important :** **Limites de pressions pour les cellules électrochimiques 6041RS et 6011RS**

- *Cellule ampérométrique*
  La limite de pression pour la cellule ampérométrique 6041RS ultra s'élève à 13.8 bar (200 psi, 1.38 MPa). Le dépassement de cette limite peut provoquer des fuites au joint de la cellule.
  La cellule ampérométrique doit toujours être le dernier élément dans le circuit fluidique, afin d'éviter d'endommager la cellule et des restrictions de débit.

- *Cellule coulométrique*

  Tenez compte des limites de pression pour les cellules coulométriques :

  Cellule coulométrique 6011RS ultra : 40 bar (580 psi, 4.0 MPa)

  Cellule coulométrique 6020RS omni : 620 bar (9000 psi, 62 MPa)

  Afin d'éviter tout dommage par la pression, la cellule coulométrique doit toujours être placée après la colonne analytique dans le trajectoire d'écoulement du système.

⚠ **Important:** If the system flow path contains ferrous metals, such as stainless steel, this can disrupt operation of electrochemical cells and electrodes. Passivate the instruments and components before connecting the electrochemical cell in the flow path.

For passivation instructions, see section 10.1 (→ page 131).

⚠ **Important:** La non passivation des composants dans le trajectoire d'écoulement du système qui contiennent des metáux ferreux, tel l'acier inoxydable, peut perturber le fonctionnement des cellules électrochimiques et des électrodes. Passivez les instruments et composants avant de raccorder les cellules électrochemiques au trajectoire d'écoulement.

Pour obtenir des instructions sur la passivation, référez-vous à la section 10.1, page 131.

4.4 Flow Connections

⚠ **Important:** If the system flow path contains ferrous metals, such as stainless steel, this can disrupt operation of electrochemical cells and electrodes. Passivate the instruments and components before connecting the electrochemical cell in the flow path.

For passivation instructions, see section 10.1, page 131.

In addition, note that Titanium components in the system flow path are not compatible with PAD detection (Pulse Mode operation) when using mobile phase with a pH value >12.

⚠ **Important :** La non passivation des composants dans le trajectoire d'écoulement du système qui contiennent des metáux ferreux, tel l'acier inoxydable, peut perturber le fonctionnement des cellules électrochimiques et des électrodes. Passivez les instruments et composants avant de raccorder les cellules électrochemiques au trajectoire d'écoulement.

Pour obtenir des instructions sur la passivation, référez-vous à la section 10.1, page 131.

De plus, observez que des composants en titanium dans le trajectoire d'écoulement ne sont pas compatibles avec la détection ampérométrique pulsée (fonctionnmenet en Pulse Mode) lorsqu'une phase mobile à valeur pH > 12 est utilisée.

4.4.1 Tips and 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:

- Observe the precautionary statements for capillaries and capillary connections in section 1.2.2 (→ page 3).
- 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.

4.4.2 Setting Up Flow Connections

The capillary connections between the detector and other modules depend on the system configuration and application. If the detector is part of an UltiMate 3000 system with a pump and an autosampler, a top-down fluidic path from pump to autosampler, with the detector in between provides the best arrangement (→ Fig. 9, page 31).

Parts required

Depending on the system operating pressure range, select the required capillary kit:

Description	Maximum system operating pressure	Part no.
Tubing and fitting kit (for HPLC configurations) Including necessary capillaries and fittings. *Recommended for connections to third-party HPLC systems.*	300 bar (4350 psi, 30 MPa)	Included in accessory kit
nanoViper capillary kit, PEEK, ultra-high sensitivity, for UltiMate 3000 systems with ECD-3000RS detector Including necessary capillaries and fittings to connect an UltiMate 3000 system.	1200 bar (17400 psi, 120 MPa)	6041.5105
The kits include all necessary capillaries and fittings to connect the detector as shown in Fig. 15, page 56.		

With system operating pressures below 300 bar, you can use the in-line filter kits:

Description	Part no.
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")	Included in accessory kit
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")	Included in accessory kit

⚠ **Important:** Use in-line filters only with system operating pressures below 300 bar. For applications with system operating pressures above 300 bar, remove the in-line filters from the system flow path.

⚠ **Important :** Utilisez des filtres en ligne uniquement avec des pressions inférieures à 300 bar. Pour des appplications avec des pressions supérieures à 300 bar, retirez les filtres en ligne du tracjectoire d'écoulement du système.

Preparations

Observe the general precautions for connecting capillaries (→ section 4.4.1, page 52).

Overview

- Tubing and fitting kit (operating pressures below 300 bar)
  See section 4.4.2.1, page 56.
- nanoViper capillary kit (operating pressures below 1200 bar)
  See section 4.4.2.2, page 58.

Follow these steps

Follow the steps below for all types of tubing.

1. *With system operating pressures below 300 bar only*
   Connect the graphite in-line filter to the pump outlet. See section 4.4.3.1, page 60.
2. Connect a capillary from the outlet of the graphite in-line filter or pump outlet to the inlet of the 6020RS omni coulometric cell.
3. Connect a capillary from the outlet of the 6020RS omni coulometric cell to the inlet of the autosampler.
4. Connect a capillary from the autosampler outlet to the waste.
5. To remove any contaminants, flush the system with mobile phase, for example with a methanol/HPLC-grade water solution, for 3-5 minutes at a flow rate of 1.0 mL/min. Monitor the fittings, and tighten them as appropriate. If you observe a significant increase in pressure, check if an obstruction or clogged component is present in the fluidic path. If it is, remove the obstruction.
6. Connect the capillary from the autosampler outlet to the column inlet.
7. Place the column in the column compartment (→ page 62).
8. *With system operating pressures below 300 bar only*
   Connect the PEEK in-line filter. See section 4.4.3.2, page 61.
9. Before connecting the analytical electrochemical cell to the system, flush the column with at least 200 mL mobile phase for several hours. Solvents that may be contained in the column should be flushed out with the mobile phase before initial use.
10. Connect a capillary from the PEEK in-line filter or column outlet to the inlet of the analytical electrochemical cell.
11. Connect the waste line to the outlet of the analytical electrochemical cell and route the waste line to the waste.
12. Start the delivery of the mobile phase (for example with a methanol/HPLC-grade water solution). Flush the system for 3-5 minutes at a flow rate of 1.0 mL/min to remove any contaminants from the system.

13. Monitor the fittings and tighten as appropriate. If you observe a significant increase in pressure, check if an obstruction is present in the fluidic path. Remove the obstruction, if possible.

4.4.2.1 Connections with the Tubing and Fitting Kit (< 300 bar)

For a detector configuration consisting of a 6020RS omni coulometric cell and an analytical cell (6011RS ultra or 6041RS ultra analytical cell), the standard connections with the Tubing and fitting kit are as follows:

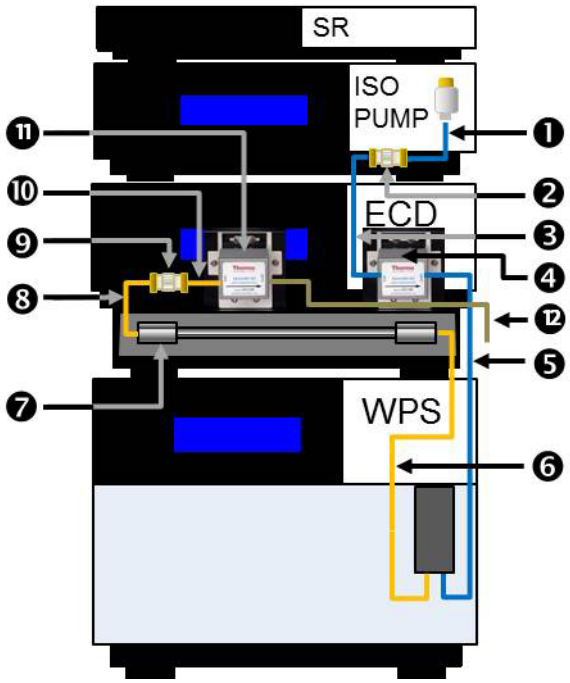


Fig. 15: Standard system capillary connections (< 300 bar) with the detector

Connect the capillaries and fittings as follows. If necessary, use the tubing cutter shipped with the kit to cut a capillary to length and to obtain the required capillary connection.

⚠ **Important:** Use in-line filters only with system operating pressures below 300 bar. For applications with system operating pressures above 300 bar, remove the in-line filters from the system flow path.

⚠ **Important :** Utilisez des filtres en ligne uniquement avec des pressions inférieures à 300 bar. Pour des appplications avec des pressions supérieures à 300 bar, retirez les filtres en ligne du tracjectoire d'écoulement du système.

No.	Components or connections	Parts overview	
		Using capillaries and fittings…	**Part no.**
1	Pump outlet to inlet of graphite in-line filter	Tubing, PEEK, I.D. 0.015" from tubing kit	6081.1420
		Lock-nut from in-line filter kit	70-3675
2	In-line filter with graphite filter element See section 4.4.3.1 (→ page 60).	---	70-0893
3	Outlet of graphite in-line filter to inlet of 6020RS omni coulometric cell	From tubing kit: • Tubing, PEEK, I.D. 0.015" • Fingertight two-piece fitting	 6081.1420 6000.0011
		Lock-nut from in-line filter kit	70-3675
4	6020RS omni coulometric cell	---	---
5	Outlet of 6020RS omni coulometric cell to inlet of autosampler, such as a WPS-3000TBRS autosampler	From tubing kit: • Tubing, PEEK, I.D. 0.015" • 2 Short lock-nut fittings, or • 2 Fingertight two-piece fittings	 6081.1420 70-4746 6000.0011
6	Outlet of autosampler to inlet of analytical column	From tubing kit: • Tubing, PEEK, I.D. 0.005" • Short lock-nut fitting • Fingertight two-piece fitting	 6081.1410 70-4746 6000.0011
7	Analytical column	---	---
8	Outlet of analytical column to inlet of PEEK in-line filter	From tubing kit: • Tubing, PEEK, I.D. 0.005" • Short lock-nut fitting	 6081.1410 70-4746
		Lock-nut from in-line filter kit	70-3675
9	In-line filter with PEEK filter element See section 4.4.3.1 (→ page 60).	Included in-line filter kit	70-4093
10	Outlet of PEEK in-line filter to…	Tubing, PEEK, I.D. 0.005" from tubing kit	6081.1410
	…inlet of 6011RS ultra coulometric cell	2 lock-nuts from in-line filter kit	70-3675
	or		
	…inlet of 6041RS ultra amperometric cell	Fitting from in-line filter kit	70-3675
		Fitting from cell ship kit	---
11	Analytical cell 6011RS or 6041RS For connecting multiple cells in series	--- Interconnecting Viper capillary, included in detector accessory kit	--- 6041.9075

No.	Components or connections	Parts overview	
		Using capillaries and fittings…	**Part no.**
12	Electrochemical cell outlet to waste Cell waste line included in detector accessories kit, fitting for cell connection included in cell accessory kit	From cell waste line kit: • Waste tubing • One-piece fitting	6070.4900

For details on the capillaries, fittings and lock nuts, see section 9.1 (→ page 125).

Tips:

- Alternatively to using the fingertight fitting 6000.0011, you can use the long seal-tight lock nut with ferrule (part no. 70-4859) from the tubing kit.
- If the space before a fluidic inlet or after a fluidic outlet is too tight for a long fitting (for example, the fitting with part no. 70-3675), use the short fitting (part no. 70-4746) from the tubing and fitting kit.

  Example: Use the short fittings when you use a column that is 25 cm long, and the cell is installed in Bay A, where the space between cell inlet and enclosure compartment is tight.

4.4.2.2 Connections with the nanoViper Capillary Kit (< 1200 bar)

For a detector configuration consisting of a 6020RS omni coulometric cell and an analytical cell (6011RS ultra or 6041RS ultra analytical cell), the connections with the optional nanoViper capillary kit correspond to the connections in the system as shown in Fig. 15 (→ page 56).

Important : Mobile phases with a high pH value can cause damage to the capillaries from the nanoViper capillary kit. Avoid using the capillaries from these kits with high pH mobile phases, as are typically used for Pulse Mode applications, such as sodium hydroxide (NaOH), or Potassium hydroxide (KOH).

Important : Les phases mobiles à valeur pH élevée peuvent endommager les capillaires contenus dans le kit nanoViper. Évitez d'utiliser des capillaires contenus dans ces kits avec des phases mobiles aux valeurs de pH élevées, typiquément utiliseés avec les applications Pulse Mode, telles que le hydroxide de sodium (NaOH), ou le hydroxide de Potassium (KOH).

Connect the capillaries as follows:

No.	Components or connections	Part no. capillary (or kit) with fittings
1	Pump outlet to inlet of 6020RS omni coulometric cell	6041.5819
2, 3	In-line filter with graphite filter element Recommended only with system operating pressures below 300 bar.	---
4	6020RS omni coulometric cell	---
5	Outlet of 6020RS omni coulometric cell to inlet of autosampler, such as a WPS-3000TBRS autosampler	6041.5821
6	Outlet of autosampler to inlet of analytical column	6041.5814
7	Analytical column	---
8	Outlet of analytical column to inlet of analytical cell (6011RS or 6041RS ultra)	6041.5811 or 6041.5812
9, 10	In-line filter with PEEK filter element Recommended only with system operating pressures below 300 bar.	---
11	Analytical electrochemical cell	---
	For connecting multiple coulometric cells in series Interconnecting Viper capillary, included in detector accessory kit	6041.9075
12	Electrochemical cell outlet to waste Cell waste line included in detector accessories kit, fitting for cell connection included in cell accessory kit	6040.4900

For details on the capillaries, fittings and lock nuts, see section 9.2 (→ page 126).

4.4.3 Installing In-Line Filters

⚠ **Important:** Use in-line filters only with system operating pressures below 300 bar. For applications with system operating pressures above 300 bar, remove the in-line filters from the system flow path.

⚠ **Important :** Utilisez des filtres en ligne uniquement avec des pressions inférieures à 300 bar. Pour des appplications avec des pressions supérieures à 300 bar, retirez les filtres en ligne du tracjectoire d'écoulement du système.

The in-line filters can be used to ensure that particulate matter does not clog the cell, resulting in high backpressure and lowered system performance.

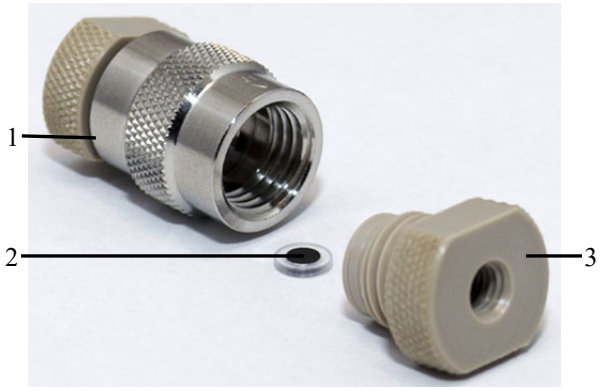
The in-line-filter elements should be replaced on a regular basis. For information about the replacement procedure, refer to section 7.4 (→ page 116).

4.4.3.1 Connecting the Graphite In-Line Filter

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

Parts required

In-line filter kit with graphite filter element, including 5 graphite filter elements
The kit is included in the accessories kit for the detector.



No.	Description
1	Filter holder
2	Graphite filter element
3	Lock nut

Fig. 16: In-line filter with graphite filter element

Follow these steps

To install the in-line filter, perform the following steps:

1. Locate the in-line filter kit with graphite filter elements.
2. Open the filter holder by turning the lock nuts counterclockwise with your fingers.
3. Install one lock nut at one end by turning the lock nut clockwise with your fingers.
   Do not use a wrench or other tools. Do not overtighten the lock nuts, because this can break the filter element.
4. Place the filter element inside the filter holder. Ensure that the element is properly centered and seated against the surface of the end nut.
5. Close the filter holder with the second lock nut by turning the lock nut clockwise with your fingers carefully until contact between the cap and the filter is felt.
   Mind the following:
   - The filter is properly installed if both end nuts are approximately an equal distance from the center of the filter holder.
   - The lock nuts should be closed finger-tight.
   - Do not use a wrench or other tools.
   - Do not overtighten the lock nuts, because this can break the filter element.
6. Connect a capillary from the pump outlet to the inlet of the in-line filter. Install the in-line filter in the direction of flow as stated on the filter holder.

Tip: Make sure that the capillary is connected correctly, with the filter holder being installed in the direction of the orientation sign on the filter. The orientation sign points downstream.

7. Connect a capillary to the outlet of the in-line filter and route it to waste.
8. Flush the filter with mobile phase for 1-2 minutes at a flow rate of 1.0 mL/min.
9. Stop the pump flow.
10. Continue with the flow connections as described in section 4.4.2, page 54.

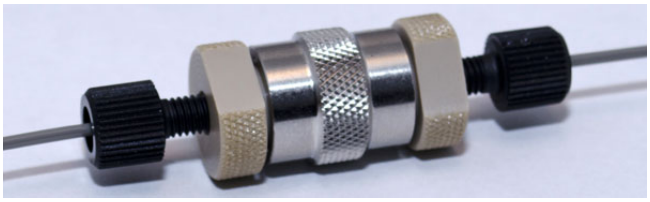


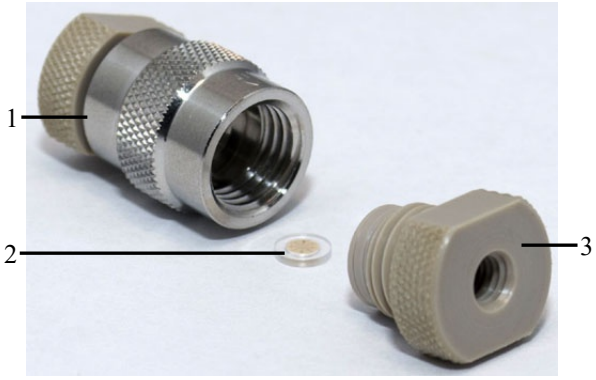
Fig. 17: In-line filter installed

4.4.3.2 Connecting the PEEK In-Line Filter

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

Parts required

In-line filter kit with PEEK filter element, including 5 PEEK filter elements
The kit is included in the accessories kit for the detector.



No.	Description
1	Filter holder
2	PEEK filter element
3	Lock nut

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

Follow these steps

1. Locate the in-line filter kit with PEEK filter elements.
2. Open the filter holder by turning the lock nuts counterclockwise with your fingers.
3. Place the filter element inside the filter holder. Ensure that the element is properly centered and seated against the surface of the end nut.

4. Close the filter holder with the second lock nut by turning the lock nut clockwise with your fingers carefully until contact between the cap and the filter is felt.

   Mind the following:

   - The filter is properly installed if both end nuts are approximately an equal distance from the center of the filter holder.
   - The lock nuts should be closed finger-tight.
   - Do not use a wrench or other tools.
   - Do not overtighten the lock nuts, because this can break the filter element.

5. Connect capillaries to the inlet and outlet of the in-line filter.
6. Connect a capillary to the outlet of the analytical column and to the inlet of the in-line filter.
7. Connect a capillary from the outlet of the in-line filter to the inlet of the analytical electrochemical cell.

4.4.4 Connecting a Column to the Column Compartment

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

Fig. 19: View into the column compartment

1. Remove the panel from the column compartment.
2. Make sure that the capillaries and components before the column are connected as described in section 4.4.2 (→ page 54).
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 74).
5. Before connecting the analytical electrochemical cell to the system, flush the column with at least 200 mL mobile phase for several hours. Solvents that may be contained in the column should be flushed out with the mobile phase before initial use.
6. Connect the capillaries and components after the column compartment as described in section 4.4.2 (→ page 54).

4.5 Equilibration

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 (→ see below).

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 72).

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

Equilibrating the Electrochemical Cell

Before you start any analysis, allow the cell to equilibrate, for the background current to stabilize. The baseline should be reasonably stable. The amount of time necessary to stabilize the cell depends on the application.

Several factors determine how long the cell needs to equilibrate, such as the nature and purity of the mobile phase, potential applied to the cell and especially the level of sensitivity needed for the analysis. The equilibration can take as little as few minutes for non-sensitive analyses to periods of hours for very sensitive analyses.

Follow the instructions in the *User Guide* for the respective electrochemical cell.

4.6 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 (→ page 82).

4.6.1 Mobile Phases

Mobile phase quality significantly affects detection limits and detector 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:

General Mobile Phase 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 18.2 MΩ 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 carbon (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 electro-active 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 66.

- 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.
- Use reagents of the highest purity to prepare the mobile phase. In many cases, impurities such as heavy metals (especially redox-active transition metals such as iron) can influence the background current at the electrochemical cell. This creates noise that compromises the detection limit. Consider two criteria: the overall purity and the level of heavy metal impurities. In addition, trace metal ions can sometimes interact with the electroactive species before the species reach the electrochemical cell.

Guidelines for Mobile Phase Use with Electrochemical Cells

- 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 EC-compatible mobile phase flow is established before the cell is turned on and that flow remains turned on whenever potential is applied to avoid permanent damage to the cell.
- Do not use nitric acid (concentrated HNO_3) to clean an electrochemical cell. Exposure may damage susceptible internal components. Refer to the *User Guide* for your respective electrochemical cell for recommended cleaning procedures.
- Make sure that you operate the cell within the specifications for backpressure and applied potential. Observe the specifications and the mobile phase guidelines for the electrochemical cells.
- 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).

4.6.2 Solvent 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 electrochemical detection for HPLC (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 131.
- 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 131).
- 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. Observe the notes and precautions on the maximum operating pressure for the in-line filters (→ page 59).

5 Operation and Maintenance

For information about how to control the detector via the Chromeleon Chromatography Management System, see section 5.3 (→ page 69).

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. Depending on the number of potentiostat modules installed, the self-test may take as long as 6-7 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 orange 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 101), 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 your local Thermo Fisher Scientific support center.
- 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

The front panel display shows the following information on the status screen:

- Signal unit (measured sensitivity)
- Applied potential settings (mV)
- Mode of operation (DC or pulse)
- Cells on/off
- Column compartment temperature

Status Screen with DC Mode Potentiostat Module

Upon successful completion of the power-up self-test for a detector with a DC potentiostat module installed, the initial screen changes to the following status screen.

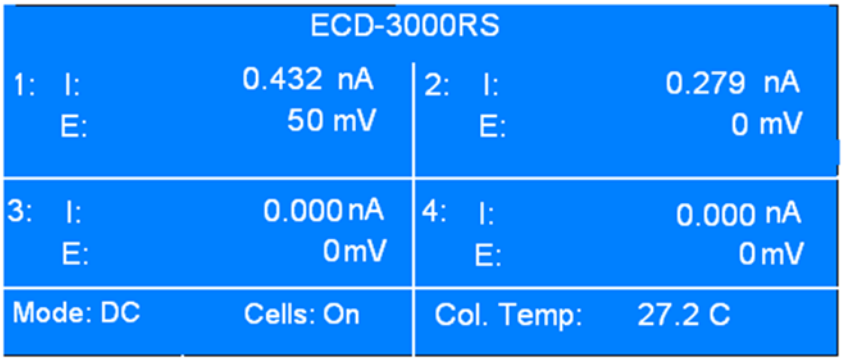


Fig. 20: Status screen for DC Mode (example)

Status Screen with Pulse Mode Potentiostat Module

Upon successful completion of the power up self-test for a detector with Pulse potentiostat module installed, the initial screen changes to the following status screen.

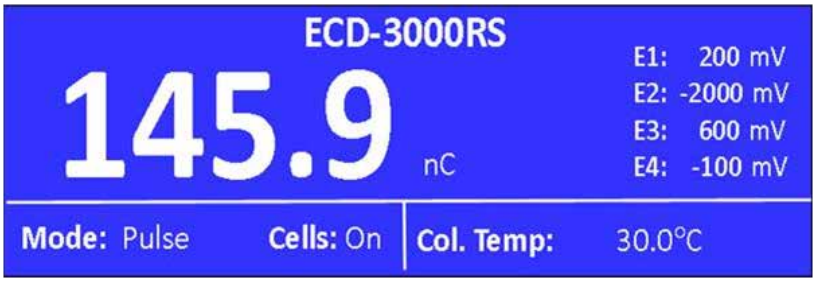


Fig. 21: Status screen for Pulse Mode (example)

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 11).
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 3.4 (→ 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 69).

Two modes of software control are available:

- *Direct control* with the parameters and commands from the **Commands** (F8) dialog box (→ page 70) or from a control panel (→ page 71).
- *Automated control* with a control program (PGM) (→ page 72).

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 70. For details about how to do this on a control panel, see page 71.

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 96).

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.

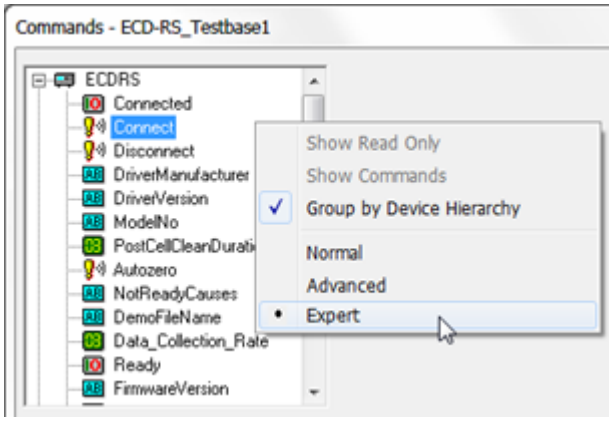


Fig. 22: Commands dialog box

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 modules 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**.

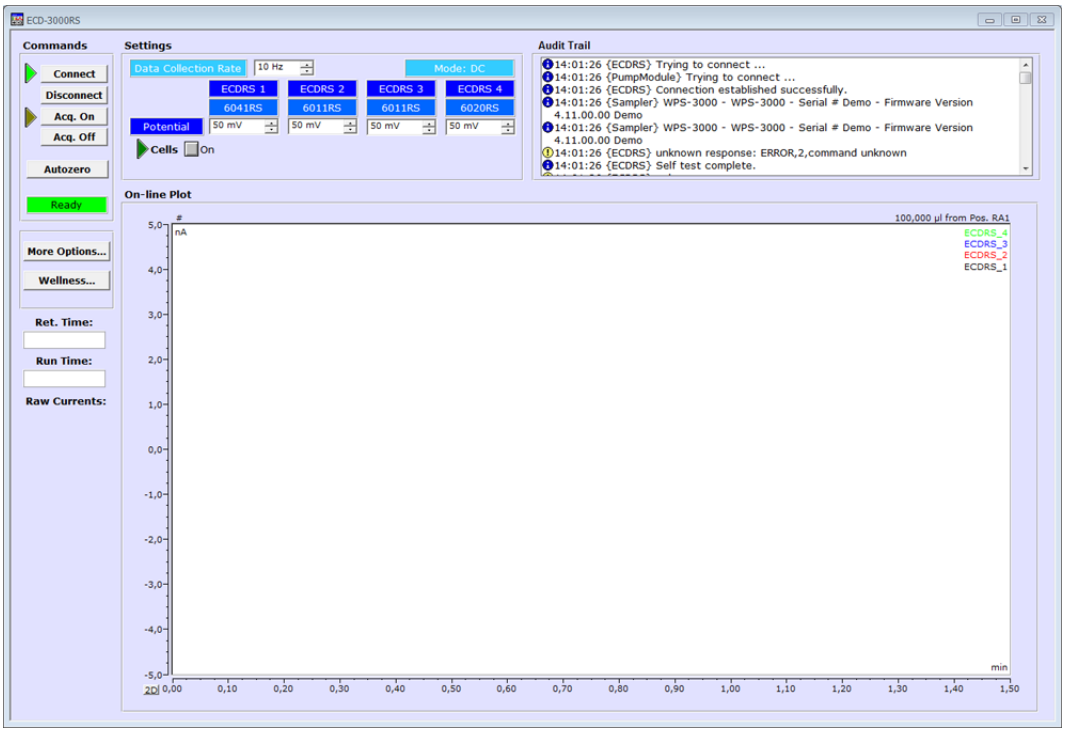


Fig. 23: Detector control panel (example, here: 4-channel operation)

The control panel provides access to the operating parameters and commands required for routine operation of the detector. Depending upon the mode of operation selected when setting up the detector in Chromeleon, the detector control panel may differ from the control panel shown in Fig. 23.

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 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.

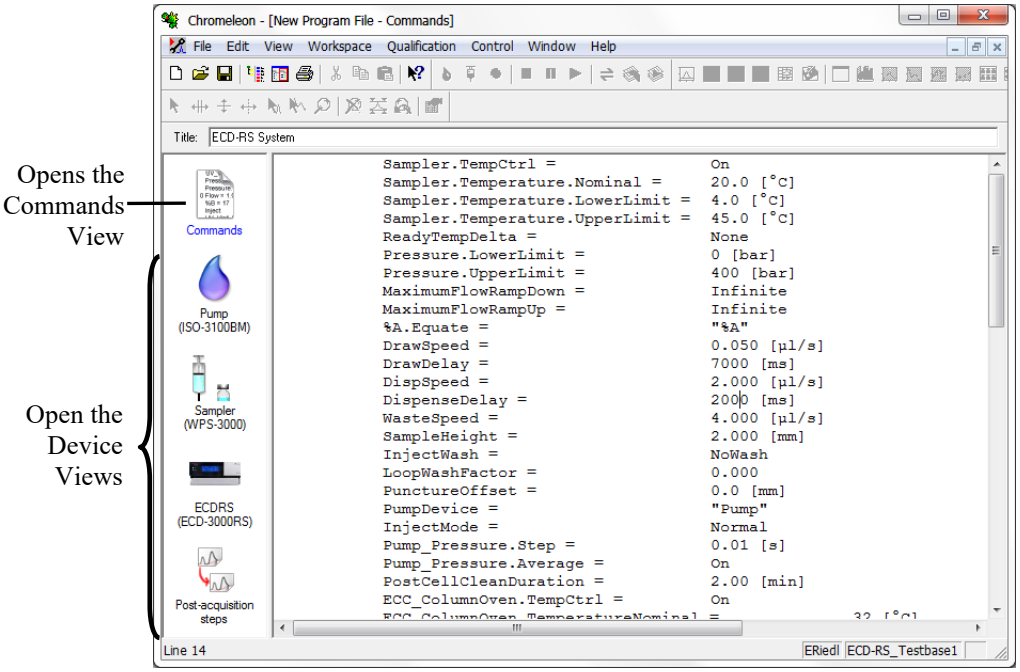


Fig. 24: Chromeleon program file (here program shown in the Commands view)

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. 24). 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 Important Operating Settings

The table below lists the most important operating settings for routine operation of the detector. You can usually access these parameters from the Chromeleon user interface.

Setting		Description
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). Observe the following: • 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. • To obtain the optimal potential, generate a hydrodynamic voltammogram. For information on optimizing the potential, see section 5.8.1 (→ page 85).
Autozero		Perform an autozero to recalibrate the zero current. When performing an autozero, 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 does not affect the baseline current that is shown on the detector display. Observe the following: • Typically, the autozero should be 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. • *When operating the detector in DC Mode* For optimum performance of the detector in DC Mode, perform the autozero with the potential to the cells being turned off on a weekly basis.
Column thermostatting		Enabling and disabling column thermostatting and setting the desired temperature.
	TempCtrl	Enables and disables the temperature control for the column compartment. • To enable temperature control, navigate in the Chromeleon **Commands** (F8) dialog box to **ColumnOven > TempCtrl** and set to **On**. • To disable the thermostatting, set **TempCtrl** to **Off**.
	TemperatureNominal	Set the desired temperature for the column thermostatting. If the temperature control is disabled when you enter a temperature value under **TemperatureNominal**, the **TempCtrl** is automatically set to **On**.

Setting	Description
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. Set the data collection rate, at which data is to be collected from the detector.
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. For guidelines on selecting a filter constant, see section 5.8.2 (→ page 85). 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.
Leak detection	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 118).
Operating modes	The detector provides different operating modes that require different potentiostat modules and operating settings. **Note:** When selecting the operational mode, note that detector can only be operated in DC Mode or in Pulse Mode at the same time.
Direct Current (DC) Mode	*Requires a potentiostat module for DC Mode* In direct current (DC) mode, the current is measured as the potential across the working electrode is held constant. For further information on the DC Mode and how to operate the detector in DC Mode, see section 5.5 Direct Current (DC) Mode, page 76.
Pulse Mode	*Requires a potentiostat module for Pulse Mode and an amperometric cell with noble-metal-based working electrode* Applying the technique of the Pulse Mode to liquid chromatography with electrochemical detection is called *pulsed amperometric detection* (PAD). Pulses of high positive and high negative potentials are applied to the electrode. A pre-programmed sequence of potentials set for specific time periods is referred to as a waveform. **Note:** In Pulse Mode, only one pulse potentiostat module can be operated at any time. For further information on the Pulse Mode and how to operate the detector in Pulse Mode, see section 5.6 Pulse Mode, page 79.

5.5 Direct Current (DC) Mode

Requires a potentiostat module for DC Mode

- Operating principle of the DC Mode → see section 5.5.1 below
- Setting the potential in DC Mode → see section 5.5.2, below
- Allow research potentials → see section 5.5.3, page 78

5.5.1 Operating Principle of the DC Mode

In direct current (DC) mode, the current is measured as the potential across the working electrode is held constant.

The electrode surface remains constant during the analysis. Typically, the oxidation (reduction) product(s) that is formed during the electrochemical detection process remains in solution and the condition of the electrode remains unchanged during the analysis.

In some cases (e.g., the analysis of alcohols and carbohydrates), oxidation of the compounds of interest produces species that may foul the electrode and cause a decrease in the current as the electrochemical reaction proceeds. This may result in the loss of analyte signal.

To overcome this effect, use the Pulse Mode (→ see section 5.6, page 79).

5.5.2 Setting the Potential in DC Mode

⚠ **Important:** 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 EC-compatible mobile phase flow is established before the cell is turned on and that flow remains turned on whenever potential is applied to avoid permanent damage to the cell.

⚠ **Important :** Un débit doit être appliqué à la phase mobile dès qu'un potentiel est appliqué sur la cellule. N'utilisez jamais la cellule électrochimique à sec lorsqu'un potentiel est appliqué sur les électrodes, ceci pourrait endommager les électrodes. Mettez en route un débit de phase mobile compatible avec les cellule electrochimiques avant d'allumer la cellule et assurez-vous que lé débit reste en route lorsqu'un potentiel est appliqué afin d'éviter tout dommage irréversible sur la cellule.

Preparations

1. Make sure that the following requirements are met:
   - The required potentiostat modules for DC Mode are installed and the desired electrochemical cells are connected. For an overview of configurations, see section 4.3.1 Configurations of Electrochemical Cell and Potentiostat Module, page 47.
   - The detector is prepared for operation. For preparations for operation, see section 4.1, page 45.
2. Verify that the DC Mode and required range is selected on the **Detector** page in the configuration of the detector (→ section 3.4.3, page 37).
3. Observe the guidelines for the operation of electrochemical cells (→ *User Guide* for the respective electrochemical cell).
4. Check and observe the potential ranges for the electrochemical cells: The potential ranges for the cells can be obtained from the **Potential** property of each cell in the **Commands** (F8) dialog box in Chromeleon.

Follow these steps

1. Locate the potential setting on the tabset panel for the detector or in the Chromeleon **Commands** (F8) dialog box.
2. Set the potential:
   - On the tabset panel, enter the desired potential.
   - In the **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.

   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. 25 below shows a typical background current after equilibration with a 6041RS ultra amperometric cell with glassy carbon working electrode, using the mobile phase MD-TM (commercially available from Thermo Fisher Scientific) at a flow rate of 0.5 mL/min and an applied potential of 300 mV.

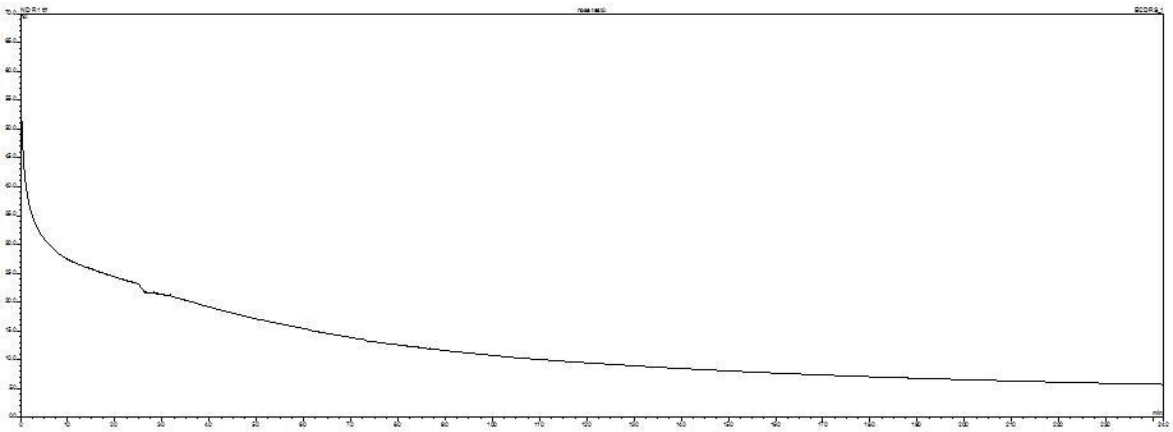


Fig. 25: Typical background current after equilibration with a 6041RS cell with GC electrode

3. 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.

5.5.3 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.

⚠ **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 applying 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.

5.6 Pulse Mode

Requires a potentiostat module for Pulse Mode and an amperometric cell with noble-metal-based working electrode

- Operating Principle of the Pulse Mode → see section 5.6.1 below
- Selecting Parameters in Pulse Mode → see section 5.6.2, page 81

⚠ **Important:** To avoid irreversible damage to the electrode, use only noble metal-based working electrodes, such as gold, with an amperometric cell for the Pulse Mode. Do not use a carbon-based or porous-graphite working electrode for pulse mode.

⚠ **Important :** Afin d'éviter des dommages irréversibles à l'électrode, utilisez uniquement des électrodes de travail en métaux nobles, comme l'or, ainsi qu'une cellule ampérométrique pour le mode d'impulsion (Pulse Mode). Ne pas utiliser une électrode de travail en carbone ou en graphite poreux pour le mode d'impulsion.

5.6.1 Operating Principle of the Pulsed Amperometric Detection (PAD)

Pulsed Amperometric Detection – Definition

Applying the technique of the Pulse Mode to liquid chromatography with electrochemical detection is called *pulsed amperometric detection* (PAD). Pulses of high positive and high negative potentials are applied to the electrode. A pre-programmed sequence of potentials set for specific time periods is referred to as a waveform.

In Pulse Mode, the potential applied to the working electrode is periodically changed during the analysis. These periodic changes in potential electrochemically "clean" or reactivate the electrode surface to prepare it for the next measurement.

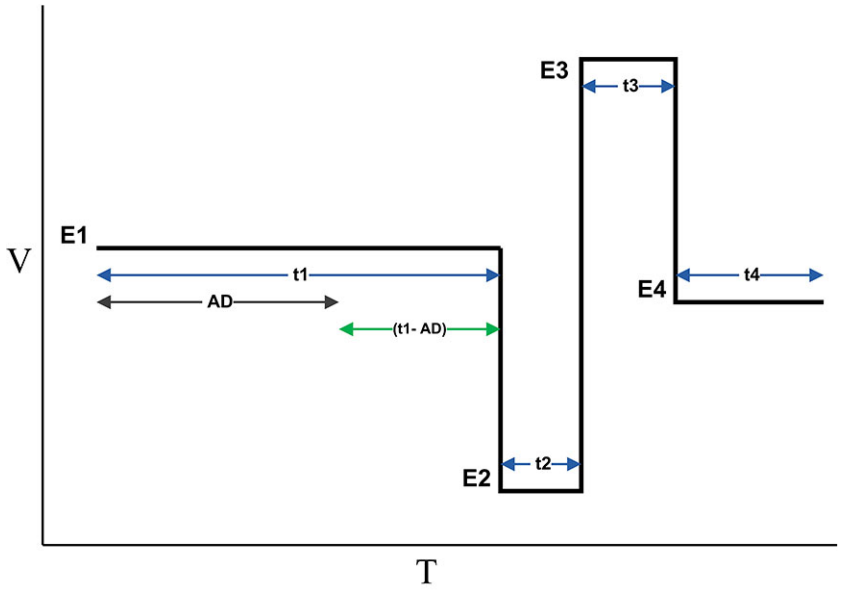
🛈 **Tip:** This operating principle considers the situation where the performance of the electrode decreases rapidly during the electrochemical reaction. Over a period of time, all electrodes will eventually become compromised due to contamination from trace components in the sample and/or the mobile phase and over time may need to be cleaned.

Four-Pulse Waveform

Fig. 26 below shows an example of a waveform diagram with four pulses ("four-pulse waveform").

This diagram shows the time (t) for which each operating potential (E) will be applied with a defined period for data acquisition (AD). This waveform is typically used for the analysis of carbohydrates using a gold working electrode.

The waveform is quickly repeated during the analytical run. The detector reads the current signal level during each waveform and sends it to a data system for collection and integration.



	Description
AD	Acquisition delay
E	Operating potential pulse (E1-E4)
t	Time (t1-t4) for which potential is applied
T	Time (duration) of the analysis
V	Potential applied

Fig. 26: Schematic of a four-pulse waveform used in the Pulse Mode

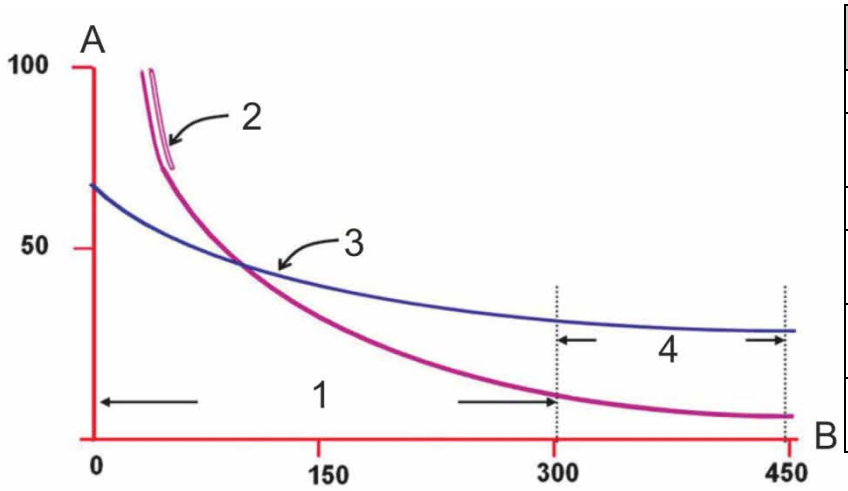
This typical four-pulse waveform includes the following steps:

1. An operating potential pulse (E1) is applied to the electrode for a time period (t1). The current produced from oxidation (or reduction) of the compound of interest is measured.
2. A second potential pulse (E2) is applied at a high negative potential for a time period (t2) to reduce (oxidize) the electrode surface.
3. A third potential pulse (E3) is applied at a high positive potential for a time period (t3) to re-oxidize (reduce) the electrode surface.
4. A fourth potential pulse (E4) is applied at an intermediate level for a time period (t4) to condition the electrode before restarting the next pulse cycle.

Acquisition Delay

The sensitivity of an assay using Pulse Mode depends on the period of time in which the detector is actually measuring the current level during a pulse cycle.

The acquisition delay is a subset of the measuring period. The acquisition delay is used to minimize the effect of the capacitive currents that result when the analytical potential that was first applied has dissipated. The observed current signal of the measuring period is then predominantly from the electrochemical process of the analyte that is occurring (→ Fig. 27 below).



No.	Description
A	Current response
B	Time in milliseconds
1	Acquisition delay
2	Charging current from electrode
3	Faradaic current from analyte
4	Current measurement

Fig. 27: Decay of electrode current after charging in the presence and absence of the analyte

5.6.2 Selecting Parameters in the Pulse Mode

Tip: In Pulse Mode, only *one* pulse potentiostat module can be operated at any time.

Preparations

1. Make sure that the following requirements are met:
   - A potentiostat module for Pulse Mode is installed and an amperometric cell with a noble-metal based working electrode, such as gold or platinum, is connected. For an overview of configurations, see section 4.3.1 Configurations of Electrochemical Cell and Potentiostat Module, page 47.
   - The detector is prepared for operation. For preparations for operation, see section 4.1, page 45.
2. Verify that the Pulse Mode and required range is selected on the **Detector** page in the configuration of the detector (→ section 3.4.3, page 37).
3. Observe the guidelines for operating the electrochemical cell (→ *User Guide* for the electrochemical cell).

4. Check and observe the potential ranges for the electrochemical cell: The potential ranges can be obtained from the **Potential** properties (E1 to E4) in the **Commands** (F8) dialog box in Chromeleon.

Follow these steps

Selecting the optimum conditions for the Pulse Mode involves setting a number of parameters:

- Set the three or four potentials
- Set the pulse width (time) for which each potential will be applied
- Define the data acquisition delay

Guidelines

To select these parameters, observe the following guidelines:

- **Analytical potential (E1)**
  Set the analytical potential (E1) at the optimum potential for the compound of interest. (Literature values for a starting point and further optimized by performing an hydrodynamic voltammogram.)
- **Conditioning potentials (E2 and E3)**
  The conditioning potentials (E2 and E3) are usually set near the upper and lower limits for the mobile phase (i.e., the potentials at which the mobile phase begins to undergo an oxidative (reductive) reaction). These limits can be obtained by using the literature or by using traditional voltammetric methods.
- Keep the time period for the potentials E2, E3 and E4 (pulse width) as short as possible to minimize the data collection period while maintaining appropriate reproducibility.
- **Acquisition delay**
  - The selection of an appropriate data acquisition delay period is a compromise between two competing processes.
  - The acquisition delay parameter may be varied to change the time at which data collection begins. Although it is possible that increasing this collection period may result in an increase in overall sensitivity, other factors come into play.

    If, for example, the acquisition delay were decreased thereby increasing the period of time that data is collected, the background current may increase too. This would cause an increase in baseline noise which would effectively reduce the sensitivity.

5.7 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 76).
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).

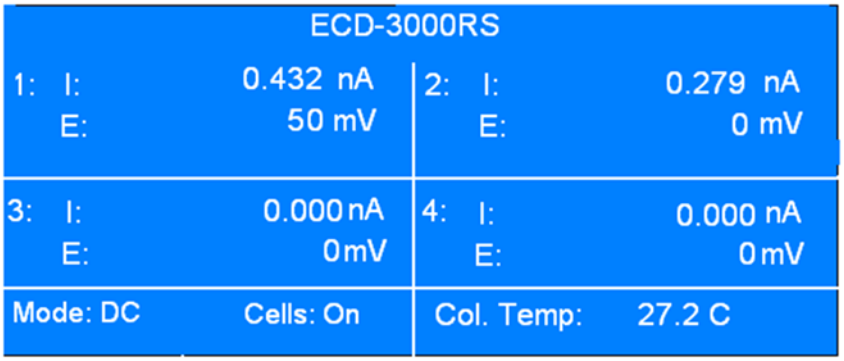


Fig. 28: Data acquisition screen for DC Mode (example)

5.8 Optimizing the 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	See page…
Analytical Potential	Selectivity, baseline noise, sensitivity	see below
Filter Constant	Sensitivity, baseline noise	85
Background Current	Sensitivity, reproducibility, baseline noise	85
Data Collection Rate	Peak resolution, disk space, possibly baseline noise	87
Selectivity	Peak resolution, response	88
Screening Operation	Selectivity, baseline noise	88
Redox Operation	Selectivity, sensitivity	93

In Chromeleon, you can set operating parameters in the **Commands** (F8) dialog box for the detector (→ page 70) and on the page for the detector of the **Panel Tabset** (→ page 71). Note that you may need to change the Display filter level to see all operating parameters (→ section 5.3.2, page 70).

Using Coulometric Cells for Optimization

The 6020RS omni and 6011RS ultra coulometric cells can be used to optimize the detector performance in multiple ways, such as for lower background currents, stable baselines and better selectivity, when used with coulometric and amperometric detection.

For details about the 6020RS omni coulometric cell and the 6011RS ultra coulometric analytical cell, refer to the *User Guide* for coulometric cells.

For information about screening and redox operation, see the pages stated above.

5.8.1 Analytical Potential

Guidelines for an Optimum Potential

Consider the following factors for an optimum potential:

- The best applied potential is typically obtained by generating a hydrodynamic voltammogram (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.

Hydrodynamic Voltammogram

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.

Construct a hydrodynamic voltammogram prior to use of each new electrochemical cell (or working electrode material) to determine its optimum potential for the desired application. Refer to the procedures in the *User Guide* of the respective cell.

5.8.2 Filter Constant

When selecting a filter constant, note the following guidelines:

- 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.

5.8.3 Baseline Optimization

To obtain a stable and quiet baseline, observe the following rules:

- When you perform data acquisition or use the system for analyes, do not recycle the mobile phase.
- Verify that the end of the waste tubing is submerged in the waste container. Do not let the waste line drip. Dripping can cause disurbances in the baseline.
- Protect cell, column and other flow path components from sudden thermal changes and electrical interferences.
- Use clean and properly prepared mobile phase. Monitor the data periodically to ensure that the mobile phase and system are not contaminated.

In addition, background current significantly contributes to baseline noise. For details on the background current and how to minimize it, see section 5.8.4 (→ page 85).

5.8.4 Background Current and Baseline Noise

Baseline noise is a function of the background current. Background current can be either faradaic (obeying Faraday's Law) or non-faradaic. Electrolysis of contaminants in the mobile phase is a typical source of faradaic current. Non-faradaic 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.

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

Optimization Factors

Baseline noise and background current depend on a number of factors that can be optimized. Observe the following guidelines to minimize background current:

Factor	Description
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.
	Use a dedicated column for each specific analysis to avoid cross-contamination of the column.
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.
	If the analytical potential setting is near the plateau of the HDV curve, decrease the electrochemical potential slightly.
	The background current 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.
	Allow sufficient time to achieve a stable baseline before performing an analysis.
Cleanliness of mobile phase reservoirs	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.
Degassing	Not degassing the mobile phase sufficiently becomes a problem at higher oxidation and reduction potentials.
	When operating at extremely high or negative potentials, take care to effectively degas the mobile phase.

Factor	Description
Metal fluidic surfaces in 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 131).
	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.
Microbes	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.
Mobile phase quality	The quality of water and the organic modifier (for example, methanol, acetonitrile) used in the mobile phase. 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.
	Minimize the impurities in the mobile phase. Observe the mobile phase guidelines in section 4.6.1, page 64.
	Using a 6020RS omni coulometric cell can decrease interference from electroactive impurities (→ section 5.9, page 88).
Mobile phase 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.
	Limit the recycling of mobile phase, especially when working with impure samples at high concentrations.

Tip: For optimum operation, use a biocompatible pump, such as the ISO-3000BM pump of the UltiMate 3000 series.

5.8.5 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 or later).
- 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 lower 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 low, 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 higher 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.

5.8.6 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 co-eluted, use a 6011RS ultra coulometric analytical 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.

Selectivity can be improved using the 6020RS omni coulometric cell (→ *User Guide for Coulometric Cells*).

5.9 Screening Operation

In screening operation, electrochemical screening is used to eliminate or screen out possible electrochemical interferences before the analytical cell. To do so, the 6020RS omni coulometric cell or the 6011RS ultra coulometric analytical cell is used as screening cell.

Ideally, in electrochemical detection, the chromatographic separation should provide a single electroactive compound to the detector at a given instant. The mobile phase should not contain any other electroactive species. The applied potential should be set to the limiting potential for the peak of interest, and a simple, direct measurement should be obtained.

However, the chromatographic separation of the sample may be quite difficult sometimes, where it is not certain that the separation presents only a single electroactive compound to the detector at a given instant. When this occurs, an interfering substance acts to increase the signal that was measured for the compound of interest, which in turn leads to an erroneous result.

At the same time, the mobile phase may contain trace levels of electroactive contaminants, which increase the background current and thus reduce the limit of detection of the assay.

The size of these concerns is related to the potential that is needed to analyze the compound of interest. If a large potential is required, interferences may become significant.

The following screening operations are available:

- Mobile phase screening using the 6020RS omni coulometric cell (→ section 5.9.1, page 89)
- Analyte screening for interfering or co-eluting analytes using the 6020RS omni or 6011RS ultra coulometric cell (→ section 5.9.2, page 90)
- Screening operation parallel to analytical operation with the 6011RS ultra coulometric cell (→ section 5.9.3, page 92)

Tip: For screening operation, DC Mode operation is required.

5.9.1 Screening the Mobile Phase with the 6020RS omni Coulometric Cell

When

The 6020RS omni coulometric cell can be used to clean and eliminate possible electrochemical interferences from the mobile phase of the system.

Parts required

- 6020RS omni coulometric cell, and analytical electrochemical cell
- 2 potentiostat modules for DC Mode
- *Depending on the operating pressure*
  Capillaries from the tubing and fitting kit or the nanoViper capillary kit

Follow these steps

To screen the mobile phase with the 6020RS omni coulometric cell, the following configuration is recommended:

1. Install at least two separate potentiostat modules for DC Mode (→ *Installation Instructions for Potentiostat Modules*). Choose the appropriate cell bay location for each potentiostat module, based on the application.

   Tip: With the recommended configuration, the 6020RS omni coulometric cell is installed in bay D.

2. Install the 6020RS omni coulometric cell and the appropriate analytical cell (→ *User Guide* for the respective electrochemical cell) and set up the flow connections (→ section 4.4, page 52).

   Observe the following:

   - The 6020RS omni coulometric cell is installed in the flow path after the pump and upstream of the autosampler. In this position, the cell electrochemically cleans and conditions the mobile phase before the mobile phase is mixed with the injected sample and enters into the analytical column.
   - The 6020RS omni coulometric cell is designed to withstand pressures up to 620 bar (9000 psi, 62 MPa).
   - For the recommended configuration with a 6020RS omni cell installed in bay D of the detector, see section 4.4.2 (→ page 54).
   - The in-line filters can be installed as required. Observe the notes and precautions for the maximum operating pressure for the in-line filters in section 4.4.3 (→ page 59).

3. Set the potential for the 6020RS omni coulometric cell.

   The potential of the 6020RS omni coulometric cell is typically set above the potential that is applied to the first working electrode of the 6011RS ultra coulometric analytical cell.

Tip: If you connect the 6020RS omni cell in the flow path before the autosampler, select the potential for the cell so that it is 25–50 mV higher than the applied potential of the first analytical electrode that is used for the application.

5.9.2 Screening Interfering or Co-Eluting Analytes

When

To eliminate most electroactive interferences if its potential corresponds to the limiting current wave of the interfering compounds.

Parts required

- 6020RS omni coulometric cell, and analytical electrochemical cell

> **Tip:** Alternatively, you can use a 6011RS ultra coulometric analytical cell as screening electrode. With the 6011RS ultra cell, it is recommended to utilize the first channel as an additional screening channel. For information, see section 5.9.3 (→ page 92).

- 2 potentiostat modules for DC Mode
- *Depending on the operating pressure*
  Capillaries from the tubing and fitting kit or the nanoViper capillary kit

Follow these steps

To screen interfering or co-eluting analytes with the 6020RS omni coulometric cell, the following configuration is recommended:

1. Install at least two separate potentiostat modules for DC Mode (→ *Installation Instructions for Potentiostat Modules*). Choose the appropriate cell bay location for each potentiostat module, based on the application.

   > **Tip:** With the recommended configuration, the 6020RS omni coulometric cell is installed in bay D.

2. Install the 6020RS omni coulometric cell and the appropriate analytical cell (→ *User Guide* for the respective electrochemical cell) and set up the flow connections (→ section 4.4, page 52).

   Observe the following:

   - The 6020RS omni coulometric cell is installed in the flow path after the autosampler. In this position, the cell eliminates most electroactive interferences if its potential corresponds to the limiting current wave of the interfering compounds.
   - The 6020RS omni coulometric cell is designed to withstand pressures up to 620 bar (9000 psi, 62 MPa).
   - For the recommended configuration with a 6020RS omni coulometric cell installed in bay D of the detector, see section 4.4.2 (→ page 54).
   - If the interferences are due to impurities in the injected sample, which oxidize (reduce) at a higher (lower) potential than the compound or compounds of interest, use an additional 6020RS omni coulometric cell prior to the analytical cell.

     In this case, install an additional 6020RS omni coulometric cell immediately before the analytical cell. The exact bay position of the additional 6020RS omni coulometric cell and the analytical cell depends on the column length.
   - The in-line filters can be installed as required. Observe the notes and precautions for the maximum operating pressure for the in-line filters in section 4.4.3 (→ page 59).

3. Set the potential for the 6020RS omni cell so that interfering or co-eluting analytes from the sample are oxidized (or reduced) while the analyte(s) of interest is not affected.

The potential of the screening electrode should be set to the highest potential at which no oxidation (reduction) of the analyte(s) of interest occurs. As a result, all solutes in the sample stream (including the mobile phase) with formal potentials less than that of the analyte of interest are also electrolyzed.

Tip: If you connect the 6020RS omni coulometric cell in the flow path before the autosampler, select the potential for the cell so that it is 25–50 mV higher than the applied potential of the first analytical electrode that is used for the application.

5.9.3 Screening with a 6011RS ultra Coulometric Analytical Cell

When

With the 6011RS ultra coulometric analytical cell, the following options are available to screen out interferences:

- The first electrode in the dual-electrode 6011RS ultra cell can be used as the screening electrode, while the second electrode can be used as the analytical electrode.
- As an alternative, the first electrode can act as an analytical electrode at one potential. The second electrode can also act as an analytical electrode at a higher potential to measure a different analyte. In this case, the first electrode acts as both screening electrode and analytical electrode.

Parts required

- 6011RS ultra coulometric analytical cell
- 1 potentiostat module for DC Mode
- *Depending on the operating pressure*
  Capillaries from the tubing and fitting kit or the nanoViper capillary kit

Follow these steps

To screen interferences with the 6011RS ultra coulometric analytical cell, the following configuration is required:

1. Install the potentiostat module for DC Mode (→ *Installation Instructions for Potentiostat Modules*). Choose the appropriate cell bay location for the potentiostat module, based on the application.

   Tip: With the recommended configuration, the 6011RS ultra cell is installed in bay C.

2. Install the 6011RS ultra coulometric cell and the appropriate analytical cell (→ *User Guide* for coulometric cells) and set up the flow connections (→ section 4.4, page 52).

   Observe the following:

- ♦ For the recommended configuration with a 6011RS ultra coulometric analytical cell installed in bay C of the detector, see section 4.4.2 (→ page 54).
- ♦ The in-line filters can be installed as required. Observe the notes and precautions for the maximum operating pressure for the in-line filters in section 4.4.3 (→ page 59).

3. Set the screening and analytical potentials for the electrodes in the 6011RS ultra coulometric analytical cell.

   The potential of the first working electrode of the 6011RS ultra cell is typically set about 50 mV higher than the potential that is applied to the second analytical electrode.

5.10 Reduction/Oxidation (Redox) Operation

5.10.1 Performing a Redox Operation

When

If the analyte can undergo both an oxidation and a reduction (redox) process, such as some biogenic amines, the Redox operation can be used to obtain very selective analyses.

Parts required

Redox operation requires two independently controlled electrodes in series. The first electrode in the flow path functions as an electrochemical derivatizing agent to change the analyte into a more easily detected form that is sensed by the second electrode.

- 6020RS omni coulometric cell and the 6011RS ultra coulometric analytical cell
- 2 potentiostat modules for DC Mode
- *Depending on the operating pressure*
  Capillaries from the tubing and fitting kit or the nanoViper capillary kit

Configuration example

The following example of a Redox operation is configured as follows:

- The first electrode is used to oxidize the analyte.
- The second electrode is used so that oxidation product is reduced (or vice versa).
- The 6020RS omni coulometric cell can be used to facilitate Redox operation with the detector: Place the 6020RS omni cell in-line between the output of the analytical column and the input to the analytical cell that is used for quantitation.

Follow these steps (based on the configuration example)

For Redox operation, the following configuration is required:

1. Install at least two separate potentiostat modules for DC Mode (→ *Installation Instructions for Potentiostat Modules*). Choose the appropriate cell bay location for each potentiostat module, based on the application.

2. Install the 6020RS omni coulometric cell and the 6011RS ultra coulometric analytical cell (→ *User Guide* for the respective electrochemical cell) and set up the flow connections (→ section 4.4, page 52). Observe the following:
   - ♦ The 6020RS omni coulometric cell is designed to withstand pressures up to 620 bar (9000 psi, 62 MPa).
   - ♦ The in-line filters can be installed as required. Observe the notes and precautions for the maximum operating pressure for the in-line filters in section 4.4.3 (→ page 59).
3. Set the screening and analytical potentials for the electrodes in the two cells. See the Potential Requirements for Redox Operation in section 5.10.2, page 94.

5.10.2 Potential Requirements for Redox Operation

To use the Redox operation successfully, observe the following requirements when setting potentials:

- The initial reaction must
  - ♦ be reversible, with a high yield (ideally, the yield should be 99.9% or better).
  - ♦ be tolerant of small variations in chromatographic conditions and sample composition.
- The applied potential for the initial oxidative or reductive process must correspond to the limiting current.
- The electrolysis product
  - ♦ should have a sufficiently long lifetime so that it can travel between the two cells without degradation.
  - ♦ must be soluble in the mobile phase.
- The analytes must initially undergo a redox reaction at a consistent oxidation state.

Tip: An additional benefit of the redox mode is the possible enhancement of sensitivity.

Example: Potential settings for the 6020RS omni cell for Redox operation

This example describes the analysis of a compound that has a large oxidation half-wave potential, for example, 1200 mV:

- To detect the compound by direct oxidation, a potential that is at least +50 – 100 mV more positive than the half-wave potential is required.
- The signal-to-noise ratio of such a measurement might be fairly poor if the mobile phase contains trace contaminants, which would be oxidized at 1300 mV. The cell lifetime would also be significantly reduced.
- With the Redox operation, the reduction of the oxidized product may be triggered at a more reasonable potential than the oxidation of the original material. If this is the case,

the presence of trace contaminants in the eluent may become less of an issue. As a result, the signal due to these contaminants will decrease. As the background current decreases, the signal-to-noise ratio, and thus the limit of detection, will increase.

Example: Selective resolution using Redox operation with a 6020RS omni coulometric cell

If a sample contains electroactive analytes that co-elute in the analytical column, it is possible to selectively resolve these analytes:

- The potential of the 6020RS omni coulometric cell is set so as to oxidize both compounds.
- The potential of the analytical cell (or cells) is set to analyze only one of the compounds. As a result, a difficult chromatographic separation can be resolved.

5.11 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.

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*.

Function	Description
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**. 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.
Predictive performance	Predictive Performance provides various functions for estimating the lifetime of consumables and for monitoring and recording service and (re)qualification information. *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, refer to the *Chromeleon Help*.
SmartChip Technology	The chip stores unique information about each cell type, including cell model and serial number. When the cell is connected to the potentiostat module, the identification chip technology: • Transmits information directly to the connected chromatography data system for electronic tracking for method validation • Automatically configures the detector with safe, established detection parameters, such as potential limits, to prevent unintended electrode damage If the mini-DIN signal cable is connected between an UltiMate 3000 pump (except the LPG-3400XRS pump) and Digital I/O port 2 on the detector, the cell chip is also able to sense pump flow errors from the connected pump, such as flow interruptions, and as a result turns off potential to the cell.

Function		Description
	SmartChip Technology *(Cont`d)*	In the Chromeleon **Server Configuration** under **View Cell Data** (the **Cell Properties** page opens), the cell information shows the cell properties for the last time the chips were read. Click **Read Smart Cells** *before* opening the **Cell Properties** page to update the cell data and obtain the latest cell information.
	Cells Off	If a pump of the UltiMate 3000 series (except for the LPG-3400XRS pump) is connected to the digital I/O port on the detector using the mini-DIN cable, the potential to the cell is automatically turned off if the pump flow is stopped during a run. This can for example be due to an error from the pump or when the pump flow is stopped manually. **Note:** 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. You can monitor the function of this input in the **Commands** (F8) dialog box if the **ECDRS_Input_3** checkbox is selected, or on the detector display. To select the input in Chromeleon, see section 3.4.3.3, page 37.
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.

5.12 Shutting Down the Detector

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

General guidelines for electrochemical cells

Observe the following precautions before interrupting operation of electrochemical cells:

- Turn off (the potential to) the detector before stopping the pump flow.
- Rinse out any solvents from the detector(s) before removing the cell from the detector.
- For longer periods, always store unused electrochemical cells in their original dust-free packaging.
- For instructions on storing electrochemical cells, observe the respective storage procedures for the cells (→ refer to the *User Guide* for the respective cell).
- *6041RS ultra amperometric analytical cell*
  In addition, note that during periods of detector inactivity, you can keep the amperometric cell assembled with working electrode and gasket being in place.

General guidelines for the detector

- Turn off the temperature control of the column compartment.
- In case of a long-term shut-down of the detector, turn off the power to the detector.
- *Shipping the detector*
  - ♦ Remove the potentiostat modules from the detector (→ section 7.2, page 114).
  - ♦ When shipping the detector, remove the electrochemical cells and the potentiostat modules and install the bay 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.
  - ♦ 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, you can order appropriate shipping containers and packing material from the Thermo Fisher Scientific sales organization. The packing instructions are included in the "Installation and Qualification Documents for Chromatography Instruments" binder and are also available on request.

5.13 Routine and Maintenance Intervals

5.13.1 General Information

The detector is made of high-quality components and materials to minimize maintenance requirements. All surfaces are 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 detector 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 detector 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.

5.13.2 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 buffer solutions are used, 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.
	Platinum thin-film electrodes only Electrochemically treat the electrode daily before sample analysis. For information on the electrochemical treatment, refer to the User Guide for amperometric cells.
Regularly	Check the drain tube connected to the drain port on the bottom right of the detector (→ page 46). 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. See section 7.4.1, page 116.

Frequency	What you should do...
Regularly *(Cont'd)*	Replace the gaskets of the amperometric cell regularly. For replacement instructions, refer to the User Guide for amperometric cells.
	Recalibrate the zero current for the signal channels of the cells by performing an Autozero command from Chromeleon (→ page 76).
Annually	Have a service representative check the detector once a year to prevent contamination and excessive wear.

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 and LEDs

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 20).

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
- the running analysis will be aborted, if applicable.

If the detector is operated with Chromeleon, a message is displayed in the Chromeleon Audit Trail (→ page 102). Check 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.

Cells Off Condition

If the detector is connected to the pump using the Digital I/O port 2, and the pump flow is stopped while potential is applied to one or more cells, the potential to the cell or cells is automatically turned off (Cells Off functionality, → section 5.11, page 96).

In Chromeleon, a message is displayed in the Chromeleon Audit Trail (→ page 102).

Remedy the situation before you continue the analysis:

- Check the pump for any errors. Remedy any errors in the pump.
- Check the components in the system flow path for blockage. Remove any blockages.

If the error has been remedied, start the pump flow again. Then turn on (the potential to) the electrochemical cell or cells.

Operating Problems

For information about operating problems that might occur during the operation of an UltiMate 3000 system, see Operating Problems (→ page 105).

If you are unable to eliminate a problem following the instructions given here, contact your local Thermo Fisher Scientific support center.

6.2 Chromeleon Audit Trail Messages

If the detector is operated by Chromeleon and an error occurs, the following messages may appear in the Chromeleon Audit Trail.

Message	Remedial Action
Bad calibration table detected.	The DC potentiostat module may be defective. Turn the detector off and on to check if the potentiostat module is defective. If the message still appears, replace the potentiostat module (→ page 114).
Cannot obtain the CAZ zero offset value.	The potentiostat module may be defective. Turn the detector off and on to check if the potentiostat module is defective. If the message still appears, replace the potentiostat module (→ page 114).
Cannot obtain the potential offset value.	The potentiostat module may be defective. Turn the detector off and on to check if the potentiostat module is defective. If the message still appears, replace the potentiostat module (→ page 114).
Cell bay leakage detected. Cells have been turned off. Address the problem; disconnect then re-connect to clear the error.	The leak sensor has reported a leak. Check all cells for indications of leakage. Retighten connections if necessary. Dry the leak sensor (→ page 118).
Cell serial number does not match the saved cell serial number.	One or more cells have been installed without updating the cell data in the Chromeleon Server Configuration program. Read and update the cell data (→ page 37).
DC mode configured, but no DC Potentiostat module(s) present. Please check configuration using the Server Configuration.	A DC potentiostat module is missing or not properly installed. Install a DC potentiostat module, or reinstall any existing potentiostat modules. Turn the detector off and on again. Follow the instructions given in the message, and update the configuration settings (→ page 37).
Expected a detector with serial number {X} but found one with serial number {Y}. Please check the configuration using the Server Configuration.	Where {X} = the serial number of the expected detector, and {Y} = the serial number of the detector connected to Chromeleon. The device configuration is incorrect. Open Chromeleon and compare the serial number stated in the Commands dialog box with the serial number on the rear panel of the detector connected to the data system computer.
Failed to close USB.	The USB connection between the detector and the Chromeleon server may be interrupted. Check the USB connection. The power supply to the detector may be interrupted. Check the power supply connection of the detector.
Failed to open USB.	The USB connection between the detector and the Chromeleon server may be interrupted. Check the USB connection. The power supply to the detector may be interrupted. Check the power supply connection of the detector.
Failed to read/write USB.	The USB connection between the detector and the Chromeleon server may be interrupted. Check the USB connection. The power supply to the detector may be interrupted. Check the power supply connection of the detector.

Message	Remedial Action
Failed to start the column heater.	The column compartment may be defective. Turn the detector off and on to check if the column compartment is defective. If the message still appears, the column compartment is defective. Contact Service.
Identification chip data is in conflict with Server Configuration setup. Please check the Server Configuration.	One or more cells have been installed without updating the cell data in the Chromeleon Server Configuration program. Read and update the cell data (→ page 37).
Input Cells Off signal received from pump. Cells have been turned off. Address the problem; disconnect then re-connect to clear the error.	Remedy the situation before you continue analysis. See section 6.1, page 101.
Leakage detected.	The leak sensor has reported a leak. Check all cells for indications of leakage. Retighten connections if necessary. Dry the leak sensor (→ page 118).
Pulse mode configured, but no Pulse/Scan Potentiostat module(s) present. Please check configuration using the Server Configuration.	One or more potentiostat modules are missing or not properly installed. Install the Pulse potentiostat module or reinstall the existing potentiostat module. Turn the detector off and on again. Follow the instructions given in the message, and update the configuration settings (→ page 37).
Self test failed on logic board with error code {X}, expanded below.	Where {X} = error code digit(s). The column compartment may be defective. Contact Service.
Self test failed on PStat {X} channel {Y (Y)} with error code {Z}, expanded below.	Where {X} = the bay to which the potentiostat module is installed, {Y} = the cell channel number, and {Z} = error code digits. The potentiostat module may be defective. Turn the detector off and on to check if the potentiostat module is defective. If the message still appears, replace the potentiostat module (→ page 114). The main board may be defective, contact Service.
The cell identification data for at least one cell does not agree with the configuration, or could not be read. Please check configuration using the Server Configuration. It is recommended that the cells be reread.	One or more cells have been installed without updating the cell data in the Chromeleon Server Configuration program. Follow the instructions given in the message, and read and update the cell data (→ page 37).
The channels configured in the Server Configuration do not agree with the potentiostat module(s) found in the system. Please check configuration using the Server Configuration.	One or more potentiostat modules have been installed or removed without updating the detector configuration settings. Follow the instructions given in the message, and update the configuration settings (→ page 37).
The serial number of the cell in bay {X} does not agree with value from Server Configuration.	Where {X} = The bay to which the cell is installed. One or more cells have been installed without updating the cell data in the Chromeleon Server Configuration program. Read and update the cell data (→ page 37).

Message	Remedial Action
The working electrode material of the cell in bay {X} does not agree with values from the Server Configuration. The driver will disconnect to prevent damage to the cell.	Where {X} = The bay to which the cell is installed. One or more cells have been installed without updating the cell data in the Chromeleon Server Configuration program. Read and update the cell data (→ page 37).
Timeout reading/writing USB.	The USB connection between the detector and the Chromeleon server may be interrupted. Check the USB connection. The power supply to the detector may be interrupted. Check the power supply connection of the detector.

6.3 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 have blown.	Replace the fuses (→ page 120).
	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 26).
The Status LED is illuminated red.	The self-test has failed.	See section 6.2 (→ page 102).
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.
		Restore the cell performance (→ *User Guide* for the cell).

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 116). 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 64). Use freshly prepared mobile phase. Growth of microorganisms in the mobile phase may lead to clogging of the filter.
Poor peak shape (double peak)	Coulometric cells Cell input and output are exchanged.	Verify that the cell input and output are connected correctly.
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 (→ *User Guide* for the cell). If necessary, replace the electrochemical cell (→ *User Guide* for the cell).
	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.

Problem	Probable Cause	Remedial Action
Strong noise, non-periodic baseline fluctuation *(Cont'd)*	The gas content of the eluent is too high.	Degas the eluent and/or install a restrictor at the cell outlet, observing the pressure specification and the general guidelines for the cell (→ *User Guide* for the cell).
	The detector is defective.	Contact Service.
	There is a problem with the cell.	Clean the cell (→ *User Guide* for the cell). If necessary, replace the electrochemical cell (→ *User Guide* for the cell).
	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.
	Coulometric cells Cell input and output are exchanged.	Verify that the cell input and output are connected correctly.
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*).
	Coulometric cells Cell input and output are exchanged.	Verify that the cell input and output are connected correctly.
	The column is overloaded or contaminated.	Clean or replace the column.

Problem	Probable Cause	Remedial Action
Peak broadening, increased dead time *(Cont'd)*	The solvent is degraded or has changed.	Use fresh solvent.
	The selected response time is too large.	Select a suitable response time, e.g., using the Chromeleon Program Wizard.
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.
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 observing the pressure specification and the general guidelines for the cell (→ *User Guide* for the cell).
	Electrical interferences from other instruments and other electrical devices in close proximity.	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 and/or post-column cooler (→ *TCC-3000RS Manual*) at the cell outlet, observing the pressure specification and the general guidelines for the cell (→ *User Guide* for the cell).
Negative Peaks	Sample solvent and mobile phase differ in composition.	Dissolve the sample in the mobile phase.
High Background Current– DC Mode	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.

Problem	Probable Cause	Remedial Action
High Background Current– DC Mode *(Cont'd)*	There are electroactive impurities in the mobile phase.	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 5.8.3 (→ page 85).
	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 (→ *User Guide* for the cell).
	Contaminants leach from system components.	Check the mobile phase reservoir filters, column end frits and replace them if necessary. Passivate the system components (→ page 131).
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 for the cell (electrode) has shifted.	Generate a new HDV to optimize operating potential (→ page 84). Perform an electrochemical treatment with the cell (→ *User Guide* for the cell).
	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	Try restoring the cell performance (→ *User Guide* for the cell).

Problem	Probable Cause	Remedial Action
Loss of response *(Cont'd)*	The wrong potential was selected.	A different potential may be required.
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 (→ *User Guide* for the cell). *Coulometric cells* Replace the cell (→ *User Guide* for the cell). *Amperometric cell* Replace the working electrode (→ *User Guide* for the cell).

7 Service

7.1 General Notes and Safety Precautions

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

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

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

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

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

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

- For all service and repair procedures, observe all precautionary statements provided in these operating instructions.
- Use only the original spare parts authorized for the device by Thermo Fisher Scientific.
- Before returning the detector for repair, contact your local Thermo Fisher Scientific support center. 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 the Thermo Fisher Scientific sales organization. The packing instructions are included in the "Installation and Qualification Documents for Chromatography Instruments" binder and are also available on request.

For instructions on shutting down the unit, see page 98.

7.2 Removing the Potentiostat Module

Parts required

Only if the potentiostat module is to be replaced:

- Potentiostat Module for dual-channel DC Mode

  –or–
- Potentiostat Module for Pulse Mode

  Note: Operation of this potentiostat module requires an amperometric cell with a noble metal-based working electrode such as gold.

  For the specifications of the potentiostat modules, see section 8, page 123.

Tip: After you have installed a potentiostat module, always perform a relay test with the SimulatorRS cell *before* you use other electrochemical cells or QualifierRS cells with the potentiostat module.

Tools required

- Electrostatic discharge protective measures

  Important: To avoid a loss of performance or functionality, take proper electrostatic discharge (ESD) protective measures.
  To avoid permanent damage to the potentiostat module and/or the detector, do not replace the potentiostat when the detector is turned on.

  Important : Afin d'éviter de perte de performances ou les fonctionnalités, prenez des mesures appropriées de précaution de décharge électrostatique.
  Afin d'éviter des dommages permanents au module potentiostat et/ou le détecteur, ne remplacent pas le potentiostat lorsque le détecteur est allumé.

- Torx™ screwdriver, size T10 (included in the accessories kit for the detector)

Preparations

1. Turn off the detector to avoid potential damage to the detector and the potentiostat module.
2. Remove the electrochemical cell from the potentiostat module. For removal instructions, refer to the *User Guide* for the respective electrochemical cell.

Follow these steps

1. With the Torx screwdriver (size T10), loosen the 2 screws that attach the potentiostat module to the interior front panel. Keep the screws for the replacement potentiostat module.

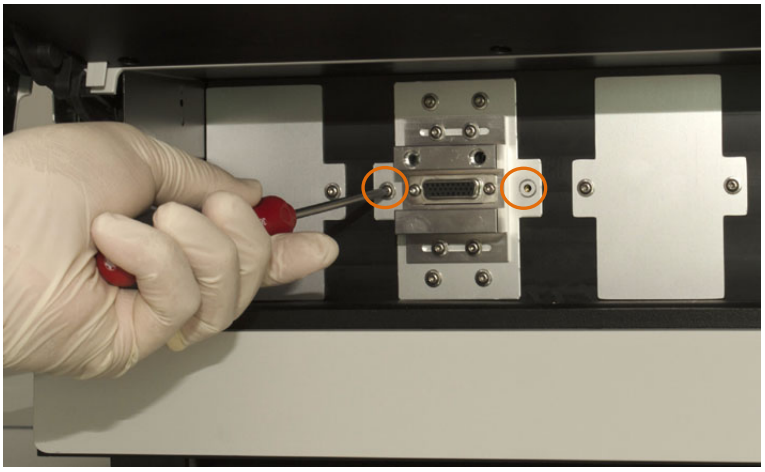


Fig. 29: Loosening the attachment screws

2. Remove the potentiostat module from the bay: Carefully with one hand grasp and pull the potentiostat module towards you. If required, hold the interior front panel with the other hand.

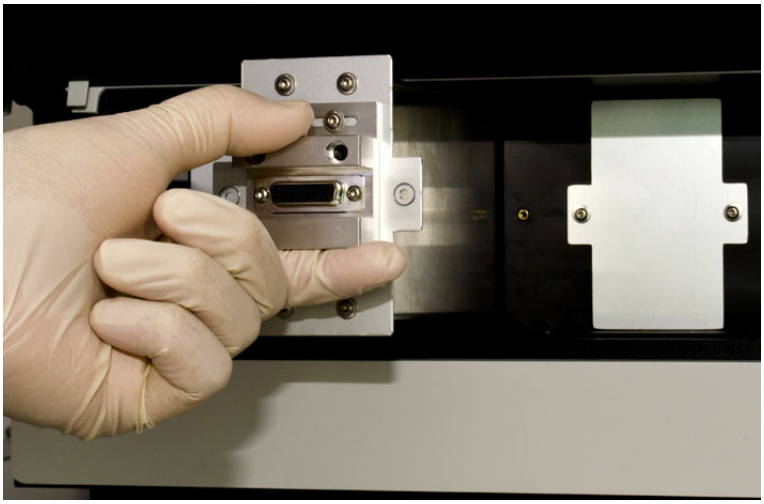


Fig. 30: Pulling out the potentiostat module (here: Bay B)

3. This step depends:

   - *If the bay on the detector front panel remains empty*

     Install the bay cover with the bay cover that was shipped with the detector to close the bay and prevent dust from entering into the cell bay and reaching sensitive electronics.

     a) Position the bay cover, aligning the screw holes.
     b) Tighten the 2 screws with the Torx screwdriver (size T10) to attach the cover to the bay.

   - *If a new or replacement potentiostat module is to be installed*

     Follow the steps in the *Installation Instructions for the Potentiostat Module*.

     Make sure that you perform a relay test as operation check for the potentiostat module after replacement. See section 4.3.3, page 48.

7.3 Electrochemical Cells

A number of procedures for performance recovery, maintenance and replacement procedures for the electrochemical cells are available.

For instructions, refer to the *User Guide* for the respective electrochemical cell that is shipped with the cell.

7.4 In-Line Filters

7.4.1 Replacing the Filter Element

When

For optimum performance, replace the filter units on a periodic basis. The frequency depends 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. For example, microbial growth may occur in mobile phases with low levels (< 3%) of organic solvents, and contribute to high background current and reduced cell performance.

Parts required

Filter element, depending on the in-line filter type:

- Graphite filter element for the graphite in-line filter
- PEEK filter element for the PEEK in-line filter

Additional items required

Deionized water

Preparations

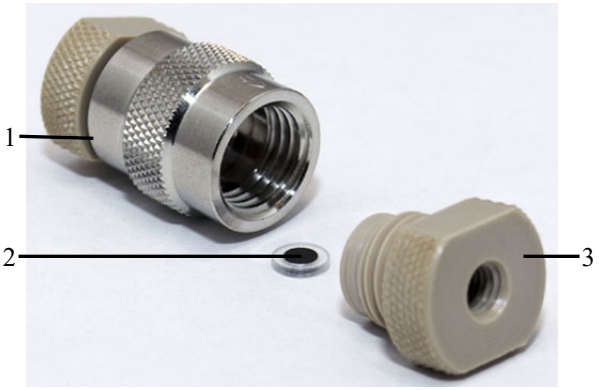
1. Turn off the potential to the electrochemical cells.
2. Stop the pump flow. Allow system pressure to drop to zero before disconnecting any components from the system flow path.

⚠ **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 :** N’ouvrez pas les raccords situés dans la partie pressurisée précédant la colonne avant que la pression ne soit retombée à zéro. Un changement rapide de pression peut endommager les différents composants de la chaîne. Attendez toujours que la pression redescende à zéro avant d’ouvrir les connexions.

Follow these steps

1. Disconnect the capillaries from the filter holder that contains the filter element.
2. Open the filter holder by turning the lock nuts counterclockwise with your fingers.



No.	Description
1	Filter holder
2	Graphite filter element
3	Lock nut

Fig. 31: In-line filter (here: 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. Install the filter element and connect the in-line filter in the direction of flow as stated on the filter holder to the system flow path. Follow the instructions for the respective in-line filter type:
   - ♦ Connecting the Graphite In-Line Filter, → section 4.4.3.1, page 60
   - ♦ Connecting the PEEK In-Line Filter, → section 4.4.3.2, page 61
6. Re-connect the in-line filter in the system flow path. For details on the flow connections, see section 4.4.2, page 54.

7.4.2 Replacing the In-Line Filter

Parts required

Depending on the in-filter type that is to be replaced:

- In-line filter kit with graphite filter elements
- In-line filter kit with PEEK filter elements

Preparations

1. Turn off the potential to the electrochemical cells.
2. Stop the pump flow. Allow system pressure to drop to zero before disconnecting any components from the system flow path.

⚠ **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 :** N'ouvrez pas les raccords situés dans la partie pressurisée précédant la colonne avant que la pression ne soit retombée à zéro. Un changement rapide de pression peut endommager les différents composants de la chaîne. Attendez toujours que la pression redescende à zéro avant d'ouvrir les connexions.

Follow these steps

1. Disconnect the capillaries from the filter holder that contains the filter element.
2. Locate the new in-line filter.

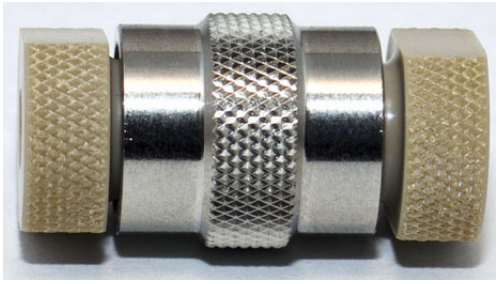


Fig. 32: Filter holder with lock nuts installed

3. Install the filter element and connect the in-line filter in the direction of flow as stated on the filter holder to the system flow path. Follow the instructions for the respective in-line filter type:
   - Connecting the Graphite In-Line Filter, → section 4.4.3.1, page 60
   - Connecting the PEEK In-Line Filter, → section 4.4.3.2, page 61

7.5 Drying the Leak Sensor

When

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.

Items required

Cloth or tissue

Follow these steps

1. Stop the detector operation:
   a) Turn off the potential to the electrochemical cells.
   b) Stop the pump flow.
   c) Disconnect the detector in Chromeleon.
   d) Turn the detector off.
2. Inspect all electrochemical cells for signs of leakage. If a cell is leaking, tighten the connections to the electrochemical cell. If necessary, replace the cell (→ *User Guide* for the respective electrochemical cell).
3. With a cloth or tissue, absorb all liquid that has collected in the tray.

   ⚠ **Important:** Be careful not to bend or damage the sensor.

   ⚠ **Important :** Assurez-vous de ni tordre, ni endommager le capteur.

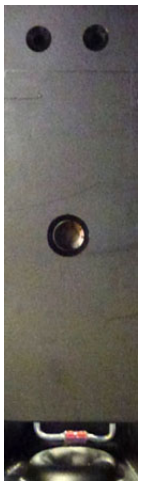


Fig. 33: Leak sensor

4. Allow the sensor to adjust to the ambient temperature for a few minutes.
5. Turn on the detector and reconnect it in Chromeleon. Wait until the self-test (→ section 5.1, page 67) is completed.
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. You can resume operation only when the cause for leakage has been completely removed.

7.6 Replacing the Main Power Fuses

Warning: Turn off the main power switch. 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.

Parts required

2 fuses, 1A, slow-blow (5 x 20 mm) from the fuses kit

For information about the kit, see section 9.3 (→ page 128).

Follow these steps

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

Fig. 34: Fuse cartridge

2. Replace the fuses.

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

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

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 detector firmware, 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 38), 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.)

   Note that the Chromeleon server needs to be in running idle mode for the download. 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.

   Tip: You can follow the firmware download in the Chromeleon Audit Trail.

7. After the firmware download has finished successfully, save and exit the Chromeleon **Server Configuration** program.

8. Stop and restart the **Server Monitor**.

9. Open the **Server Configuration** program and re-configure the detector (→ page 37). If a problem occurs during the configuration, delete the detector from the timebase and set it up anew.

Tip: In Chromeleon, click **Wellness** on the tabset panel to check if the firmware has been updated correctly.

8 Technical Information

The table below lists the specifications for the detector. For specifications on the electrochemical cells, refer to the *User Guide* for the respective electrochemical cell.

Specification	DC Mode	Pulse Mode
Detection type:	Electrochemical detection	
Operating mode:	Direct Current (DC) Fixed potential	Pulse (PAD) Periodic changes in potential
Potential control (potentiostat module):	Up to 4 channels (1 or 2 dual-channel DC potentiostat modules required)	Single-channel with a 4 potential waveform (pulse potentiostat module required)
DC potential range:	max. ± 3300 mV in 1 mV steps (acceptable range determined by cell type)	± 3300 mV in 1 mV steps (per pulse segment)
Signal range:	10 pA to 100 µA	10 pA to 100 µA
Automatic gain ranging:	Supported	Not supported
Acquisition delay:	n. a.	50 ms to 1 ms – 5 ms
Pulse width (time):	n. a.	T1 = acquisition delay +5 ms to 1000 ms T2 = 4 ms to 1000 ms T3 = 0 ms to 1000 ms T4 = 0 ms to 1000 ms
Potential resolution:	1 mV	1 mV
Typical noise:	< 750 fA (0.75 pA) with SimulatorRS cell (DC Mode), filter constant: 5 seconds	< 2 % of full-scale range
Noise filtering:	Advanced multi-level digital filtering, adjustable filter constant	Settable: None, Low, Medium, High
Data collection rate:	2 Hz, 5 Hz, 10 Hz, 20 Hz, 50 Hz, 100 Hz; Up to 200 Hz (under Chromeleon 7.1 or later)	
Autozero	Supported	
Column compartment:	Integrated temperature control with liquid leak detection	
Temperature range:	Adjustable from ambient +5 °C 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.	

Specification	DC Mode	Pulse Mode
Safety features:	Leak sensor; SmartChip technology for limitation of electrochemical cell potential and cell monitoring; cell potential shutdown mechanism	
Display:	Multi-line 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)	
Power requirements:	100 - 120 V AC, 220 - 240 V AC, ± 10%; 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 Operating altitude: Maximum 2000 m above sea level Overvoltage category: II Pollution degree: 2	
Dimensions (h × w × d):	19 × 42 × 51 cm (7.6 × 16.5 × 20 in.)	
Weight:	Approx. 12.5 kg (approx. 27.5 lbs) with 1 potentiostat module for DC Mode	

Technical information: October 2019
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, consumables and spare parts, contact the Thermo Fisher Scientific sales organization.

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.

Description	Part no.	Quantity in the accessories kit
6-pin mini-DIN signal cable For connection to UltiMate 3000 pumps (except the LPG-3400XRS pump). For further information, see page 33.	6070.9911	1
Accessories for the ECD-3000RS		
Attachment screws for cell bay covers (size Torx T10)	----	8
Fuses Kit for ECD (2 fuses, 1A, slow-blow, 5 x 20 mm)	70-6666	1
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
Parts for connecting the detector to the drain system:		
Tee piece for drainage system	----	1
L piece for drainage system	----	2
Flexible hose (11.4 mm O.D. x 8.3 mm I.D.)	----	1 m
Screwdriver, Torx, size T10, for cell bay cover screws	----	1
SimulatorRS simulation cell, with installation instructions	6070.4100	1

Description	Part no.	Quantity in the accessories kit
Tubes and fittings, including		
Tubing, PEEK (I.D. x O.D. 0.005" x 1/16"), red	6081.1410	2 m
Tubing, PEEK (I.D. x O.D. 0.015" x 1/16"), gray (for use as cell waste line)	6081.1420	1.5 m
Capillary, PEEK (I.D. x O.D. 0.18 mm x 1/16")	6827.5002	1.5 m
Capillary, Viper, PEEK (I.D. x L. 90 µm x 75 mm)	6041.9075	1
Lock nut, long, seal-tight, with ferrule (PEEK, O.D. 1/16")	70-4859	2
Lock nut, short, seal tight, with ferrule (PEEK, O.D. 1/16")	70-4746	10
Fitting, two-piece, fingertight (PEEK, 1/16" O.D.), RheFlex™	6000.0011	4
Tubing cutter for PEEK capillaries	6300.0401	1
USB cable type A to type B, 5m	6911.0002	1

9.2 Optional Accessories

Description	Part no.	Remarks
6011RS ultra Coulometric Analytical Cell Dual-electrode coulometric cell for multiple analyte detection	6070.2400	For installation instructions, refer to the *Coulometric Cells User Guide*.
6020RS omni Coulometric Cell Single-electrode coulometric cell, recommended for single-analyte detection, interference screening or analyte conversion	6070.2100	For installation instructions, refer to the *Coulometric Cells User Guide*.
6041RS ultra Amperometric Analytical Cell, for analytical purposes Including • Thin-layer amperometric cell for single-channel detection • Gaskets • Anti-static tweezers *Note:* The 6041RS amperometric cell is shipped without working electrode. Select the required working electrode for the cell.	6070.3000	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Adapter for thin-film electrode (without electrode)	6070.3005	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.

Description	Part no.	Remarks
Capillary kit, nanoViper, for UltiMate 3000 systems with ECD-3000RS detector	6041.5105	Includes the following nanoViper capillaries, PEEK, 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 Shipped with installation instructions.
Drain kit for UltiMate 3000 systems	6040.0005	The kit includes all required components and detailed installation instructions.
Polishing kit for GC and Au working electrodes, including • 1 polishing disc on glass plate • 1 bottle alumina suspension, 25 mL	6070.3110	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Potentiostat module for Pulse Mode	6070.1420	Including installation instructions.
Potentiostat module for DC Mode	6070.1400	Including installation instructions.
Pulse QualifierRS simulation cell for Pulse Mode	6070.4300	Including installation instructions.
QualifierRS simulation cell for DC Mode	6070.4200	For OQ/PQ purposes, including installation instructions.
SimulatorRS simulation cell for relay testing, including installation instructions	6070.4100	Including installation instructions.
Thin-film working electrode kit, platinum (Pt), including • Thin-film working electrode, platinum (Pt) • Adapter for thin-film electrode	6070.3510	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Thin-film working electrode, platinum (Pt) (without adapter)	6070.3500	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Working electrode (plate type), boron-doped diamond (BDD)	6070.3100	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Working electrode (plate type), glassy carbon (GC), high efficiency	6070.3200	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.
Working electrode (plate type), gold (Au)	6070.3300	For installation instructions, refer to the *Amperometric Cell and Working Electrodes User Guide*.

9.3 Consumables and Spare Parts

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

For details on capillary connections, see section 4.4.2 (→ page 54).

Description	Part no.
Bay cover, including 2 attachment screws	6070.1411
Capillaries, nanoViper, for UltiMate 3000 systems with ECD-3000RS detector For ordering information for the capillary kit, see section 9.2, page 126.	
Capillary, nanoViper, I.D. x L 0.1 x 150 mm, PEEK For connection between 6011RS ultra coulometric and 6041RS ultra amperometric analytical cell.	6041.5811
Capillary, nanoViper, I.D. x L 0.1 x 250 mm, PEEK, 2 capillaries	6041.5812
Capillary, nanoViper, I.D. x L 0.1 x 450 mm, PEEK	6041.5814
Capillary, nanoViper, I.D. x L 0.15 x 250 mm, PEEK	6041.5819
Capillary, nanoViper, I.D. x L 0.15 x 450 mm, PEEK	6041.5821
Capillary, nanoViper, I.D. x L 0.15 x 550 mm, PEEK	6041.5822
Capillary, nanoViper, I.D. x L 0.15 x 750 mm, PEEK For example, for connection of an LPG-3400 pump to graphite in-line filter.	6041.5823
Capillary and fitting kit for ECD-3000RS detector, including • Capillary, PEEK (I.D. x O.D. 0.18 mm x 1/16"), L 1.5 m • Tubing, PEEK (I.D. x O.D. 0.005" x 1/16"), red, L 2.0 m • Tubing, PEEK (I.D. x O.D. 0.015" x 1/16"), gray, L 1.0 m (for use as cell waste line) • Lock nut, long, seal-tight, with ferrule (PEEK, O.D. 1/16"), 2 lock nuts • Lock nut, short, seal tight, with ferrule (PEEK, O.D. 1/16"), 10 lock nuts • Fitting, two-piece, fingertight (PEEK, 1/16" O.D.), RheFlex, 4 fittings	6070.4800
Capillary, PEEK (I.D. x O.D. 0.18 mm x 1/16"), L 2.0 m	6827.5002
Cell waste line kit, including • Capillary (PEEK, 0.015" x 1/16" I.D. x O.D., L 1.5 m, gray) • 2 fitting screws (PEEK, 1/16")	6070.4900
Column chamber panel	6070.1502
Filter elements set, graphite, 5 filter elements	70-0898
Filter elements set, PEEK, 5 filter elements	70-3824
Fitting, two-piece, fingertight (PEEK, 1/16" O.D.), RheFlex, 4 fittings	6000.0011
Fuses kit for ECD-RS (2 fuses, 1A, slow-blow, 5 x 20mm)	70-6666
Gasket, volume: 25 nL, BoPET, 5 gaskets For 6041RS ultra Amperometric Analytical Cell	6070.2528
Gasket, volume: 50 nL, BoPET, 5 gaskets For 6041RS ultra Amperometric Analytical Cell	6070.2529

Description	Part no.
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
Lock nut, long, seal-tight, with ferrule (PEEK, O.D. 1/16")	70-4859
Lock-nut, short, seal tight, with ferrule (PEEK, O.D. 1/16"), 10 lock nuts	70-4746
Mini-DIN signal cable, 6-pin For connection to UltiMate 3000 pumps (except the LPG-3400XRS pump). For further information, see page 33.	6070.9911
Power cord, Australia	6000.1060
Power cord, China	6000.1080
Power cord, Denmark	6000.1070
Power cord, EU	6000.1000
Power cord, India/SA	6000.1090
Power cord, Italy	6000.1040
Power cord, Japan	6000.1050
Power cord, Switzerland	6000.1030
Power cord, UK	6000.1020
Power cord, US	6000.1001
Tubing cutter for PEEK capillaries	6300.0401
Tubing, PEEK (I.D. x O.D. 0.005" x 1/16"), red, L 2.0 m	6081.1410
Tubing, PEEK (I.D. x O.D. 0.015" x 1/16"), gray, L 1.5 m (for use as cell waste line)	6081.1420
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

10 Appendix

10.1 Passivation

When

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.

Items required

- Solvent mixture of 50% isopropyl and 50% water
- HPLC-grade water
- 6N nitric acid
- 2% ethylenediaminetetraacetic acid (EDTA)

Preparations

⚠ **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, vérifiez 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; retirez la colonne et la cellule électrochimique avant de commencer le processus de passivation.

Follow these steps

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

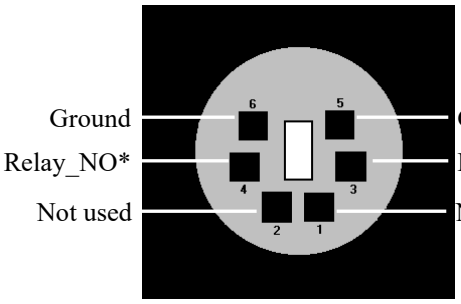
   Warning: To avoid damage to the skin and eyes, wear appropriate protective clothing and goggles when using nitric acid.

   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. 35: Mini-DIN Digital I/O port

To connect an UltiMate 3000 pump (except for 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. 36: Pin assignment (port and cable)

Tip: The input has a pull-up resistor.

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