Thermo Scientific Dionex UltiMate 3000 Series

6011RS ultra Coulometric Cell
6020RS omni Coulometric Cell

For Electrochemical Detector ECD-3000RS

User Guide

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1 General Information

1.1 About this Guide

This user guide provides instructions for installation, set up, operation, shut down and maintenance of the 6011RS ultra Coulometric Analytical Cell and 6020RS omni Coulometric Cell for the ECD-3000RS Electrochemical Detector of the Thermo Scientific™ Dionex™ UltiMate™ 3000 series.

This guide is intended as a supplementary document to the *UltiMate 3000 ECD-3000RS Detector Operating Instructions*.

Refer to the *Operating Instructions* for the detector for general safety information on the device, and the conventions used throughout this guide regarding safety messages, notices and typography.

Keep this user guide with the *Operating Instructions* for the detector for quick reference.

The following conventions apply to throughout this user guide:

- The cell is referred to as *cell* or *coulometric cell* in this user guide. If other cell types are referenced, they are identified by name.
- The cell configuration may vary. Therefore, not all descriptions necessarily apply to your particular cell configuration.
- Illustrations in this user guide are provided for basic understanding. They can vary from the actual model of the cell or component. However, this does not influence the descriptions. No claims can be derived from the illustrations in this user guide.
- If not otherwise stated, the descriptions for the Viper™ capillary connections apply also to the nanoViper™ and possible other Viper capillary connections.

1.2 Safety

1.2.1 Safety Symbols and Signal Words

At various points throughout the user guide, messages of particular importance are indicated by certain symbols and signal words:

Warning: Indicates that failure to take note of the accompanying information may result in personal injury.

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

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

1.2.2 Safety Precautions and Guidelines

Warning: Observe all safety precautions and guidelines as stated in the *Operating Instructions* for the detector.

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

- Electrostatic discharge can cause performance degradation or a loss of functionality of the detector or electrochemical cell. To avoid this, take proper electrostatic discharge (ESD) protective measures.

- 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.
- The electronic connector and the contacts of the cell identification chip on the rear side of the cell body are sensitive to contamination and damage.
  - ♦ Hold the cell only by the cell body. Do not touch the sensitive electronics.
  - ♦ Inspect the connector and ensure that there are no bent pins before installing the cell.
  - ♦ If liquid comes into contact with the identification chip, immediately remove it and wipe it dry to prevent any salt buildup or corrosion.
- The cell is sensitive to contamination. Wear protective gloves when handling the cell components and working electrodes. Before connecting the cell in the system flow path, flush the instruments in the flow path before the detector to waste, and flush the cell separately to waste, without a column connected.
- 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, refer to the *Operating Instructions*.

- Do not expose a coulometric cell to the following chemicals if these are present individually or in a mixture in high concentration (above 50%). These chemicals include: Aqua regia, benzene sulphonic acid, chlorosulphonic acid, chromic acid, hydrobromic acid, hydrofluoric acid, nitric acid, oleum, sulphuric acid, trifluoromethyl sulphonic acid, phenol, dimethylsulphoxide, or diphenylsulphone.
- 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 (8992 psi, 62 MPa)
  Exceeding this pressure may lead to leaks in the cell.

  With the 6011RS ultra Coulometric Analytical 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.

  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.
- 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 specifications, see chapter 6, page 43.
- Make yourself familiar with further safety guidelines for electrochemical cells in the *Operating Instructions* for the detector.

2 Cell Overview

2.1 Cell Operating Principle

In a coulometric cell, the liquid chromatography (LC) eluent actually flows through the high surface area porous graphite electrode. The electrode ensures that diffusion and convection controlled processes do not limit the electrochemical response of the detector. This allows complete oxidation (reduction) of the electroactive compound in solution and maximizes the sensitivity of the analysis. This type of electrochemical cell provides essentially complete conversion (~ 100%) as an electroactive species passes through.

This capability of coulometric flow-through electrodes permits them to be sequentially connected in the flow path to provide multi-electrode detection capabilities with the UltiMate 3000 electrochemical detector. This capability alone provides the capacity to resolve co-eluting analytes in a single analytical run.

2.2 Cell Description

Coulometric electrochemical cells incorporate a coulometric-style, flow-through design with high surface area, micro-porous electrode(s).

Solid-state palladium reference electrodes are used exclusively within every electrochemical cell. They provide stability and reliability for maintenance-free operation by eliminating the need for periodic filling., cleaning or replacement.

The coulometric electrochemical cells have been specifically designed for use in liquid chromatography (LC) analyses using Direct Current (DC) Chronocoulometry, such as in quantitation, compound modification or alternatively for screening purposes.

SmartChip Technology

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

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

2.2.1 6011RS ultra Coulometric Analytical Cell

The 6011RS ultra Coulometric Analytical Cell incorporates dual-inline flow-through, micro-porous graphite working electrodes, with independent potential control and dual-channel signal output. This internal flow path inside the cell is designed for conventional and ultra-fast liquid chromatography and is optimized for minimized dispersion and improved resolution.

The 6011RS ultra Coulometric Analytical Cell can be used for analytical detection purposes in dual-channel DC Mode or for selective screening of interferences.

- For dual-channel detection purposes, the first electrode can act as an analytical electrode at one set potential. The second electrode can then be used to act as an analytical electrode at a higher potential to measure a different analyte.

- Alternatively, for screening purposes, the first electrode in the 6011RS ultra cell may also be used as the screening electrode, while the second electrode can be used as the analytical electrode.
- With more than one 6011RS ultra cell in the detector, the 6011RS ultra cells can be combined in series for detection set to selectively higher potentials for detection of multiple analytes.

2.2.2 6020RS omni Coulometric Cell

The 6020RS omni Coulometric Cell incorporates a single-channel flow-through, micro-porous graphite working electrode in a pressure-resistant housing, with potential control and single-channel signal output.

The 6020RS omni cell is designed to provide a steady electrochemical potential to a porous, flow-through electrode that can be used as follows:

- For analyte detection in single-channel DC Mode
- To oxidize (or reduce) interfering electroactive species and contaminants from the mobile phase
- To selectively change the oxidation state of an analyte to improve detection by a down-stream electrode

The 6020RS omni cell can eliminate interferences present in the mobile phase or from the injected sample. This cell can also act to condition the sample stream that exits the analytical column before the analytes enter the analytical electrochemical cell. This is typically known as *electrochemical screening*, where other interferences from the sample itself are eliminated to improve selectivity. In this case, the electrode in the 6020RS omni coulometric cell is referred to as a *screening electrode*.

The 6020RS omni coulometric cell can be placed in a variety of positions within the flow path of the system. These include pre-injection, post-injection and post-column positions.

For information on *electrochemical screening* and details on the use of the 6020RS omni coulometric cell, refer to the *Operating Instructions for the detector*.

For details on the operational modes, refer to the *Operating Instructions* for the detector.

2.3 Cell Components

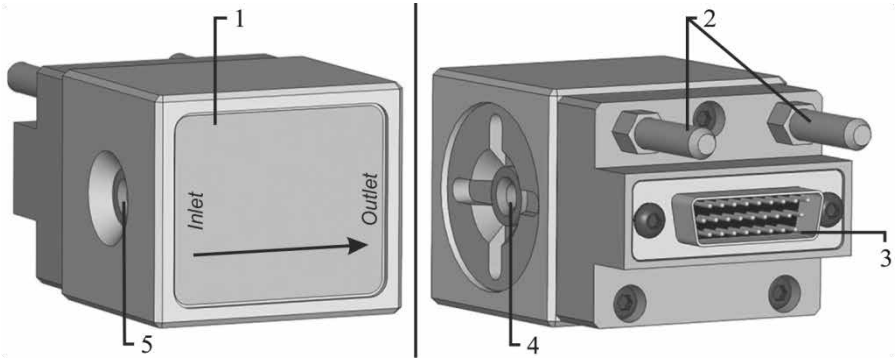

Fig. 1: Coulometric cell, left: front view; right: rear view

No.	Description
1	Cell body with label
2	Guiding pins for connection to potentiostat module
3	Cell connector for connection to potentiostat module
4	Flow outlet for cell waste line
5	Flow inlet from system flow path

3 Installation

3.1 Cell Installation and Flow Connections

The procedure for replacing or reinstalling the cell corresponds to the installation steps upon first-time installation.

⚠ **Important:** 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 (8992 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.

3.1.1 Guidelines for Installation

When installing the coulometric cell, observe the following guidelines:

- When connecting capillaries to a coulometric cell, observe the direction of flow indicated on the cell label. Make sure that the flow connections on the IN and OUT ports are correctly made. Do not invert input and output.
- No tools are required to remove and install an electrochemical cell when using Viper capillaries.

- In-line filters that are provided with the ECD-3000RS detector can be installed in the system flow path to protect the sensitive electrochmical cell from particulate contamination.
- To avoid that the capillaries are pinched when the door is closed, route the capillaries to the outside through the slots in the detector enclosure.
- Capillary connections between the analytical column outlet and analytical electrochemical cell should be as short as possible to avoid peak broadening effects due to excessive volume.
- When installing a new cell to the detector (for example, upon replacement), observe the following guidelines:
  - ♦ Always update the cell ID and working electrode information in the chromatography data system, such as Chromeleon.
  - ♦ 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.
    For further information on the hydrodynamic voltammogram, see section 4.3.2, page 22.
- The 6020RS omni coulometric cell can be placed in a variety of positions within the flow path of the system. These include pre-injection, post-injection and post-column positions.
- For more information on the system flow path, refer to the *Operating Instructions* for the detector.

3.1.2 Installing the Cell and Flow Connections

Parts required

- Coulometric cell
- Protective gloves (powder-free)
- Potentiostat module (installed in the detector)
- Inlet capillary
- Cell waste line (such as from cell waste line kit), consisting of:
  - ♦ Capillary, PEEK, I.D. 0.015", gray
  - ♦ 2 tubing fittings, PEEK, 1/16"
- Analytical mobile phase

Preparations

1. Wear the protective gloves.
2. Unpack the cell.
3. *If the cell is new or was stored*
   Remove the storage plugs from the inlet and outlet of the coulometric cell.

   Tip: Keep the storage plugs of the cell, for example in the cell packaging, to have them easily available when storing or transporting the cell.
4. Install the potentiostat module to the bay to which you want to install the cell. Refer to the *Installation Instructions* for the potentiostat module.

5. *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.

6. Perform a relay test with the potentiostat module, using the SimulatorRS cell. Refer to the *Operating Instructions* for the detector.

7. Prepare the waste line: Slide one tubing fitting over one end of the waste capillary.

8. Set up the flow connections in the system flow path before the detector.

9. Flush the system modules in the flow path before the detector to waste using an appropriate mobile phase for at least 1 hour at a flow rate of 1 mL/min to remove any potential contaminants.

Follow these steps

1. Set up the flow connections to the cell.

   ℹ **Important:** When connecting capillaries to a coulometric cell, observe the direction of flow indicated on the cell label. Make sure that the flow connections on the IN and OUT ports are correctly made. Do not invert input and output.

a) Connect the capillary to the inlet of the cell.

b) Connect cell waste line to the outlet of the cell: Screw the fitting end into the outlet port of the cell. Route the waste line to waste.

2. Install the cell to the potentiostat module:

a) Align the guiding pins of the cell with the alignment holes on the potentiostat module.

b) Push the cell connector completely into the potentiostat module connector.

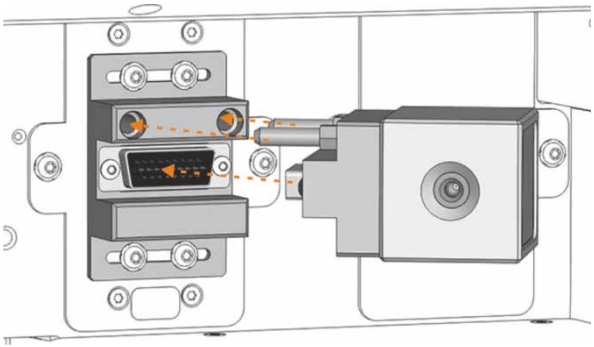

Fig. 2: Installing the cell to the potentiostat module

3. Flush the cell. Leave the potential turned off during flushing.

a) Connect the pump outlet directly to the cell inlet.

⚠ **Important:** To avoid contamination, make sure that no analytical column is connected in the flow path when flushing a cell.

b) Check that the cell waste line routes to waste.

c) Set the pump flow rate as required for the intended application. Turn the pump flow on.

d) Check the cell for leaks and the leak sensor setting.

e) Flush the cell to waste with analytical mobile phase at the flow rate required for analysis for at least 20 minutes to remove impurities and help conditioning the cell. Observe all *Mobile Phase Guidelines* in the *Operating Instructions* for the detector.

Tip: 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.

4. Set up the flow connections in the system and to the cell. Refer to the *Operating Instructions* for the detector.

5. Configure the cell (→ page 15).

3.1.3 Configuring the Cell

To use the cell with a chromatography data system, such as Chromeleon, configure the cell.

This section describes the cell configuration using the Chromeleon Chromatography Data System. For other chromatography data systems, refer to the documentation of the data system.

1. Open the Chromeleon **Server Configuration** program.
2. On the configuration pages for the detector on the **Detector** page, click **Read Smart Cells**. The cell information for this bay is updated. For more information, refer to the *Operating Instructions* for the detector.
3. Click **View Cell Data** to open the **Cell Properties** dialog.
4. Select the cell bay to which you installed the cell in the **Cell Properties** dialog.
5. Click **Update** and confirm the settings.
6. Save the detector configuration and close the Chromeleon **Server Configuration** program.
7. *If the coulometric cell is used as analytical cell*
   Equilibrate the cell. See next section.

3.2 Equilibrating the Cell

If the coulometric cell is used as analytical cell, observe the following:

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.

Preparations

Install and configure the cell. Follow the instructions in section 3.1, page 9.

Follow these steps

1. Set the flow rate for the analyses and start the pump flow.
2. Set the potential(s) to the value(s) required for the application in the chromatography data system.
3. Start the data acquisition.

4. *If you use the Chromeleon software*
   On the panel tabset, select **More Options** to open the More Options dialog. Select **Start Equilibration** for the cell that you want to equilibrate.

   Tip: When the equilibration of the cell has been successful, the boxes that displayed **Measuring** during the equilibration will show values.

5. Monitor the current for the cell that is equilibrated.

   Initially, the current from the working electrode will be high. Over time, this current decays exponentially. The time required for equilibration depends on the desired sensitivity.

   The following equilibration period is intended only as guideline and is subject to change, based on the application: Allow the coulometric cell to equilibrate for at least 1 hour before routine analysis (DC Mode only).

6. The equilibration has been successful once the baseline signal of the cell is stable and the noise has diminished.

Tip: When using a new cell coulometric cell, always perform an HDV to optimize the potential for optimum cell performance.

4 Operation

4.1 General Information about Control

As part of the ECD-3000RS detector, the coulometric cell is operated using chromatography data systems, such as the Chromeleon Chromatography Management System.

For information on the chromatography data system, refer to the data system documentation and its Help.

4.2 Guidelines for Operation

⚠ **Important:** Do not operate coulometric cells in Pulse Mode.

Operating coulometric cells in Pulse Mode (pulsed amperometric detection, PAD) with the Pulse potentiostat module will cause irreparable damage to the porous electrodes inside the cell.

Observe the following guidelines for operation of the cell:

- The coulometric cells 6011RS ultra and 6020RS omni can be used for analytical purposes in DC Mode.
- Alternatively, the coulometric cells can be used for screening operation and redox operation.

  For details on the coulometric cells, see section 2.2, page 5.

  For information on screening operation and redox operation, refer to the *Operating Instructions* for the detector.
- Monitor background currents. Changes in the background current may be indicative of a possible problem.
- Do not apply potentials to the cell if no electrolyte is present in the mobile phase and during organic cleaning or aqueous washing procedures.

- Remove the electrochemical cell from the detector when connecting a new column. Allow the column to flush for several hours to remove particles from the column before re-attaching the cell.
- Be sure to turn off the (potential to the) cell and remove it from the detector when chemically cleaning it or any other component of the system.
- If a problem occurs in the system, first check other components of the system before making the conclusion that the problem is a result of the cell.
- If a cell leaks, remove the cell from the detector immediately.
  With coulometric cells, remove and replace the cell
  (→ section 5.5, page 42).
- When problems with the cell or a loss in cell performance occur, check the *Operating Problems* section. Refer to the *Operating Instructions* for the detector for remedial action.
- 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.
- If a sample analysis is critical, it may help to have a replacement cell available before starting the analysis.

4.3 Optimizing 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).

4.3.1 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.

4.3.2 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.

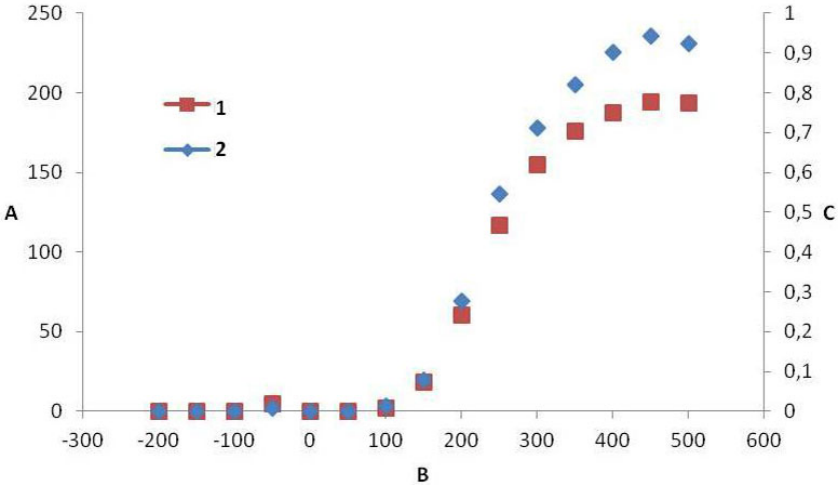

Fig. 3: Example of an HDV for 3.4 dihydroxybenzylamine

Letter	Description
A	Peak height of DHBA, nA
B	Applied potential, mV
C	Peak area of DHBA, nC

No.	Description
1	Peak height
2	Peak area

The information contained in an HDV is used in selecting the optimal applied potential to the working electrode for the detection of an analyte under a given set of chromatographic conditions.

A primary goal of an HDV is to determine the lowest practical applied potential for detection of your analyte of interest.

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

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

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

4.4 Optimizing Cell Performance

4.4.1 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.

4.4.2 Optimization Key Factors

The performance of the cell can be optimized by careful consideration of the following key factors.

Optimization aspect	What to consider
Air bubbles	The coulometric cell has been specifically designed to overcome problems with air bubbles. If air bubbles are trapped in the cell pathways nevertheless, attach a filter or tubing with narrow bore to the waste line to increase the backpressure. The increased pressure may dissolve or prevent air bubbles from occurring in the cell.

Optimization aspect	What to consider
Air bubbles *(continued)*	Air bubbles trapped in the cell is a potential source of noise. Usually, this noise manifests itself as *spikes*. To further prevent air bubbles from forming in the cell, it is recommended that the mobile phase be degassed off-line prior to installation on the system.
Grounding	Make sure that all of the system modules and components are well grounded to a central grounding location to avoid "ground loops" which may form and can lead to increased baseline noise and other baseline artifacts. An electrician may need to be consulted to check on the condition of the electrical grounds. Grounding various components of the system (for example pump, columns, autosampler etc.) may result in reduced baseline noise.
Mobile phase	When changing mobile phase, always flush the cell with the new mobile phase to ensure a stable reference potential. If using mobile phases that are prone to microbial growth (low organic content), the cell should be flushed periodically to waste to remove any possible contamination.
Potential	Due to the nature of analytes of interest, and small variations in working electrode perfromance, the optimum detection potential should be determined experimentally. This can best be achieved by constructing an hydrodynammic voltammogram (HDV) curve for the analyte of insterest. After a potential has been applied to the cell, the current will reach a peak, then rapidly decay at first and then slowly decay before finally settling to a value that changes little with time. The rapid current decay is predominantly due to the charging current, whereas the slower decaying current is associated with the equilibration of the cell.

Optimization aspect	What to consider
Pump flow	• Do not allow the flow to the coulometric cell to be interrupted when a potential is turned on. This may foul the working electrode or possibly lead to permanent damage of the cell. • Also, do not allow the cell to become dry while potentials are applied to it. Always turn off the potential to the cell (cells on/off functionality in the chromatography data system) when working on the cell. • Always make sure that there is enough mobile phase to last for the intended analysis or to last overnight, etc. • The use of low flow rates or recycling of the mobile phase may be used during these times.

5 Maintenance and Service

5.1 General Notes and Safety Precautions

The following sections describe the routine maintenance and service and repair procedures 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).

Important: Coulometric cells do not contain servicable items and are not designed for disassembly, as this renders the cell unusable. Do not attempt to disassemble any coulometric cell.

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

- For all service and repair procedures, observe all precautionary statements provided in the *Operating Instructions* for the detector.
- Use only the original spare parts and accessories authorized for the device by Thermo Fisher Scientific.

5.2 Routine and Preventive Maintenance

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

Frequency	What you should do...
Daily	When buffer solutions are used, flush the system thoroughly after use. Use a solvent that does not contain buffers or salts.
Regularly	Replace the graphite and PEEK filter elements of the in-line filters at least on a quarterly basis. For replacement instructions, refer to the *Operating Instructions* for the detector.

5.3 Restoring Cell Performance

Restoring the performance of the coulometric cell may be required if you observe one or more of the following symptoms:

- Gradual loss in cell performance
- Tailing peaks
- Increase in background current and/or increase in baseline noise
- A cell does not produce the expected response or is no longer useable
- Observed increase in system operating pressure over time

Tip: If a sample analysis is critical, it may help to have a replacement cell available before starting the analysis.

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

If the effect is isolated to the cell, the loss of response can be a result of:

- Contamination of the electrode(s)
- Shift in the hydrodynamic voltammogram (HDV)

  Tip: In many cases, the first two effects occur simultaneously.

- Age of the cell or working electrode
- Physical damage to the working electrode

- Deposit of eluent contaminents or sample compounds in the cell

  Occasionally, eluent or sample compounds may deposit in the cell or on the electrodes. As a result, they increase the level of the baseline noise and adversely affect the response. In many cases, cleaning the cell may improve the performance of the cell.

- Clogging of porous graphite electrodes

Procedures for the coulometric cell

Perform the following procedures in the given order. If a procedure could not recover the cell response, try with the next procedure:

1. Electrochemically Treating a Coulometric Cell (→ section 5.3.1, page 31)
2. Back-Flushing a Coulometric Cell (→ section 5.3.2, page 33)
3. Cleaning a Coulometric Cell (→ section 5.3.3, page 35)
4. Flushing the Cell with Strong Acid Solution (→ section 5.3.4, page 37)

If none of the above procedures could restore the cell performance, replace the coulometric cell.

5.3.1 Electrochemically Treating a Coulometric Cell

The electrochemical treatment can minimize small variations in HDV characteristics between cells by shifting the HDV curve back to its initial position.

Verifying if a shift in the HDV is present

Set the potential to +300 mV higher than normally used for a known analysis. If the response is restored, the problem is probably a shift in the HDV.

Electrochemically treat the working electrodes

To electrochemically treat the porous-graphite working electrodes and sharpen the hydrodynamic voltammogram (HDV):

1. Turn off (the potential to) the cell.
2. Stop the pump flow to the cell.
3. Make sure that the cell is installed to the appropriate potentiostat module in the detector.
4. Replace the mobile phase with fresh mobile phase. The mobile phase should be of low organic solvent composition (less than 15%) flowing at about 1.0 mL/min. Do not recycle the mobile phase during the treatment.
5. Start the pump flow. Apply a potential of +1000 mV to the cell for up to 10 minutes with mobile phase flowing.
6. After the treatment, return the detector to the initial operating conditions.
   a) Reset the potential to the potential for the analysis.

b) Establish a stable baseline and test the response. Perform an equilibration with the cell (→ *Equilibrating the Cell* section earlier in this guide).

   If there is no observed improvement to the response, perform an additional pre-treatment step by applying a negative potential. The resulting high magnitude current (either positive or negative) can remove unwanted materials on the electrode.

7. *If there is still no observed improvement to the response*

   In addition to the above treatment, try a combination of potentials:

   a) With fresh mobile phase flowing, set the potential in the range of -350 mV to -450 mV for 10 minutes.

   b) Set the potential to +1000 mV for additional 10-15 minutes.

Tip: Under some conditions, this procedure may provide only minimal improvement in response, or may deteriorate the cell performance even more.

If you could not restore the response with the above procedure and/or are still observing a high backpressure, back-flush the coulometric cell (→ section 5.3.2, page 33).

5.3.2 Back-Flushing a Coulometric Cell

Occasionally, a coulometric cell will exhibit normal performance but with a unusually high backpressure. You may use the cell even with the increased backpressure if it exhibits normal analytical behavior, as long as the system pressure does not exceed safe limits.

⚠ **Important:** If the following procedure is performed at a flow rate outside the safe pressure limits, damage to coulometric cell may occur. To avoid damage to the cell, perform the back-flushing procedure only at a reduced flow rate.

To reduce the backpressure in the coulometric cell, perform the following back-flushing procedure with the cell:

1. Turn off (the potential to) the cell.
2. Stop the pump flow.
3. Reverse the flow connection on the coulometric cell by inverting the capillaries on the inlet and outlet of the cell. Ensure that the post-column PEEK in-line filter remains connected to the incoming flow 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.

4. Turn on the pump flow through the coulometric cell at a flow rate of 0.25 mL/min for 10 minutes.

5. Stop the pump flow.

6. Return the flow connections on the coulometric cell to its correct orientation. Return the cell to operating conditions. Evaluate the result of the back-flushing procedure.

If the backpressure remains high, clean the coulometric cell (→ section 5.3.3, page 35).

5.3.3 Cleaning a Coulometric Cell

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

Tip: Cleaning the cells can have an additional benefit: The cleaning procedure may recover the response from contaminated and/or fouled electrodes.

Tools and additional items required

- Protective gloves
- HPLC-grade water, methanol, acetonitrile, acetone (each 100%)

Follow these steps

1. Turn off (the potential to) the cell.
2. Stop the pump flow to the cell.
3. Remove the analytical column from the flow path.
4. Remove any mobile phase from the coulometric cell: Flush the cell with 100% HPLC-grade water for 10 minutes at a flow rate of 1.0 mL/min.
5. Flush the coulometric cell at a flow rate of 1.0 mL/min with solvents in the following order for 10-15 minutes per solvent:
   a) 100% methanol
   b) 100% acetonitrile
   c) 100% acetone
   d) 100% acetonitrile
   e) 100% methanol
   f) 100% HPLC-grade water

6. Stop the pump flow to the cell.
7. Replace the PEEK filter element in the in-line filter (→ *Operating Instructions for the detector*).
8. Start the pump flow and flush the system with the application mobile phase for 10 minutes.
9. Turn on (the potential to) the cell at the working potentials of the application and test response while monitoring backpressure.
10. The next step depends
    - ♦ If the system pressure is not approaching unsafe limits and if the cell exhibits otherwise normal performance characteristics, the cell may be used with the higher backpressure.
    - ♦ If the backpressure of the cell remains unusually high and if it is adversely affecting the assay
      Flush the cell with strong acid (→ section 5.3.4, page 37).

5.3.4 Flushing the Cell with Strong Acid Solution

If electrochemically treating the working electrodes and cleaning the cell could not restore the cell performance and/or did not reduce the high backpressure, flush the cell with a strong acid solution.

Warning: The handling of the substances for this procedure can pose health and safety risks. Observe the safety precautions in the *Operating Instructions for the detector*. To avoid personal injury to skin and eyes, wear personal protective clothing and googles and follow good laboratory practice.

Important: Perform the following procedure only if electrochemically treating the working electrodes and cleaning the cell did not improve the cell performance.

Tools and additional items required

- Protective gloves and eyewear
- Phosphoric acid
- HPLC-grade water

Preparations

1. Turn off (the potential to) the cell.
2. Stop the pump flow to the cell.
3. Wear the protective gloves and eyewear.

4. Prepare a solution of 60% phosphoric acid: Mix 200 mL HPLC-grade water and 500 mL of 85% phosphoric acid.

Warning: The mixing of water and phosphoric acid can cause a dangerous chemical reaction and lead to personal injury. To avoid this, only carefully add the phosphoric acid to the water.

Follow these steps

1. Flush the coulometric cell with HPLC-grade water for at least 10 minutes at a flow rate of 0.5 mL/min.
2. Flush the cell with the solution of 60% phosphoric acid for 15 to 60 minutes at a flow rate of 0.5 mL/min.
3. Flush the cell with HPLC-grade water until the liquid leaving the cell is pH neutral (with a pH value of approximately 7.0).

If you could not restore the cell performance, replace the coulometric cell (→ section 5.5, page 42).

5.4 Shutdown and Storage

5.4.1 Guidelines for Cell Shutdown

Observe the following precautions before interrupting operation of electrochemical cells:

- Turn off (the potential to) the cell before stopping the pump flow.
- Rinse out any solvents from the cell(s) before removing the cell from the detector.
- For longer periods, always store unused electrochemical cells in their original dust-free packaging.
- When interrupting cell operation, observe the following differentiation for the duration of storage:
  - *If the cell is to be stored for one week or less*
    Observe the steps for short-term storage. See section 5.4.2 below.
  - *If the cell is to be stored for more than one week*
    Observe the steps for long-term storage. See section 5.4.3, page 41.

5.4.2 Short-Term Storage (1 week or less)

Tools and additional items required

- Protective gloves (powder-free)
- HPLC-grade water, organic solvent (without buffer salts)
- Storage plugs and original cell packaging

Follow these steps

1. Wear the protective gloves.

2. Turn off (the potential to) the cell in the chromatography data system, such as Chromeleon.

3. Flush the cell with a mixture of HPLC-grade water and organic solvent that does not contain buffer salt additives for 5 minutes at a flow rate of 1.0 mL/min. Use the same percentage as the previously used mobile phase to remove all traces of buffer salts from the cell.

4. Flush the cell with organic solvent similar to the previously used mobile phase for 5 minutes at a flow rate of 1.0 mL/min to remove water and prevent microbial growth.

5. Stop the pump flow to the cell.

6. Without pump flow, proceed as required:

   ♦ Leave the cell installed on the detector, without flow.

   –or–

   ♦ Remove the cell from the detector:

   a) Disconnect the capillaries from the cell inlet and outlet.

   b) Carefully disconnect the cell connector from the connector of the potentiostat module.

   c) Install the storage plugs on the cell inlet and outlet. Use the plugs that were installed when the cell was shipped. Using different plugs and tightening them may damage the cell inlet and outlet.

   d) Store the cell in its original packaging.

   To restart operation with the cell, reinstall and configure the cell (→ section 3.1, page 9).

5.4.3 Long-Term Storage (more than 1 week)

Tools and additional items required

- Protective gloves (powder-free)
- HPLC-grade water, HPLC-grade methanol
- Storage plugs and original cell packaging

Follow these steps

1. Wear the protective gloves.
2. Turn off (the potential to) the cell in the chromatography data system, such as Chromeleon.
3. Flush the cell with a mixture of 20% HPLC-grade methanol and 80% HPLC-grade water that does not contain buffer salt additives for 5 minutes at a flow rate of 1.0 mL/min. Flush long enough to remove any trace of the application mobile phase from the cell.
4. Flush the cell with 100% HPLC-grade methanol for 5 minutes at a flow rate of 1.0 mL/min to remove water and prevent microbial growth.
5. Stop the pump flow to the cell.
6. Remove the cell. See section 5.5, page 42.
7. Install the storage plugs on the cell inlet and outlet. Use the plugs that were installed when the cell was shipped. Using different plugs and tightening them may damage the cell inlet and outlet.
8. Store the cell in its original packaging.

5.5 Removing the Cell

When

- For replacement of a cell
- For long-term storage of the cell

Tools and additional items required

Protective gloves (powder-free)

Preparations

1. Wear the protective gloves.
2. Turn off (the potential to) the cell.
3. Stop the pump flow to the cell.

Follow these steps

1. Disconnect the capillaries from the cell inlet and outlet.
2. Carefully disconnect the cell connector from the connector of the potentiostat module.
3. The next steps depend:
   - ♦ *If the cell is to be stored*
     Continue with the steps for long-term storage on page 41.
   - ♦ *If the cell is to be replaced*
     Install a new cell. See chapter 3, page 9.

6 Technical Information

Specification	6011RS ultra Coulometric Analytical Cell	6020RS omni Coulometric Cell
Cell design:	Flow-through, micro-volume, dual-series electrodes	Flow-through, micro-volume, single-channel electrode
Working electrode:	Micro-porous graphitic carbon	
Potential range:	-300 to +1100 mV (vs. Palladium)	
Flow rate:	> 0.3 mL/min	Up to 1.0 mL/min
	(optimum flow rate determined by application and mobile phase composition used)	
Operating pressure:	40 bar (580 psi, 4.0 MPa)	620 bar (8992 psi, 62 MPa)
Internal volume:	4.3 µL	5.7 µL
Flow connections:	Inlet/Outlet: 10-32 thread female port (compatible for nanoViper fingertight fitting)	
Parametric control:	Automatic parameter configuration through chromatography data system (such as Chromeleon) via SmartChip cell recognition. The SmartChip identifies and records sensor type and defines data collection.	
Wetted parts:	PEEK, porous graphite, palladium, PTFE	
Environmental conditions:	Operating temperature: 10-45 °C (50-113 °F) Air humidity: 18 to 80% relative humidity, non-condensing	
Solvent compatibility:	Compatible with typical reverse- and normal-phase compositions	

Specification	6011RS ultra Coulometric Analytical Cell	6020RS omni Coulometric Cell
Dimensions (h × w × d):	39 × 45 × 73 mm (1.5 × 1.8 × 2.9 in.)	
Weight:	Approx. 224 g (7.9 oz)	Approx. 245 g (8.6 oz)

Technical information: July 2016.
All technical specifications are subject to change without notice.

7 Consumables and Spare Parts

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

Description	Part no.
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
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
Capillary, Viper, I.D. x L 90 µm x 75 mm, PEEK, 2 capillaries For interconnecting adjacent cells for multi-cell operation.	6041.9075