

Dionex ASE 350 Accelerated Solvent Extractor Operator's Manual

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General Laboratory Equipment—Not for Diagnostic Use.

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1.1 Overview

The Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor (see [Figure 1-1](#)) is an automated system for extracting organic compounds from a variety of solid and semisolid samples. The Dionium™ internal pathway allows extraction of mildly acidic or basic samples.



Figure 1-1. Dionex ASE 350

The Dionex ASE 350 uses a combination of elevated temperature and pressure with common solvents to increase the efficiency of the extraction process. The result is faster run times and a significant reduction in solvent use. To further

reduce solvent use, the Dionex ASE 350 supports a unique extraction method called the solvent saver mode (see [Section 2.7.1](#)).

During a run, the cell is filled with solvent and then heated and pressurized to maintain the solvent in a liquid state. After the run, the extract is rinsed from the cell into a collection vessel (a bottle or vial). If adsorbents are used in the sample cell, post-extraction cleanup may not be required. If adsorbents are not used, sample cleanup may be required.

Control Options

The Dionex ASE 350 can be controlled *locally*, from the front panel keypad and display, or *remotely*, from a PC (personal computer) running:

- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (release 7.3 or later) on the Microsoft™ Windows™ 10 operating system
- or–
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (release 7.2.10) on the Microsoft™ Windows™ 8.1 operating system

With Chromeleon software, you can create and run methods and sequences that use the same extraction modes available from the Dionex ASE 350 front panel, as well as two additional modes (fixed volume and gradient) available only in the software (for details, see [Section 2.7.1](#)).

Communication between the Dionex ASE 350 and Chromeleon software requires a connection from the Dionex ASE 350 to a USB (Universal Serial Bus) port on the PC. Up to four Dionex ASE 350 systems can be automated by one Chromeleon software workstation.

1.2 About This Manual

1.2.1 Overview

The electronic version (*.pdf file) of the Dionex ASE 350 operator's manual contains numerous links that enable you to quickly move from one location to another within the file. These links include:

- Table of contents entries
- Index entries

- Cross-references (underlined in blue) to sections, figures, and tables

Chapter 1 Introduction	Introduces the Dionex ASE 350 system; explains the conventions used in this manual, including safety-related information.
Chapter 2 Description	Describes Dionex ASE 350 operating features and the extraction process.
Chapter 3 Operation and Maintenance	Provides operating instructions and routine preventive maintenance requirements.
Chapter 4 Troubleshooting	Lists error messages and explains how to troubleshoot them; lists operating problems and how to resolve them.
Chapter 5 Service	Provides step-by-step instructions for routine service and parts replacement procedures that users can perform.
Appendix A Specifications	Provides specifications and installation site requirements.
Appendix B Installation	Explains how to install the Dionex ASE 350.
Appendix C Diagnostic Screens	Illustrates and describes the Dionex ASE 350 front panel diagnostic screens.
Appendix D Reordering Information	Lists spare parts for the Dionex ASE 350.
Appendix E Theory of ASE	Describes the theory behind the ASE (accelerated solvent extraction) technique.

1.2.2 Safety Messages and Notes

This manual contains warnings and precautionary statements that, when properly followed, can prevent personal injury to the operator and/or damage to the Dionex ASE 350. Safety messages appear in bold type and are accompanied by icons, as shown here.



Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, may result in death or serious injury.



Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury.



Indicates that the function or process of the instrument may be impaired. Operation does not constitute a hazard.

Messages d'avertissement en français



Signale une situation de danger immédiat qui, si elle n'est pas évitée, entraînera des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures graves à mortelles.



Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures mineures à modérées. Également utilisé pour signaler une situation ou une pratique qui pourrait gravement endommager l'instrument mais qui n'entraînera pas de blessures.

Warnhinweise in Deutsch



Bedeutet unmittelbare Gefahr. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.



Bedeutet eine mögliche Gefährdung. Mißachtung kann zum Tod oder schwerwiegenden Verletzungen führen.



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

Informational messages also appear throughout this manual. These are labeled NOTE and are in bold type.

NOTE NOTES call attention to certain information. They alert the user to an unexpected result of an action, suggest how to optimize instrument performance, and so on.

1.3 Safety and Regulatory Information

The Dionex ASE 350 is manufactured by Thermo Finnigan LLC at the following location: 355 River Oaks Parkway, San Jose, CA 95134-1991 U.S.A. The Dionex ASE 350 is designed for solvent extraction applications and should not be used for any other purpose. Operation of a Dionex ASE 350 in a manner not specified by Thermo Fisher Scientific may result in personal injury.

If there is a question regarding appropriate usage, contact Technical Support for Dionex products:

- In the U.S. and Canada, call 1-800-532-4752 and select **option 2**.
- Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

1.4 Safety Labels

The cTUVus Mark safety label and the CE Mark label on the Dionex ASE 350 indicate that the system is in compliance with these standards.

The system is in conformity with the relevant Community harmonization legislation:

- Low Voltage Directive 2014/35/EU
- EMC Directive 2014/30/EU
- RoHS Directive 2011/65/EU

References to the relevant harmonized standards used or references to the specifications in which conformity is declared:

Safety Specifications

- EN 61010-1:2010

Dionex ASE 350 Solvent Extraction System

EMC Specifications

- EN 61326-1:2013

RoHS Specifications

- EN 50581:2012

The symbols below appear on the Dionex ASE 350 or on Dionex ASE 350 labels.

 Alternating current

 Primary protective conductor terminal

 Secondary protective conductor terminal

 Power supply is on

 Power supply is off

 Hot surface

 Indicates a potential hazard. Refer to this operator's manual for an explanation of the hazard and how to proceed.

2 • Description

- [Section 2.1](#) describes Dionex ASE 350 operating features and components.
- [Section 2.6](#) describes the extraction process.
- [Section 2.7](#) describes both method and sequence control of the Dionex ASE 350.

2.1 Operating Features

[Figure 2-1](#) illustrates some of the key operating features of the Dionex ASE 350.



Figure 2-1. Dionex ASE 350 Operating Features

Control Panel

The control panel on the front of the Dionex ASE 350 contains the liquid crystal display (LCD) and membrane keypad (see [Figure 2-2](#)) required for local control of Dionex ASE 350 operation.

Cell Tray and Sample Cells

The cell tray, on the upper right of the Dionex ASE 350, holds up to 24 sample cells of various sizes and types. The prepared sample is loaded into these cells.

Stainless steel cells are available in the following sizes: 1 mL, 5 mL, 10 mL, 22 mL, 34 mL, 66 mL, and 100 mL. Two cell sizes (66 mL and 100 mL) are available in zirconium, also.

Collection Tray and Collection Vessels

The collection tray, on the lower right of the Dionex ASE 350, holds the collection vessels (250-mL bottles or 60-mL vials). After a run, the collection vessels contain solvent and the analytes extracted from the sample.

Static Valve

The static valve, located above the collection tray, controls the flow of solvent from the sample cell.

Safety Shield

For the protection of the user, the entire run is performed behind a safety shield. A method, sequence, or rinse cannot be started unless the safety shield is closed. Closing the shield automatically locks it; the shield remains locked until the function in progress finishes (or is aborted).

Oven Area

The oven is housed at the rear of the Dionex ASE 350. This area also houses the AutoSeal arm. The arm moves the cell into and out of the oven, and seals the cell during a run.

Needle Mechanism

When positioned above a collection vessel, the needle mechanism pierces the collection vessel septum, allowing the extract to flow from the cell into the vessel.

Solvent Reservoir Tray

The solvent reservoir tray can hold up to three 2-liter reservoirs (P/N 045901). Connectors for solvent and nitrogen gas sources are located next to each reservoir.

Waste Bottle

A waste bottle is installed in a holder on the inside wall, to the left of the collection tray.

Service Area

The service area on the left side of the system contains the Dionex ASE 350 pump, pressure regulators, pneumatic valves and components, and electronics.

Users occasionally need access to the service area to perform routine maintenance procedures such as replacement of pump seals and check valve cartridges. To access the service area, rotate the three quarter-turn screws along the edge of the door and swing the door open.

2.1.1 Control Panel Display

The LCD (or *screen*) displays Dionex ASE 350 status and operating information. Fields that are in reverse video (white letters on a blue background) can be edited by the user. Normal video fields are read-only (the information displayed cannot be edited).

Screen Contrast: To adjust the screen contrast, press the **Contrast** Δ and **Contrast** ∇ buttons on the control panel keypad.

Screen Saver Mode: If there is no input to the Dionex ASE 350 for two hours (that is, no control panel buttons are pressed during this time), the LCD backlight automatically turns off. To turn on the backlight again, press any keypad button.

When the Dionex ASE 350 power is turned on, the **POWER-UP** screen appears (see [Figure 2-2](#)). The screen identifies the Dionex ASE 350 serial number and the Dionex ASE 350 firmware version.

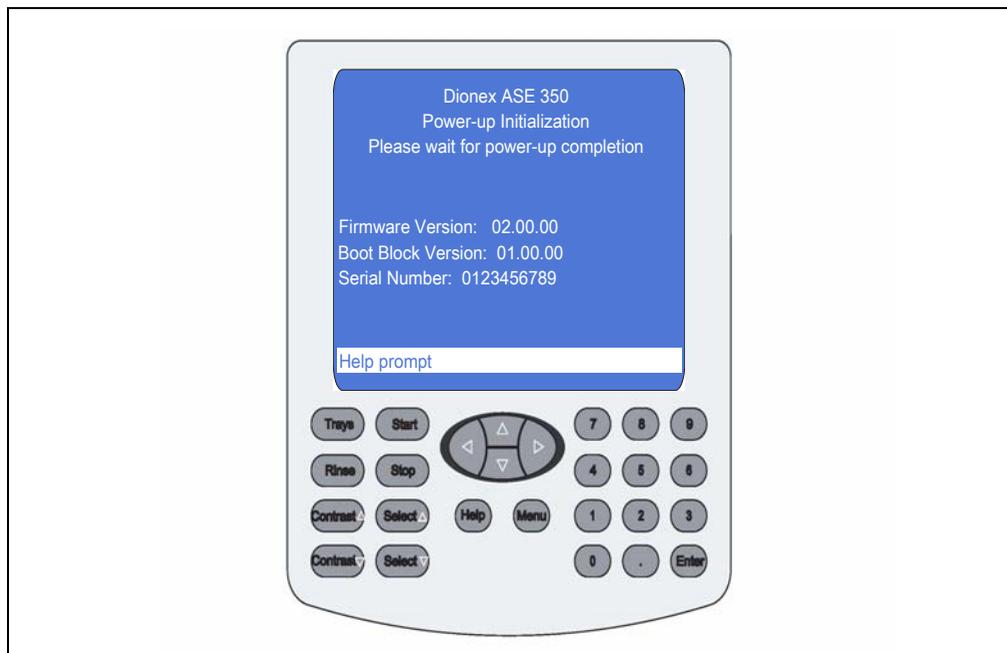


Figure 2-2. Dionex ASE 350 Control Panel

After a few seconds, the **POWER-UP** screen is automatically replaced by the **MAIN MENU OF SCREENS** (see [Figure 2-3](#)).

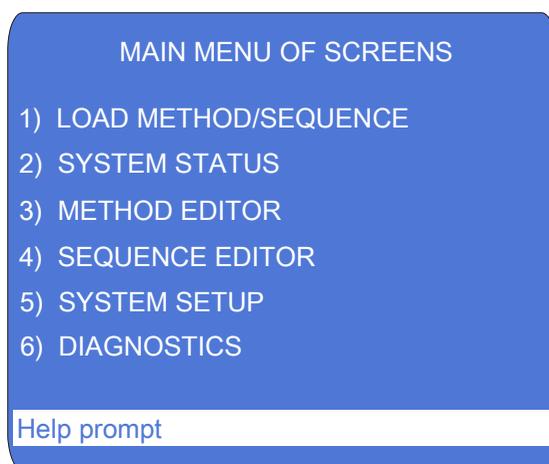


Figure 2-3. Main Menu of Screens

The **MAIN MENU OF SCREENS** provides six options: five screens for the control of Dionex ASE 350 operation, plus a **DIAGNOSTIC MENU** screen. There are two ways to select a menu option:

- Press the numeric button that corresponds to the screen name.
- or–
- Press an up or down arrow button to move the cursor to the screen name, and then press the **Enter** button.

2.1.2 Control Panel Keypad

Pressing a keypad button either directly affects Dionex ASE 350 operation or affects a screen function. [Table 2-1](#) summarizes the button functions.

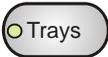
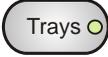
Button	Function
 (Free Spin)	Pressing the Trays button toggles the cell and collection trays between the <i>engaged</i> and <i>free spin</i> modes. LEDs on the button indicate the current mode. When the left LED is lighted (free spin mode), the collection tray and cell tray can be rotated manually. Select free spin mode to load and unload collection vessels and cells.
 (Engaged)	When the right LED is lighted (engaged mode), the tray drive mechanisms are engaged and the trays cannot be moved manually. Toggling from free spin to engaged mode rotates the trays to the home position. Starting a run automatically engages the trays. During a run, the Trays button is disabled.
	Starts a manual rinse, which is used to prime the pump or to rinse after a solvent change, after refilling a solvent reservoir, and so on. The trays rotate to the nearest rinse bottle and rinse tube. A user-specified volume of solvent is then pumped through the system and into the rinse bottle. Unless the Dionex ASE 350 is idle, the button is disabled.
	Starts the currently loaded method or sequence. When a run is in progress, the LED on the right side of the button is lighted.

Table 2-1. Dionex ASE 350 Front Panel Button Functions

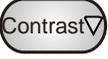
Button	Function
	Interrupts the current run. The pump turns off, valves close, and all flow stops. The screen displays a list of options (see Section 3.5.3) for selection.
	Pressing Contrast Δ increases the screen contrast; pressing Contrast ∇ decreases the screen contrast.
	
	The Select buttons step through predetermined options in entry fields on the screen. To confirm the selected value, press Enter (or move the cursor out of the field by pressing an arrow button). In fields with predetermined numeric choices, Select Δ increases the value by one unit and Select ∇ decreases the value by one unit. Holding down a Select button increases (or decreases) the value continuously.
	
	The arrow buttons move the cursor in the direction of the arrow to the next entry field on the screen (if one exists). After entering a new value in an entry field, pressing an arrow button to move the cursor to another field saves the change.
	Displays a context-sensitive help screen.

Table 2-1. Dionex ASE 350 Front Panel Button Functions (Continued)

Button	Function
	<p>Displays a list of screens that can be selected for display. There are two ways to select menu options:</p> <ul style="list-style-type: none"> • Press the numeric button that corresponds to the screen name. <p>–or–</p> <ul style="list-style-type: none"> • Press an up or down arrow button to move the cursor to the screen name, and then press Enter. <p>When an operational screen is displayed, pressing Menu returns you to the MAIN MENU OF SCREENS. When a diagnostic screen is displayed, pressing Menu returns you to the DIAGNOSTIC MENU. When the DIAGNOSTIC MENU is displayed, pressing Menu returns you to the MAIN MENU OF SCREENS.</p>
	<p>When the cursor is in an entry field, pressing a numeric button enters the selected number into the current entry field. When a menu screen is displayed, pressing a numeric button opens the corresponding menu option.</p>
	<p>Saves changes made in entry fields. When a menu screen is displayed, pressing Enter opens the highlighted screen.</p>

Table 2-1. Dionex ASE 350 Front Panel Button Functions (Continued)

2.1.3 Sample Cells, Rinse Tubes, and Cell Tray

NOTE [Appendix D](#) contains part numbers for cells, collection vessels, and other Dionex ASE 350 accessories.

Sample Cells

Stainless steel cells are available in the following sizes: 1 mL, 5 mL, 10 mL, 22 mL, 34 mL, 66 mL, and 100 mL. Two cell sizes (66 mL and 100 mL) are available in zirconium, also.

Both stainless steel and zirconium cells are suitable for extraction with solvents. Zirconium cells can also be used for the extraction of mildly basic or acidic matrices. For details about acid and base usage with the Dionex ASE 350, see [“Guidelines for Selecting and Preparing Solvents” on page 50](#).

A sample cell consists of a cell body and two interchangeable caps which screw onto the ends of the cell body. The cell body and end caps are made of the same material.

- Inside each cell end cap is a frit in the same material as the cell, as well as a PEEK[™] (polyether ether ketone) seal. During a run, the cell end caps are compressed to form a tight seal between the end caps and the cell body.

IMPORTANT

Always tighten the cell end caps by hand. Use of a wrench or other tool can damage the cell, as well as the seals inside the end caps, and will void the product warranty.

- Each cell end cap contains an external O-ring. White PTFE (polytetrafluoroethylene) O-rings (P/N 049457, pkg. of 50) are standard. Use Viton[™] O-rings (P/N 056325, pkg. of 50) for dioxins and other high temperature applications.

IMPORTANT

If the external O-ring is Viton, do not use acetone or other ketones.

Rinse Tubes

Rinse tubes (P/N 068074) are small-diameter metal tubes (see [Figure 2-4](#)) that fit into the two designated rinse positions in the cell tray (between positions 6 and 7 and positions 18 and 19). During a rinse cycle, the tray rotates to the nearest rinse position. Solvent passes directly through the rinse tube during the cycle.



Figure 2-4. Rinse Tube

- For details about manual rinse cycles, see [Section 3.1.9](#).
- For details about automatic rinsing between samples, see [Section 3.1.10](#).

Cell Tray

The cell tray can accommodate up to 24 sample cells of any size, plus two rinse tubes. If you plan to run under *method* control, you can load cells on the tray in any order. To run under *sequence* control, make sure the cell position matches the position specified in the sequence. During a run, the Dionex ASE 350 SmartRun™ feature will sense the cell size in each tray position.



To avoid personal injury, exercise caution whenever the tray is in motion.



Pour éviter de vous blesser, faites bien attention lorsque le plateau est en mouvement.



Um Verletzungen vorzubeugen, seien Sie vorsichtig, wenn sich der Probensteller bewegt.

2.1.4 Collection Vessels, Rinse Bottles, and Collection Tray

NOTE [Appendix D](#) contains part numbers for cells, collection vessels, and other Dionex ASE 350 accessories.

Collection Vessels

Two types of collection vessels can be installed in the Dionex ASE 350: bottles and vials. All collection vessels have a cap that contains a solvent-resistant septum. During a run, the needle mechanism pierces the septum, creating a liquid flow path from the sample cell to the collection vessel.

The following collection vessels are available:

Collection Vessel	Part Number	Quantity
Clear vials, 60 mL	048784	Pkg. of 72
Amber vials, 60 mL*	048781	Pkg of 72
Clear bottles, 250 mL	056284	Pkg. of 12

*Thermo Fisher Scientific recommends amber vials for light-sensitive samples.



Before each run, carefully inspect all collection vessels for chips, scratches, or cracks. If a collection vessel shows any sign of damage, do not use it. Use each collection vessel once only.



La présence de fêlure ou d'égratignures sur les flacons de collecte doit être vérifiée avant chaque extraction. N'utilisez jamais un flacon endommagé. Les flacons sont à usage unique.



Untersuchen Sie vor jedem Lauf alle Sammelgefäße auf Abplatzungen, Kratzer oder Risse. Wenn ein Sammelgefäß eine Beschädigung aufweist, sollten Sie es nicht mehr verwenden. Verwenden Sie nicht graduierte Sammelgefäß nur ein Mal.



Use each collection vessel cap and septum once only. This prevents solvent leaks caused by piercing the septum in the cap multiple times.

Rinse Bottles

The rinse positions (labeled R1 and R2) each accommodate one collection bottle. Before the start of the rinse cycle, the cell tray rotates to the nearest rinse position and bottle sensors check that the rinse bottle is present and is not full. Solvent is then pumped through the system and collected in the rinse bottle.

If the first rinse bottle is either missing or full, a sensor will check the status of the next rinse bottle. If both rinse bottles are missing or full, an error message is displayed on the control panel screen.

Collection Tray and Vial Tray Insert

The collection tray capacity varies, depending on whether a vial tray insert (P/N 066176) is installed. By itself, the collection tray can hold up to 14 collection bottles on the outside row, plus five collection bottles and two rinse bottles on the inside row.

The vial tray insert (see [Figure 2-5](#)) is required to accommodate collection vials. The vial tray insert resizes the holes in the collection tray to ensure that the vials, which are smaller than the collection bottles, are secured in place.



Figure 2-5. Vial Tray Insert

When the vial tray insert is in place, the collection tray can hold up to 28 60-mL collection vials on the outside row, plus five 250-mL collection bottles and two rinse bottles on the inside row.

A vial tray insert is provided in both the Small Volume Startup Kit (P/N 068254) and the 34-mL Volume Startup Kit (P/N 068255).



To avoid personal injury, exercise caution whenever the tray is in motion.



Pour éviter de vous blesser, faites bien attention lorsque le plateau est en mouvement.



Um Verletzungen vorzubeugen, seien Sie vorsichtig, wenn sich der Probensteller bewegt.

2.1.5 Solvent Reservoir Tray

The Dionex ASE 350 Ship Kit (P/N 066060) includes one 2-liter glass reservoir with shatterproof plastic coating (P/N 045901) and one bottle cap assembly (P/N 068077) with the tubing and fittings needed to connect the reservoir to the Dionex ASE 350.

Up to two additional solvent reservoirs can be installed. When more than one reservoir is installed, the Dionex ASE 350 can do the following:

- Change solvents between runs. This enables you to extract the same sample with a different solvent or to extract each remaining sample with a solvent other than the one used for the previous sample.
- Select from up to three different solvent reservoirs for extractions.
- Mix two or three solvents, at a user-selected ratio, during the run.

2.1.6 Waste Bottle

System waste is collected in a 250-mL collection bottle (without the bottle cap). The waste bottle sits in a holder located to the left of the collection tray. Three vent lines, one from the pressure relief valve and two from the needle mechanism, are connected to the top of the waste bottle holder. The waste bottle collects the small amounts of solvent vented through the three lines.

A vent outlet line is also connected to the waste bottle holder. Gas is vented out this line to the rear panel, from where it is routed to a fume hood. The Dionex ASE 350 Ship Kit (P/N 066060) includes 3.05 meters (10 ft) of external vent tubing (P/N 053514) for this purpose.

NOTE Check the waste bottle daily and empty it whenever necessary.

2.1.7 Heat Exchanger (Optional)

A heat exchanger (P/N 068247) is required for applications that use volatile solvents at temperatures above 175 °C. The purpose of the heat exchanger is to cool the solvent as it leaves the cell, before it enters the static valve and is delivered to the collection vessel. Use of a heat exchanger minimizes the loss of solvent and analytes during collection. The heat exchanger must be installed by qualified Thermo Fisher Scientific personnel.

2.1.8 Service Area

The service area on the left side of the Dionex ASE 350 (see [Figure 2-1](#)) houses the pump, pressure regulators, pneumatic valves and components, and electronics.

Users occasionally need access to the service area to perform routine maintenance procedures such as replacement of pump seals and check valves. To access the service area, rotate the three quarter-turn screws along the edge of the door and swing the door open.



Always close the door to the service area before starting a run. If the door is open during operation, the oven may not reach and maintain the set temperature.



Do not adjust the pressure regulators inside the service area. If you think the regulators require adjustment, contact Technical Support for Dionex products.



Do not remove any of the electronics boards from the service area. There are no user-serviceable components on the boards. If servicing is required, it must be performed by qualified Thermo Fisher Scientific personnel and appropriate electrostatic discharge (ESD) handling procedures must be followed.



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



Halten Sie sich von der Elektronik des Dionex ASE 350 fern. Die Elektronik kann nicht vom Anwender gewartet werden. Falls ein Service erforderlich ist, ist dieser von qualifiziertem Personal durchzuführen. Dabei müssen die geeigneten Verfahren zur elektrostatischen Entladung (ESD) eingehalten werden.

2.2 Right-Side Panel

[Figure 2-6](#) illustrates the Dionex ASE 350 right-side panel.

- The **POWER** switch provides on/off control of the main power for the system. The power receptacle also includes a fuse drawer containing two IEC127 fast-blow fuses (P/N 954746) rated at 10 amps. For instructions on how to replace the fuses, see [Section 5.10](#).

NOTE The top plate and oven heaters are automatically turned off after four hours of system inactivity (that is, if no control panel buttons are pressed and no methods or sequences run during a four-hour period).

- The model data label lists fuse and power information, as well as the Dionex ASE 350 serial number. You will be asked to provide the serial number when ordering replacement parts for the Dionex ASE 350.
- The voltage configuration label identifies the voltage for which the module is configured. Each Dionex ASE 350 is configured at the factory for either 120 VAC or 240 VAC. If the voltage from your power source does not match the voltage indicated on the label, contact Technical Support for Dionex products.

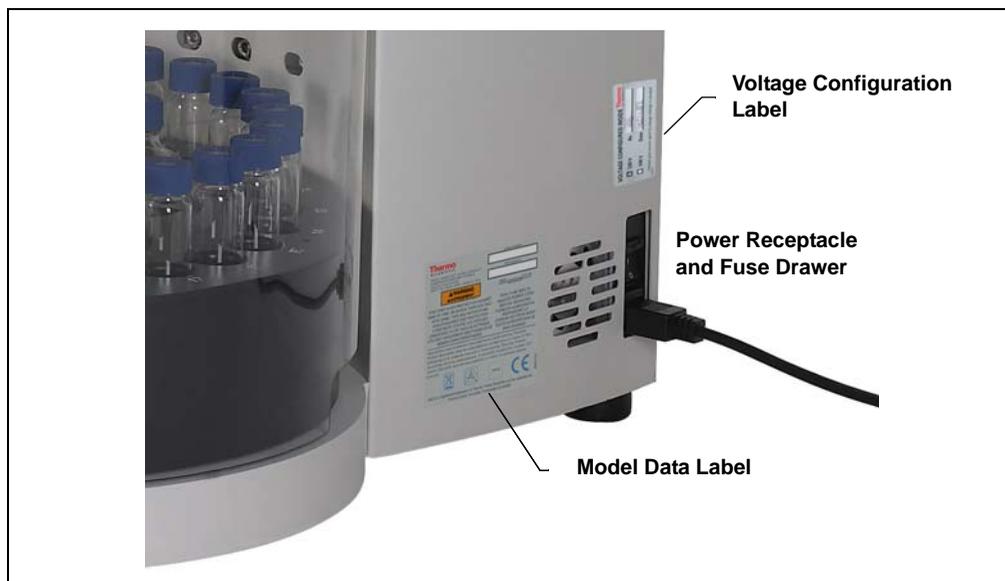


Figure 2-6. Dionex ASE 350 Right-Side Panel

2.3 Rear Panel

[Figure 2-7](#) illustrates the lower-right corner of the Dionex ASE 350 rear panel.

- The **AIR** connector provides a connection to a source of compressed air. For installation instructions, see [Section B.3](#).
 - If the air source does not contain VOCs or oil, regulate the pressure to between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.
 - If the air source is suspected of containing VOCs or oil, install an inlet filter assembly (P/N 049492). When the filter is installed, do not exceed 0.86 MPa (125 psi); 0.69 MPa (100 psi) is recommended.
- The **NITROGEN** connector provides a connection to a nitrogen gas supply regulated to between 0.97 and 1.38 MPa (140 to 200 psi). For installation instructions, see [Section B.3](#).

- The **VENT** connector provides a connection for the vent outlet line. For installation instructions, see [Section B.3](#).



Do not obstruct or pressurize the vent outlet.



Ne bouchez ni ne mettez sous pression la sortie d'aération.



Halten Sie den Lüftungsauslaß frei, setzen Sie ihn nicht unter Druck.

- The USB receptacle provides a connection to the computer on which Chromeleon software is installed. Connect a USB cable (P/N 960777) between this receptacle and a USB port on the computer. For USB connection instructions, see [Section B.4](#).

Note on Dionex ASE 350 Firmware Upgrades

When Chromeleon software is installed, use the Instrument Configuration Manager to download new firmware versions. For instructions, refer to the Chromeleon software Help.

If you do not have access to Chromeleon software, connect the USB receptacle to any available computer, and then download new firmware to the Dionex ASE 350. Installation instructions are provided with the firmware upgrade.

- The drip tray drain port removes any liquid that collects in the drip tray inside the Dionex ASE 350. A clear corrugated drain hose (P/N 055075) is connected to the port during installation (see [Section B.3](#)).

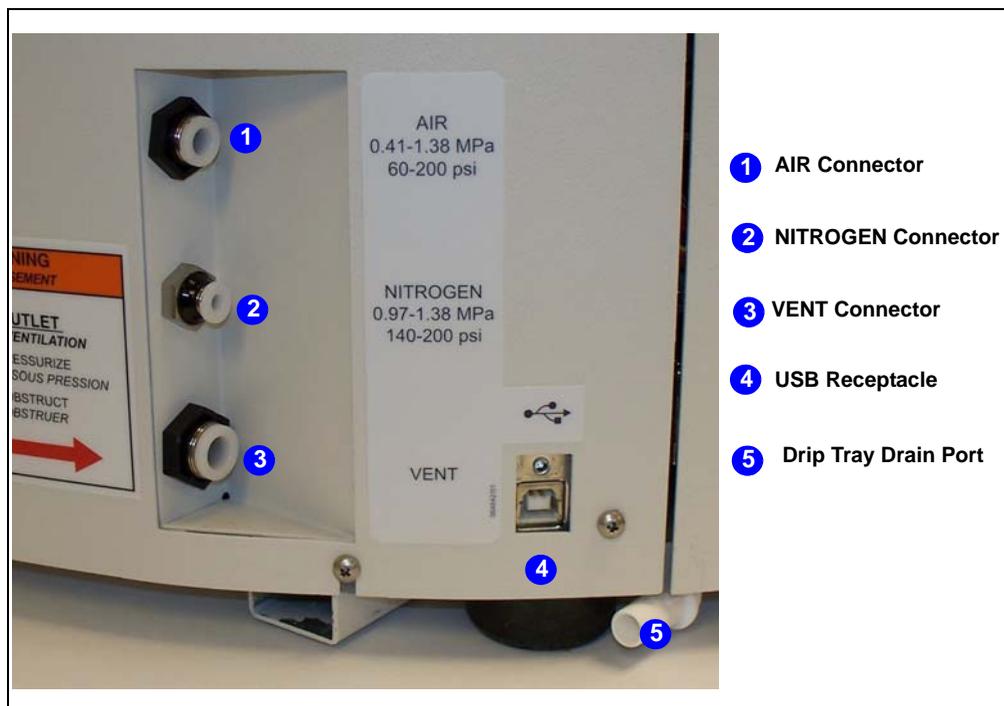


Figure 2-7. Dionex ASE 350 Rear Panel

2.4 Dionex ASE 350 Control

The Dionex ASE 350 can be controlled *locally*, from the front panel keypad and display (see [Section 2.4.1](#)), or *remotely*, with a personal computer on which Chromeleon software is installed (see [Section 2.4.2](#)).

2.4.1 Local Control

In Local mode, the Dionex ASE 350 receives commands from the front panel buttons and screens. When the Dionex ASE 350 is powered up, it is always in Local mode.

Note that the fixed volume and gradient extraction modes (see [Section 2.7.1](#)) are not available in Local mode. Access to these options requires Chromeleon software.

2.4.2 Remote Control

In Remote mode, Chromeleon software sends commands from the host computer to the Dionex ASE 350 via the USB interface. Connecting the Dionex ASE 350 to a Chromeleon ePanel (see [Figure 2-8](#)) immediately selects Remote mode. (An ePanel is the window from which you monitor and control Dionex ASE 350 operation. ePanels are displayed in the Console in the **Instruments** view.)

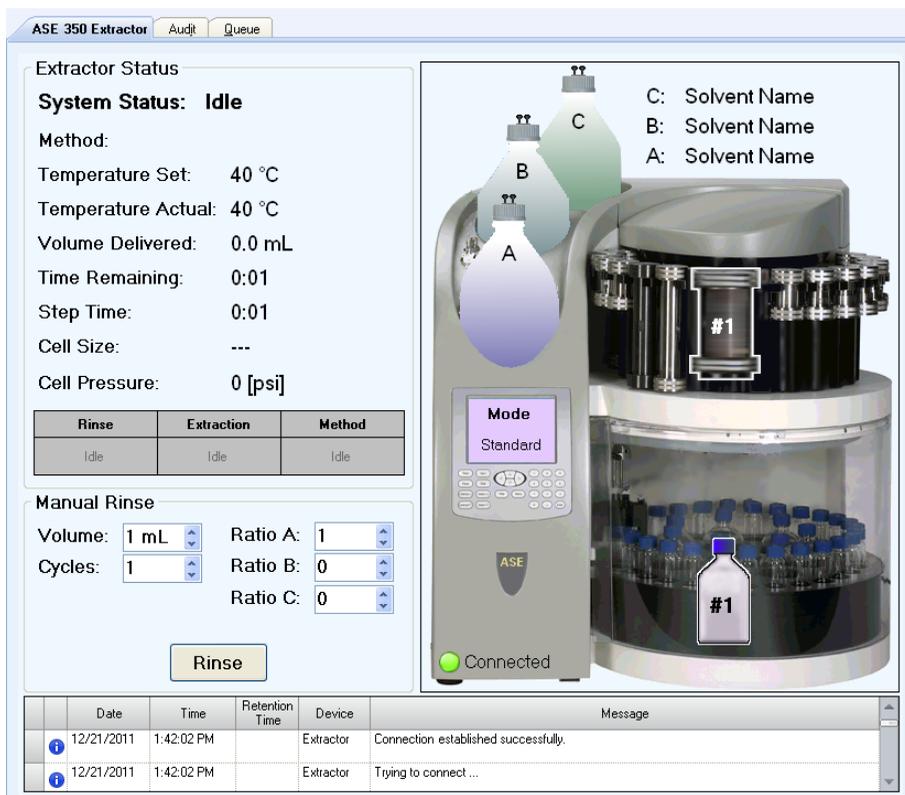


Figure 2-8. Chromeleon Software: Example Dionex ASE 350 ePanel

In Remote mode, most Dionex ASE 350 front panel functions are disabled in order to prevent inadvertent changes to operating parameters. However, you can still navigate the front panel screens, clear errors on the **ERROR LOG** screen (see [Section C.5](#)), and use the following buttons:

- **Trays** (manually rotates the collection tray and cell tray to enable loading and unloading of collection vessels and cells)

- **Stop** (stops the current run)
- **Contrast** Δ and **Contrast** ∇ (adjusts the screen contrast)

To return the Dionex ASE 350 to Local mode:

- Click the **Connected** button on the ePanel. This disconnects the system from Chromeleon software (and changes the button label to **Disconnected**).
- or–
- Turn off the Dionex ASE 350 power briefly.

2.5 Chromeleon Software

The Dionex ASE 350 can be controlled by a PC on which Chromeleon Chromatography Data System (release 7.1 or later) is installed. For complete information, refer to the Chromeleon software Help or to the manuals provided on the Chromeleon software DVD.

NOTE Support for Chromeleon software requires Dionex ASE 350 firmware version 2.00.00 (or later).

2.5.1 Chromeleon Software Quick Start

Configuring the Dionex ASE 350

1. Open the Chromeleon Services Manager and click **Start Instrument Controller**.
2. Open the Instrument Configuration Manager and click the **Connect** toolbar button to connect to Chromeleon software.
3. To create a new instrument, click the **Add Instrument** toolbar button.
4. Enter a name for the new instrument.
5. Right-click the name of the instrument in the Instrument Configuration Manager, and then click **Add Module**. The **Add module to instrument** dialog box appears.
6. In the **Manufacturers** list under **Dionex**, select **Extraction Modules**. In the **Modules** list, select **ASE 350 Accelerated Solvent Extractor**. Click **OK**. The **Properties** dialog box appears.

7. On the **General** tab page, specify the following options:
 - **Device Name:** The name used to identify the Dionex ASE 350 in Chromeleon software.
 - **Mode:** Select **Live** mode when configuring a Dionex ASE 350 that is physically connected to the Instrument Controller PC. Note that the Dionex ASE 350 must be running firmware version 2.00.00 (or later).

If a firmware update is required, select the serial number of the Dionex ASE 350 being connected in the **Module Serial No.** list, click the **Download** button, and select the appropriate firmware file.

Do not select **Simulation** mode unless you are configuring the Dionex ASE 350 in order to demonstrate the software.

8. On the **Options** tab page, specify the following options:
 - Select the pressure units to be displayed in Chromeleon software (psi, MPa, bar, or atm).
 - If the vial tray insert is installed in the collection tray, select the **Vial Tray Insert** check box.
 - Enter names for the solvents in positions A, B, and C of the reservoir tray.

Using Chromeleon Software to Control the Dionex ASE 350

When you start Chromeleon software, the Chromeleon Console appears. The Console displays an ePanel with a graphic of the Dionex ASE 350, as well as an overview of the system status (see [Figure 2-8](#)).

Use the **Manual Rinse** controls below the status display to specify the rinse volume, number of rinse cycles, and solvent ratio for a manual rinse. To start the rinse, click the **Rinse** button.

Creating Instrument Methods

1. In the Chromeleon Console, on the **Create** menu, click **Instrument Method** to open the Instrument Method Wizard.
2. Use the wizard to program the following method options:
 - **Rinse Settings** page: Specify the volume and solvent ratio to be used during a rinse. The rinse will occur in between extraction cells on a rinse tube to rinse the solvent lines after each extraction.
 - **Extraction Method** page: Specify the extraction conditions for the extraction cell.
 - **Completion** page: Add a comment (optional) and click the **Finish** button. This opens the method in the Instrument Method Editor.
3. Review the settings for **Comment**, **Name**, **Description**, and **Run Time**; make any changes required.

NOTE The Run Time setting must remain at 0.000 minutes.

4. Save the method before exiting the Instrument Method Editor.

Creating Sequences

1. In the Chromeleon Console, on the **Create** menu, click **Sequence** to open the New Sequence Wizard.
2. Select the Dionex ASE 350 for which the sequence is being created.
3. Use the wizard to program the following sequence options:
 - **Unknown Injections** page: Specify the extraction name pattern, starting cell position, starting vial position, and number of extractions.
 - **Methods & Reporting** page: Specify the extraction method to be used in the sequence and the default report template used when reporting results for each extraction.
 - **General Sequence Settings** page: Add a comment (optional) and click the **Finish** button.

- **Save Sequence** dialog box: Assign an object name to the sequence and click **Save**. The Chromeleon Console (**Data** view) automatically appears, displaying the newly created sequence.
4. To start the sequence, click the **Start** button on the Sequence Control Bar (above the extraction list).

2.5.2 Overview of Chromeleon Software Features

This section contains two tables:

- The first table lists the most frequently used Dionex ASE 350-related functions and parameters in Chromeleon software and explains where to find them. Unless otherwise specified, these features are in the Chromeleon software client.
- The second table contains navigation instructions for key elements of the Chromeleon software user interface.

Chromeleon Software Feature	Location of Feature
Audit trail	Audit ePanel
Bypass heatup	System Setup page (Instrument Method Editor)
Cell type	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Current conditions	Dionex ASE 350 Extractor ePanel
Extraction modes	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Firmware download	Properties dialog box, General tab page (Instrument Configuration Manager)
Oven temperature	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Preheat function	System Setup page (Instrument Method Editor)
Preheat purge	System Setup page (Instrument Method Wizard)
Pressure units	Properties dialog box, Options tab page (Instrument Configuration Manager)

Chromeleon Software Feature	Location of Feature
Purge time	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Rinse (manual)	Dionex ASE 350 Extractor ePanel
Rinse settings	Rinse Settings page (Instrument Method Wizard), Rinse Settings page (Instrument Method Editor)
Rinse volume	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Queue	Queue ePanel
Solvent names	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Solvent names (default)	Properties dialog box, Options tab page (Instrument Configuration Manager)
Solvent ratios	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Static cycles	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Static time	Extraction Method page (Instrument Method Wizard), Extraction Method page (Instrument Method Editor)
Vial tray insert	Properties dialog box, Options tab page (Instrument Configuration Manager)
Wellness panel	Command window
Chromeleon Software Feature Location	How to navigate to this feature
Dionex ASE 350 Extractor ePanel	Chromeleon Console (Instruments view) – Click Dionex ASE 350 Extractor tab
Audit ePanel	Chromeleon Console (Instruments view) – Click Audit tab

Chromeleon Software Feature Location	How to navigate to this feature
Command window	Chromeleon Console (Instruments view) – Command toolbar button – Expand Extractor – Click Wellness
Extraction Method page (Instrument Method Wizard)	Chromeleon Console – Create > Instrument Method – Click Next – Extraction Method page
Extraction Method page (Instrument Method Editor)	Chromeleon Console (Data view) – Select folder or sequence – Double-click existing method (launches Instrument Method Editor) – Extractor module – Click Extraction Method tab
Properties dialog box (Instrument Configuration Manager)	Chromeleon Services Manager – Configure instruments – Dionex ASE 350 module – Properties – Click General or Options tab
Queue ePanel	Chromeleon Console (Instruments view) – Click Queue tab
Rinse Settings page (Instrument Method Wizard)	Chromeleon Console – Create > Instrument Method – Click Next – Rinse Settings page
Rinse Settings page (Instrument Method Editor)	Chromeleon Console (Data view) – Select folder or sequence – Double-click existing method (launches Instrument Method Editor) – Extractor module – Click Rinse Settings tab
System Setup page (Instrument Method Editor)	Chromeleon Console (Data view) – Select folder or sequence – Double-click existing method (launches Instrument Method Editor) – Extractor module – Click System Setup tab

2.5.3 System Wellness

You can monitor the usage of replaceable parts on the Dionex ASE 350 Wellness panel in Chromeleon software. Chromeleon software monitors the number of extractions, pump strokes, pump seal strokes, static valve cycles, static valve seal cycles, hours the display is on, and hours the system is on.

In addition, the Wellness panel displays usage counters for several parts. After replacing a part, contact Technical Support for Dionex products to reset the usage counter to zero.

The Wellness panel is part of the **Command** window (see [Figure 2-9](#)). To view the panel:

1. Press the **F8** key, or click **Command** on the toolbar above the ePanel Set.
2. Under **Extractor**, click **Wellness**.

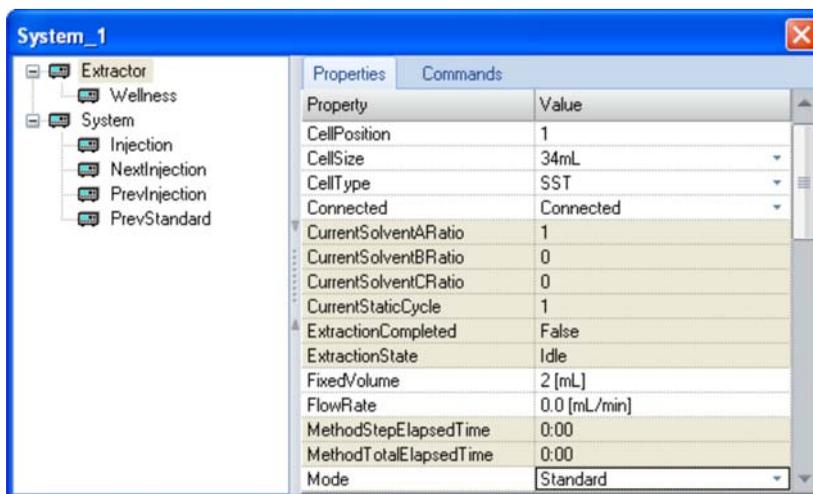


Figure 2-9. Chromeleon Software: Dionex ASE 350 Command Window

2.6 Extraction Process

Before starting an extraction, complete the following preliminary steps. See [Chapter 3](#) for detailed instructions for each step.

- Prepare samples and load them into the cells.
- Place cells in the cell tray.
- Place collection bottles or vials in the collection tray.
- Close the safety shield.
- Create one or more methods.
- Create a sequence.
- Load the method or sequence.

The remainder of this section describes the automatic portion of the extraction process—the steps that the Dionex ASE 350 performs after you press the **Start** button to begin a run (in Local mode) or click **Start** on the Sequence Control Bar in Chromeleon software (in Remote mode).

The key steps in the extraction process include:

- Bringing the oven to the correct temperature for the run
- Loading the cell into the oven
- Filling the cell with solvent
- Heating the cell (equilibration)
- Static extraction
- Rinsing with fresh solvent
- Purging solvent from the system
- Relief
- Unloading the cell

You can monitor the progress of a run from two locations:

- The **SYSTEM STATUS** screen (see [Figure 2-10](#)) on the Dionex ASE 350 front panel. To exit the **SYSTEM STATUS** screen, press **Menu**. For a description of the screen parameters, see [Table 2-2](#).

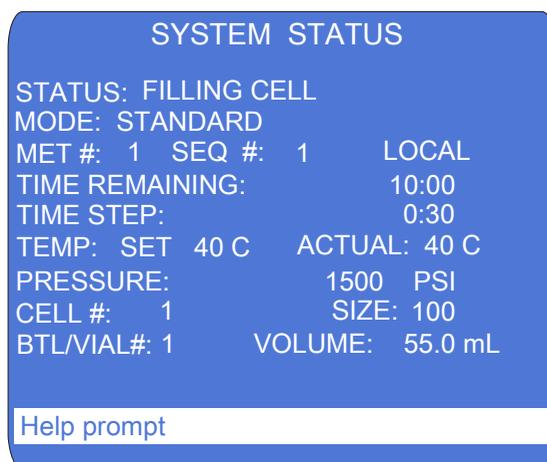


Figure 2-10. System Status Screen

- The Dionex ASE 350 ePanel in Chromeleon software.

Parameter	Description
STATUS	The current system status. For example, during a method, this field displays the following status states (in the order listed): Oven Preheat, Filling Cell, Heating Cell, Static, Rinsing, Purge, Relief, and Finished.
MODE	The solvent saver mode (pressure or flow). If solvent saver mode is turned off, STANDARD is displayed. NOTE: Two solvent saver modes (fixed volume and gradient) are available only in Chromeleon software.
MET #	In Local mode: The method currently running is displayed. In Remote mode: “__” is displayed.
SEQ #	The sequence currently running.
LOCAL/REMOTE	The control mode (Local or Remote).
TIME REMAINING	The time remaining until the current run finishes.
TIME STEP	The elapsed time since the start of the current step.
TEMP SET	The current temperature set point for the oven.
TEMP ACTUAL	The current oven temperature.

Parameter	Description
PRESSURE	The current cell pressure reading. Select the unit of measure on the SYSTEM SETUP screen (see Section B.13) or in the Chromeleon Instrument Configuration Manager.
CELL #	The current sample cell.
CELL SIZE	The size of the sample cell.
BTL/VIAL #	The current collection bottle or vial.
VOLUME	The approximate volume of solvent delivered by the pump.

Table 2-2. System Status Screen Parameters

The following pages briefly describe each step in the extraction process.

Step 1: Loading the Cell

- The cell and collection trays rotate to the initial positions specified in the method or sequence. The needle mechanism pierces the collection vessel septum.
- When the oven reaches the set temperature, the AutoSeal arm picks up the cell and determines the cell size.

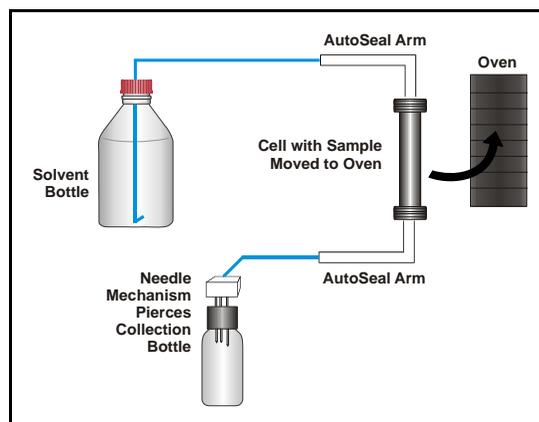


Figure 2-11. Extraction Process: Loading

- The oven temperature is adjusted for the cell size, to ensure that the cell contents reach the set point at the beginning of the static cycle. When the temperature adjustments are complete, the AutoSeal arm moves the cell into the oven.
- The oven applies pressure to seal the cell.

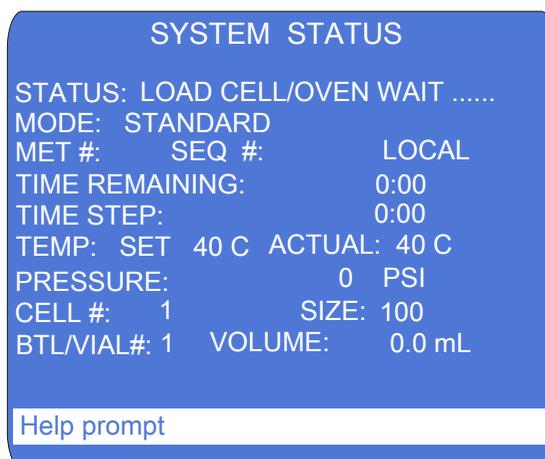


Figure 2-12. System Status Screen: Load Cell

NOTE The oven begins heating to the preprogrammed set point immediately after a method is loaded (before **Start** is pressed to begin the run). Cell loading begins after **Start** is pressed, when the oven is within 1 °C of the set point.

Step 2: Filling the Cell

- With the static valve closed, the pump begins pumping solvent into the cell.
- When the pressure reaches approximately 10.34 MPa (1500 psi), flow stops.
- The static valve opens when the pressure reaches 11.72 MPa (1700 psi).

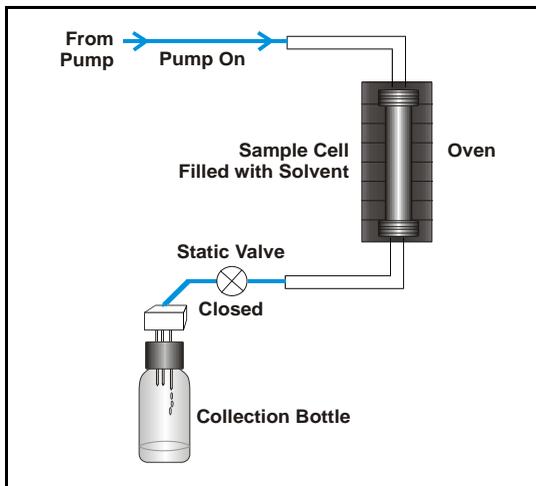


Figure 2-13. Extraction Process: Filling

SYSTEM STATUS

STATUS: FILLING CELL

MODE: STANDARD

MET #: 1 SEQ #: 1 LOCAL

TIME REMAINING: 10:00

TIME STEP: 0:30

TEMP: SET 40 C ACTUAL: 40 C

PRESSURE: 1500 PSI

CELL #: 1 SIZE: 100

BTL/VIAL#: 1 VOLUME: 55.0 mL

Help prompt

Figure 2-14. System Status Screen: Filling the Cell

Steps 3 and 4: Heating and Static

- The cell is heated for a fixed time to ensure that the sample reaches thermal equilibrium.
- The static cycle occurs. The duration (usually 5 minutes) is specified in the method.
- In standard mode: During the heating and static steps, the static valve opens at 11.72 MPa (1700 psi). A small amount of solvent is pumped each time the static valve opens.

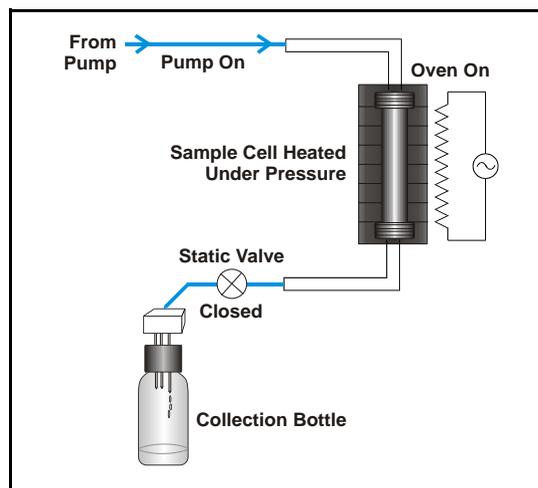


Figure 2-15. Extraction Process: Heating and Static

- In solvent saver pressure mode: During the heating and static steps, the static valve opens at 11.72 MPa (1700 psi), but the pump is off.
- In solvent saver flow mode: During the heating step, the static valve opens at 11.72 MPa (1700 psi). During the static step, the static valve opens at 9.65 MPa (1400 psi). Solvent is pumped throughout the static step.

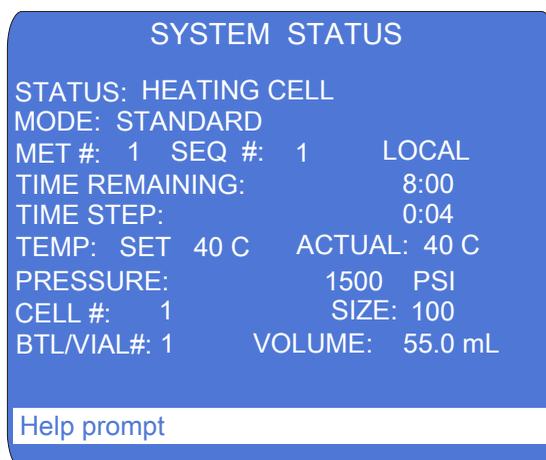


Figure 2-16. System Status Screen: Heating

Step 5: Rinsing the Cell

- The static valve opens and the extract flows into the collection vessel.
- (Optional) Pump fresh solvent (usually 50% to 100% of the cell volume) through the cell. In solvent saver mode (pressure and flow), the default rinse volume is zero.

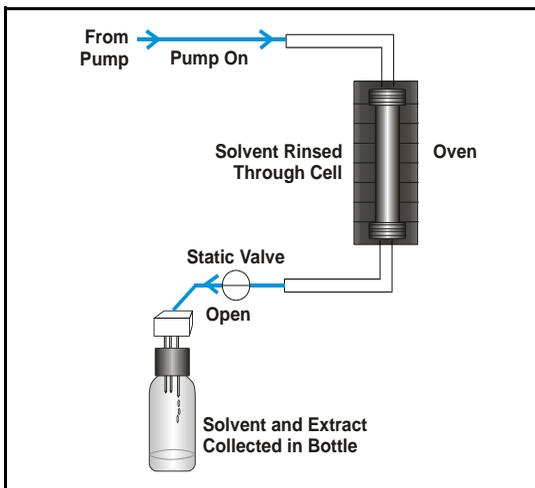


Figure 2-17. Extraction Process: Rinsing

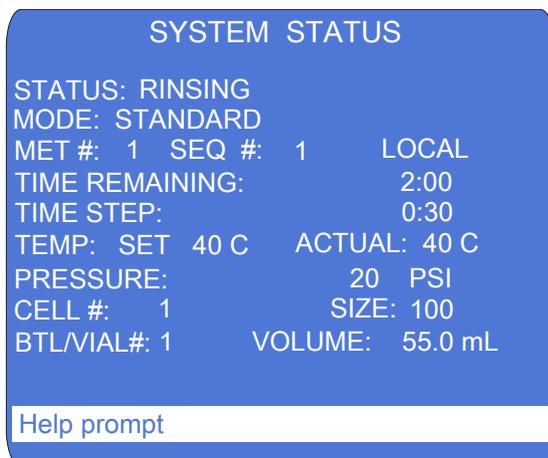


Figure 2-18. System Status Screen: Rinsing

Step 6: Purging

- The remaining solvent is displaced with purge gas.
- The collection vessel now contains all of the solvent and the analytes extracted from the sample.

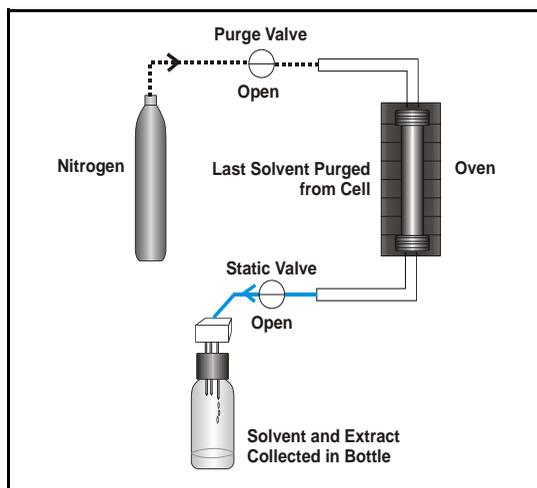


Figure 2-19. Extraction Process: Purging

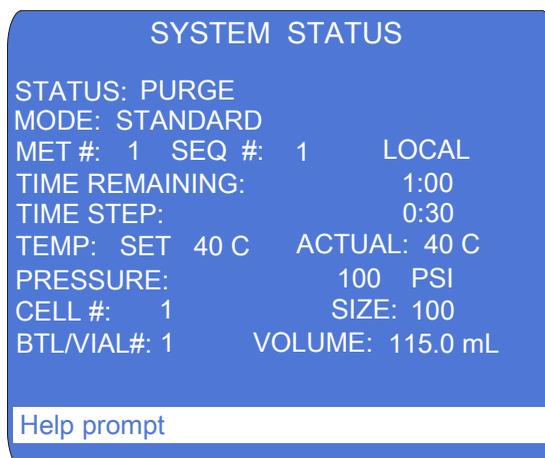


Figure 2-20. System Status Screen: Purging

Step 7: Relief

- Residual pressure is released from the sample cell.
- Pressure is vented from the system.

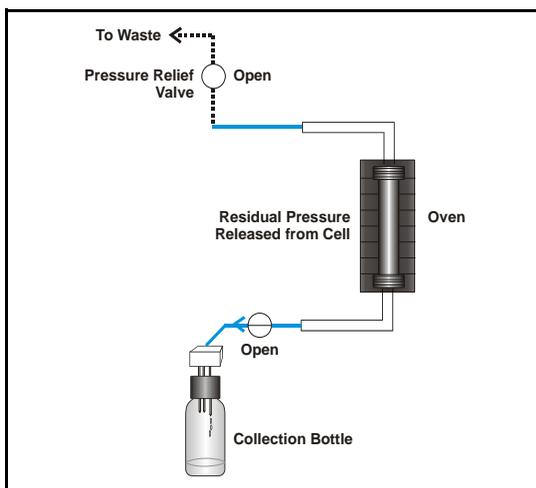


Figure 2-21. Extraction Process: Relief

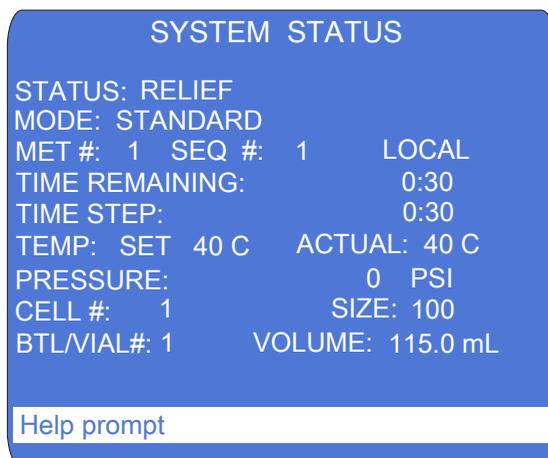


Figure 2-22. System Status Screen: Relief

Step 8: Unloading the Cell

- The cell is unloaded from the oven and returned to the tray.
- The needle mechanism is removed from the collection vessel.
- The trays advance to their next positions, and the next run starts.
- When all runs are completed, the Dionex ASE 350 stops.

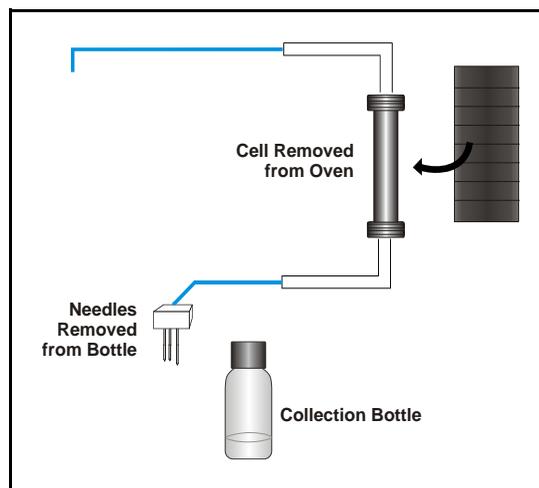


Figure 2-23. Extraction Process: Unloading the Cell

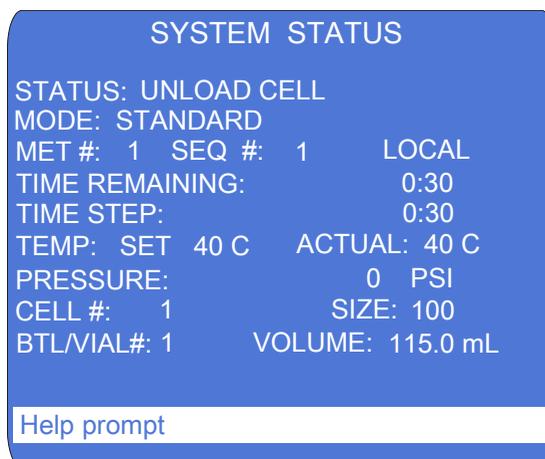


Figure 2-24. System Status Screen: Unloading the Cell



Cells are extremely hot after an extraction. Allow the cells to cool for at least 15 minutes before handling, especially if the cells were heated over 50 °C.



Les cellules sont extrêmement chaudes après une extraction. Laissez-les refroidir pendant au moins 15 minutes avant de les manipuler. Faites particulièrement attention aux cellules qui ont été chauffées à plus de 50 °C.



Die Zellen sind nach ihrer Entnahme sehr heiß. Lassen Sie die Zellen mindestens 15 Minuten abkühlen. Seien Sie besonders vorsichtig, wenn Zellen über 50 °C erhitzt wurden.

During a run, pneumatic solenoid valves turn the pump flow on and off. In addition, the following valves control solvent flow and pressure in the system:

- The static valve controls the flow of solvent to the collection vessel. Pressure in the cell increases when the pump is on and the static valve is closed.
- The purge valve controls gas flow to the cell.
- The pressure relief valve releases any residual pressure after an extraction.
- The prime valve controls gas pressure to the solvent reservoir.

[Figure 2-25](#) illustrates the positions of the valves at each step of the extraction process.

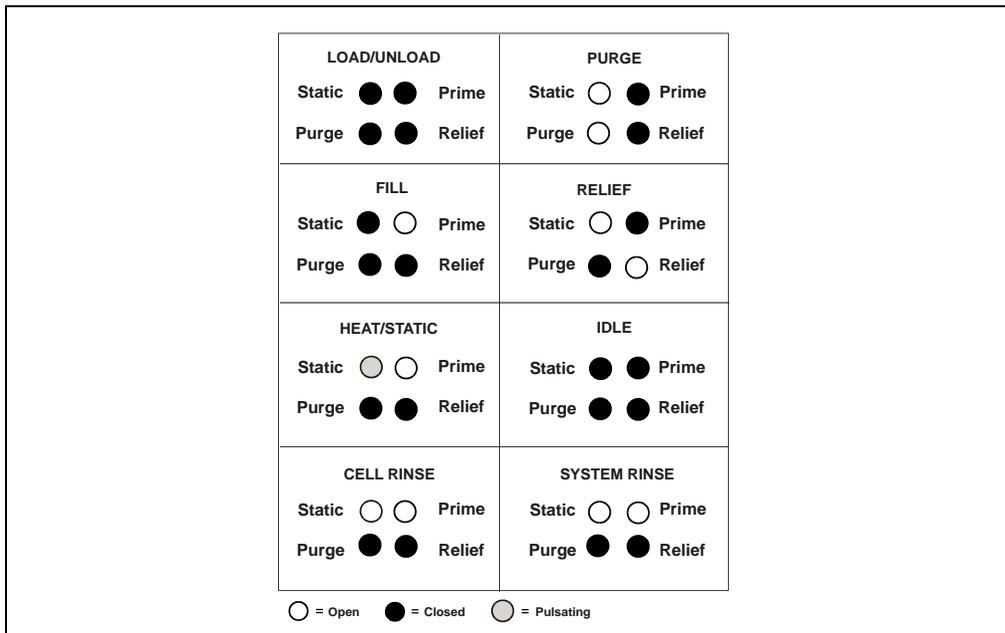


Figure 2-25. Valve Positions During the Extraction Process

2.7 Method and Sequence Control

Before starting a run, you must select a *method* that defines the operating conditions for the run. A method specifies the following parameters:

- Oven temperature
- Time allowed for the sample to reach thermal equilibrium
- Static time
- Number of static cycles
- Amount of solvent to rinse through the cell
- Purge time
- Solvent types and ratios (if more than one solvent reservoir is installed)
- Cell material (stainless steel or zirconium)
- Standard or solvent saver mode

The Dionex ASE 350 can store up to 24 methods. [Section 3.2.1](#) describes how to create methods from the Dionex ASE 350 front panel.

If you plan to perform a series of runs, it is more efficient to define a *sequence*. A sequence specifies the method to run on each sample in the sequence, the sample cell assigned to each collection vessel, and the rinse status after each sample run. The Dionex ASE 350 can store up to 24 sequences. [Section 3.2.3](#) describes how to create sequences from the Dionex ASE 350 front panel.

The Dionex ASE 350 provides two control modes:

- *Method* control runs the same method on each consecutive sample loaded in the tray.
- *Sequence* control runs a series of methods according to the sequence definition.

NOTE For an overview of how to control the Dionex ASE 350 with Chromeleon software, see [Section 3.3](#). For detailed instructions, refer to the Chromeleon software Help.

2.7.1 Solvent Saver Mode

Solvent saver mode is an extraction method available exclusively with the Dionex ASE 350. Solvent saver methods are recommended for:

- Modifying and enhancing existing extraction methods
- Exploring new extraction methods
- Simulating extraction on a small scale before scale-up to a plant scale extraction module

There are four solvent saver options; two options (the fixed volume and gradient modes) are available only in Chromeleon software.

- The *pressure mode* is a static mode of extraction that minimizes solvent usage by maintaining a fixed cell pressure and eliminating the introduction of fresh solvent during the heating and static cycles. This mode uses less solvent than any other Dionex ASE 350 extraction mode.
- The *flow mode* is a continuous dynamic extraction mode in which small aliquots of solvent are delivered to the cell at the specified flow rate throughout the static step. The cell temperature remains constant throughout the run.
- The *fixed volume mode* specifies the solvent volume to be used in an extraction. After this volume has been pumped to the cell, the pump is turned off.
- The *gradient mode* is an extension of the flow mode. In addition to the flow rate used during the static step, solvents A, B, and C have start and end proportions that produce a solvent gradient.

You can select the extraction mode from these locations:

- The **METHOD EDITOR** screen (see [Figure 3-19](#)) on the Dionex ASE 350 front panel.
- or–
- The **Extraction Method** page in the Chromeleon Instrument Method Wizard (see [Figure 2-26](#)).

Instrument Method Wizard - Extractor (ASE 350: Extractor): Extraction Method

Extraction Method

Mode: FixedVolume Cell Type: SST

Oven Temperature

Off

On: 150 [40...200 °C]

At this temperature, the cell will be heated for 7 minutes to ensure that the sample reaches thermal equilibrium.

Static Cycle

Time: 10 [0...2000 min]

Cycles: 1 [1...9]

Rinse Volume: 0 [0...150 %]

Purge

Time: 100 [20...900 s]

Solvents

Type	Ratio
A: Solvent_A	1 [0...5]
B: Solvent_B	0 [0...5]
C: Solvent_C	0 [0...5]

Cell Size: 66mL [1mL...100mL]

Volume: 20 [2...175 mL]

< Back Next > Cancel Help

Figure 2-26. Chromeleon Software: Example Extraction Method Page

Solvent Saver Pressure Mode

The solvent saver pressure mode is a simplified extraction protocol. The sample cell is filled inside the oven; when the fill pressure of 10.34 MPa (1500 psi) is reached, the pump is turned off. The cell (and solvent) are heated at the extraction temperature for a set time. When the pressure reaches 11.72 MPa (1700 psi), the static valve opens to relieve pressure by dispensing some of the solvent out of the cell and into the collection vessel. The run proceeds with no additional fresh solvent entering the

sample cell and no additional static cycles. The default rinse volume is zero; however, you can add a rinse at the end of the cycle, if needed.

The solvent saver pressure mode is recommended for lipid extraction methods with Thermo Scientific™ Dionex™ ASE™ Prep CR Resin (P/N 080024). Although standard extraction methods can be used for this application, use of the pressure mode reduces solvent usage.

Solvent Saver Flow Mode

In the solvent saver flow mode, the pump delivers small aliquots of solvent to the cell, mimicking a dynamic extraction during the static period while maintaining all the steps of a standard accelerated solvent extraction during the filling and heating steps.

The sample cell is filled inside the oven, and the heating step then begins. The cell (and solvent) are heated to the extraction temperature for a set time and the static step begins. The pump meters in solvent at the set flow rate; thus, the heated solvent is in contact with the sample, as well as the small aliquots of incoming solvent. If the pressure in the cell exceeds 10.34 MPa (1400 psi) during the static step, the static valve opens to release some solvent, while fresh solvent increases the cell pressure and the run proceeds. As with the solvent saver pressure mode, the flow mode has just one static cycle.

Because fresh solvent is added in small aliquots throughout the entire static step, the recommended rinse volume is zero (the default). However, you can include a rinse at the end of the static step, if needed.

Solvent Saver Fixed Volume Mode

NOTE Operation in the solvent saver fixed volume mode requires Chromeleon software.

In the solvent saver fixed volume mode, you specify the total amount of solvent to be used during the extraction. Only one static cycle is allowed. Although the rinse volume parameter is not available in this mode, it is possible to perform a rinse by selecting a solvent volume that exceeds the cell size. When the sample cell is placed in the oven, solvent is delivered to the cell until either the selected solvent volume is reached or the cell is full. Method parameters, including the solvent volume, are selected in the Chromeleon Instrument Method Wizard.

- If you select a solvent volume that is *less than* the cell volume: The pump stops at the selected volume and is turned off for the remainder of the extraction time. At the end of the extraction time, nitrogen purges the cell of all the solvent into the collection bottle.
- If you select a solvent volume that is *more than* the cell volume: After the cell is filled, the extraction continues as a standard extraction until the selected volume is reached. If the selected volume has not been reached at the end of the static time, the pump delivers fresh solvent (acting like a final rinse) until the volume has been met.

Solvent Saver Gradient Mode

NOTE Operation in the solvent saver gradient mode requires Chromeleon software.

In the solvent saver gradient mode, the extraction begins with one solvent composition and finishes with a different solvent composition. The solvent transition, which takes place during the static cycle time, is controlled by the flow parameter. Method parameters, including the start and end ratios for each solvent, are selected in the Chromeleon Instrument Method Wizard.

For example, assume that you plan to extract with solvent A and transition to solvent B at a flow rate of 2.0 mL/min for 8 minutes. The sample cell is filled with solvent A inside the oven, and the heating step then begins. The cell (and solvent) are heated to the extraction temperature for a set time and the static step begins. The solvent composition is 100% A at the start of the static time. At 2 minutes into the static time, the composition reaches 75% A and 25% B. At 4 minutes, the ratio is 50% A and 50% B, and so on. At the end of the static time, the solvent composition is 100% B. Only one static cycle is allowed. If any rinse volume is selected, it will be done with the end ratio, which in this case would be 100% B.

3 • Operation and Maintenance

3.1 Preparing to Run

3.1.1 Selecting and Preparing Extraction Solvents

Precautions



WARNING

Do not use solvents with an autoignition point below 200 °C. The table below lists some solvents that should *not* be used with the Dionex ASE 350. If there is a question about solvent suitability, contact Technical Support for Dionex products.

Do Not Use These Solvents	Autoignition Point
Carbon disulfide: CS ₂	100 °C
Diethylether: (C ₂ H ₅) ₂ O	180 °C
1,4-dioxane: C ₄ H ₈ O ₂	180 °C



AVERTISSEMENT

N'utilisez pas de solvants ayant un point éclair inférieur à 200 °C. Le tableau ci-dessous liste quelques solvants qui ne doivent pas être utilisés avec le Dionex ASE 350. Contactez Thermo Fisher Scientific si vous avez un doute concernant l'usage d'un solvant absent de la liste.

Solvants à ne pas utiliser	Point d'auto-inflammation
Disulfure de carbone: CS ₂	100 °C
Éther diéthylique: (C ₂ H ₅) ₂ O	180 °C
1,4-dioxane: C ₄ H ₈ O ₂	180 °C



WARNUNG

Verwenden Sie keine Lösungsmittel, deren Selbstentzündungstemperatur unter 200°C liegt. Die untenstehende Tabelle zeigt einige Lösungsmittel, die Sie nicht mit dem Dionex ASE 350 verwenden sollten. Bei Fragen zur Eignung von Lösungsmitteln wenden Sie sich bitte an Thermo Fisher Scientific.

Nicht zu verwendende Lösungsmittel	Selbstentzündungstemperatur
Kohlendisulfid: CS ₂	100 °C
Diethyläther: (C ₂ H ₅) ₂ O	180 °C
1,4-dioxan: C ₄ H ₈ O ₂	180 °C

Guidelines for Selecting and Preparing Solvents

- When developing a new method, select a solvent or solvent mixture that has a high solubility for the analytes of interest, but not for the sample matrix. If you previously used another method (Soxhlet, for example), use the same solvent with the Dionex ASE 350 that you used with the other method.
- Use HPLC-grade or pesticide-grade solvents.
- Use organic or aqueous solvents.
- Use single-component or multiple-component solvents.
- Solvents do not usually need to be degassed. Degas solvents only if the analyte of interest oxidizes easily.
- If Viton external O-rings are installed in the cell end caps, do not use acetone or other ketones.
- Although the zirconium components in the Dionex ASE 350 flow path are more resistant to acids and bases at elevated temperatures than other metallic materials, follow these guidelines to prevent degradation and ensure optimal performance:
 - a. As a general rule, strong mineral acids such as hydrochloric acid should not be used (at any concentration) as extraction solvents. This is because there are stainless steel components in the flow path of the system (for example, the pump head) that will corrode when used with strong acids. Sulfuric acid and nitric acid can be pumped as a solvent at concentrations less than 0.1% by volume. For more details about acids that can be pumped as solvents, see [Table 3-1](#).

IMPORTANT

Using corrosive acids or bases can damage stainless steel cells and will void the product warranty.

- b. Strong bases such as sodium hydroxide or potassium hydroxide can be pumped as extraction solvents at concentrations less than 0.1% by volume. For more details about bases that can be pumped as solvents, see [Table 3-1](#).
- c. Weak acids such as phosphoric or acetic acid can be used as extraction solvents in small percentages (<5% by volume). Weak

bases such as ammonia can be used at small percentages (<5% by volume).

- d. Noncorrosive additives such as buffers (acetate, phosphate, and so on) can be used as extraction solvents at moderate percentages.
- e. In some cases, nitric or hydrochloric acid can be added directly to the sample before it is placed into a zirconium cell. **(Do not use a stainless steel cell.)** For example, 1 to 2 mL of 5% to 10% HCl can be added directly to a soil sample before it is loaded into a zirconium cell for extraction.
- f. [Table 3-1](#) lists the allowable concentrations for several acids and bases. If a sample needs to be treated with acids stronger than the concentrations here, pretreat the sample with the concentrated acid before loading it into a zirconium cell. **(Do not use a stainless steel cell.)** For more details about sample preparation, see [Section 3.1.3](#).

Acid or Base Concentration	Okay to pump from solvent reservoir? (Use SST or Zr cells)	Add to sample and transfer to Zr cell? (Do not use SST cells)	Add to sample with ASE Prep CR and transfer to Zr cell? (Do not use SST cells)
HCl 8 M	No	No	Yes
HCl 10%	No	Yes	Yes
HCl 0.1 M	No	Yes	Yes
NaOH 8 M	No	No	N/A
NaOH 10%	No	Yes	N/A
NaOH 0.1 M	Yes	Yes	N/A
H ₂ SO ₄ 8 M	No	No	Yes
H ₂ SO ₄ 10%	No	Yes	Yes
H ₂ SO ₄ 0.1 M	Yes	Yes	Yes
KOH 8 M	No	No	N/A
KOH 10%	No	Yes	N/A

Table 3-1. Guidelines for Use of Acids and Bases with the Dionex ASE 350

Dionex ASE 350 Solvent Extraction System

Acid or Base Concentration	Okay to pump from solvent reservoir? (Use SST or Zr cells)	Add to sample and transfer to Zr cell? (Do not use SST cells)	Add to sample with ASE Prep CR and transfer to Zr cell? (Do not use SST cells)
KOH 0.1 M	Yes	Yes	N/A
HNO ₃ 8 M	No	No	Yes
HNO ₃ 10%	Yes	Yes	Yes
HNO ₃ 0.1 M	Yes	Yes	Yes
NH ₄ OH 8 M	No	No	N/A
NH ₄ OH < 5%	Yes	Yes	N/A
NH ₄ OH 0.1 M	Yes	Yes	N/A
Trifluoroacetic acid < 5%	Yes	Yes	Yes
Acetic acid < 5%	Yes	Yes	Yes
Phosphoric acid <5%	Yes	Yes	Yes

Table 3-1. Guidelines for Use of Acids and Bases with the Dionex ASE 350

IMPORTANT

If acids, bases, salts, or buffers are used as extraction solvents, rinse the system with 100% polar organic solvent (for example, acetone or methanol) or distilled water before turning off the power.

3.1.2 Filling the Solvent Reservoir

The solvent reservoir tray can hold up to three reservoirs. Follow the procedure below to fill each reservoir.



Use only Thermo Fisher Scientific solvent reservoirs. These are glass reservoirs with a plastic, shatterproof coating. Make sure the pressure applied to the reservoirs does not exceed 0.07 MPa (10 psi).



Utilisez uniquement des réservoirs à solvant Thermo Fisher Scientific. Ce sont des réservoirs en verre à revêtement incassable en plastique. Veillez à ce que la pression exercée sur ces réservoirs ne dépasse pas 0,07 MPa.



Verwenden Sie ausschließlich die Lösemittelbehälter von Thermo Fisher Scientific. Dabei handelt es sich um Glasbehälter mit einer splittersicheren Plastikbeschichtung. Vergewissern Sie sich, daß der Druck, der auf die Behälter ausgeübt wird, 0,07 MPa nicht übersteigt.



Never fill the solvent reservoir or disconnect the tubing connections to the solvent and gas connectors (see [Figure 3-1](#)) during a run or rinse cycle. At these times, the solvent reservoir is pressurized. If you remove the cap when the reservoir is pressurized, the Dionex ASE 350 may not operate to specification.

1. Fill the solvent reservoir (P/N 045901) with prepared solvent to the level indicated in [Figure 3-1](#).



The solvent level in the reservoir must remain below the gas line. This prevents solvent from coming into contact with the pneumatic valves.

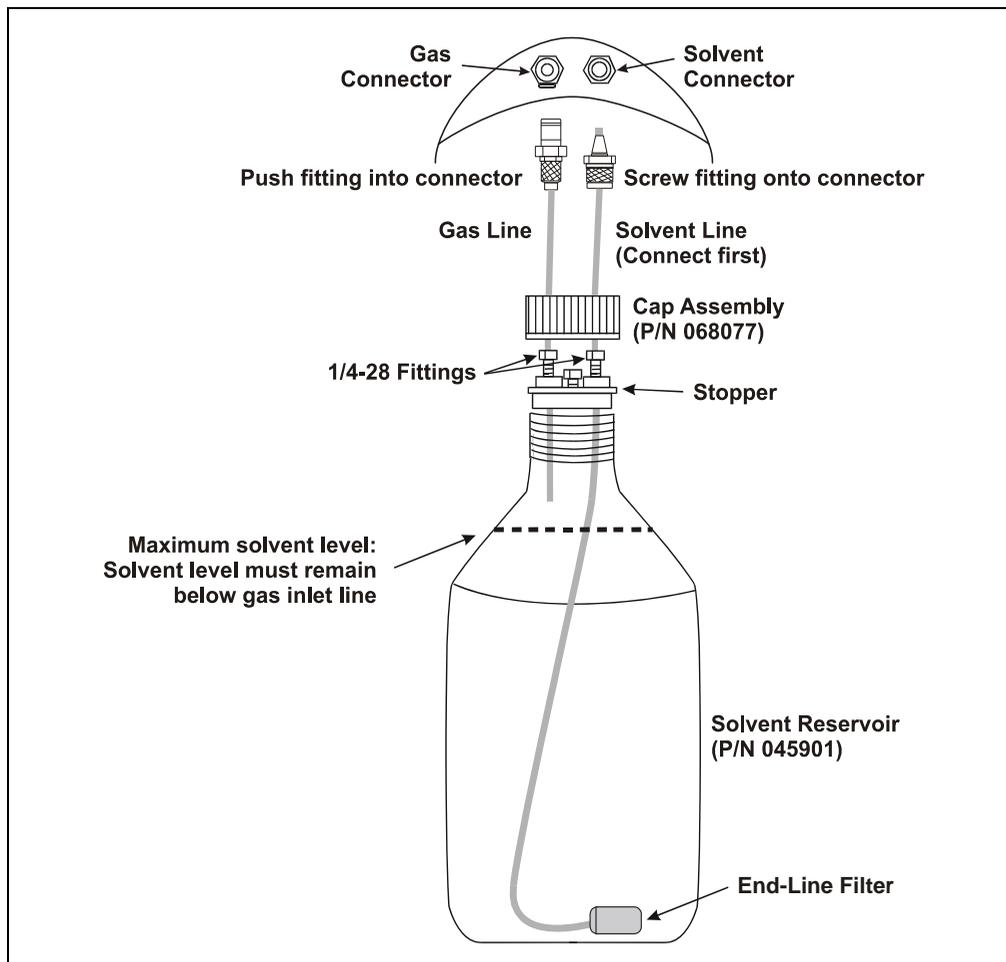


Figure 3-1. Solvent Reservoir Connections

2. Set the reservoir in the designated location in the Dionex ASE 350 solvent reservoir tray (see [Figure 3-2](#)).

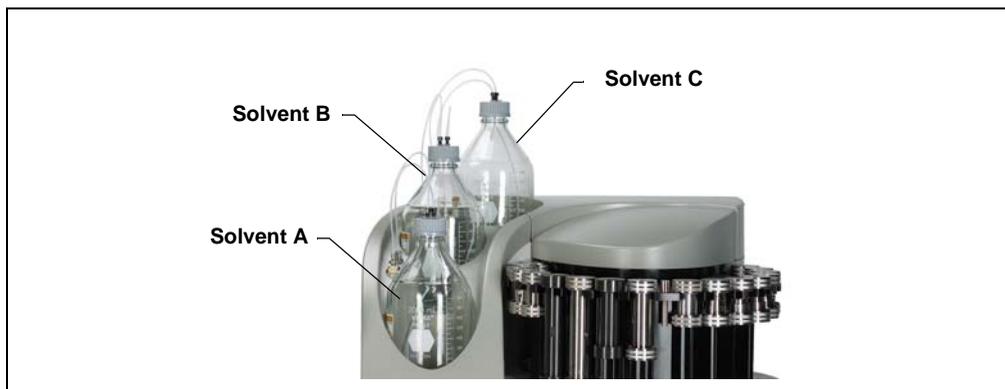


Figure 3-2. Designated Locations for Solvent Reservoirs

3. Insert the solvent line extending from the underside of the reservoir cap assembly into the reservoir (see [Figure 3-1](#)). Make sure the end-line filter rests on the bottom of the reservoir.

If necessary, loosen the fitting on top of the stopper, gently pull on the solvent line to bring more tubing into the reservoir, and then tighten the fitting.

4. Hand-tighten the gray lock ring cap securely over the stopper.

NOTE Always connect the solvent line to the solvent connector first, and then the gas line to the gas connector. When disconnecting the lines, first disconnect the gas line, and then the solvent line. This prevents inadvertent pressurization of the reservoir and solvent flow when it is not connected.

To refill a solvent reservoir:

It is not necessary to disconnect the solvent and gas lines before refilling the reservoir. Loosen the gray lock ring cap, and then remove the stopper and attached tubing from the reservoir. Remove the reservoir from the solvent reservoir tray and place it on a bench for refilling.

3.1.3 Preparing the Sample

If you have successfully followed a particular sample pretreatment procedure for another extraction method, continue using this procedure with the Dionex ASE 350. If you have never run an extraction—or if you are preparing a new sample—follow the guidelines here. This section discusses two sample pretreatment procedures:

- Samples that are dry, wet, or liquid should be mixed with a drying or dispersing agent before being loaded into the cells.
- Samples with large particle sizes should be ground before being loaded into the cells.

NOTE Most problems with the ASE (accelerated solvent extraction) process are caused by errors made during the sample preparation phase. To obtain high quality analytical results, pay careful attention when preparing samples. For more information, see [Appendix E](#).

Guidelines for Selecting a Drying or Dispersing Agent

- The sample preparation guidelines on [page 57](#) recommend the use of Thermo Scientific™ Dionex™ ASE™ Prep DE (P/N 062819) (pelletized diatomaceous earth) as a drying agent. DE provides a clean transfer of the mixtures to the cell, and extracts well.
- Although sodium sulfate is readily available, do not use it in Dionex ASE 350 cells. The use of sodium sulfate with very wet samples (30% moisture) will clog system components (such as the solvent line or needles) with recrystallized sodium sulfate. This occurs particularly if a mixed solvent with a polar component such as methanol or acetone is used. In these cases, use Dionex ASE Prep DE as a drying agent and mix it with the sample before loading into the sample cell. (Alternatively, Dionex ASE Prep DE can be used as a drying agent in the cell in place of sodium sulfate for all levels of moisture.)
- For very wet samples, regardless of which drying agent is used, you may add sodium sulfate to the collection vessels after collection and then pass the extracts through either a drying column or drying cartridge to dry the extract completely. At the temperatures used

during Dionex ASE 350 extractions, more water is co-extracted than with other extraction procedures. To ensure good analyte recovery, thoroughly rinse the sodium sulfate from the collection vessel and the cleanup column.

- If a sample needs to be treated with acids stronger than the concentrations listed in [Table 3-1](#), pretreat the sample with the concentrated acid before loading it into the cell. After the pretreatment, mix the sample with Dionex ASE Prep CR Resin (P/N 080024) to partially neutralize the acid to approximately 0.1 M. Note that the sample may need to be mixed with Dionex ASE Prep DE (P/N 062819) to absorb water, depending on how much acid was used to pretreat the sample.

For example, if a food sample is treated with 10 mL of 8 M HCl, mix the liquid hydrolysate with 30 to 40 g of Dionex ASE Prep CR and 15 g of Dionex ASE Prep DE. When the sample is thoroughly mixed, add it to a zirconium cell. **(Do not use a stainless steel cell.)** Use this ratio of acid to Dionex ASE Prep CR whenever using strong acids for pretreatment of samples.

Sample Preparation with a Drying or Dispersing Agent

The following mixtures are recommendations only; adjust the proportions as required.

- If the sample appears dry, use this mixture:
4 g sample to 1 g Dionex ASE Prep DE
Mix the sample and the DE thoroughly in a small bottle, beaker, or mortar.
- If the sample appears wet, use this mixture:
4 g sample to 2 g Dionex ASE Prep DE
Mix the sample and the DE thoroughly in a small bottle, beaker, or mortar.

- If the sample is pure liquid, use 5 g sample to 3 g Dionex ASE Prep DE.

Fill the cell with DE and then add the sample.

NOTE Certain pure liquid samples may be mixed with drying or dispersing agents other than Dionex ASE Prep DE. For a current list of approved materials, contact Technical Support for Dionex products.

Guidelines for Grinding

- For an efficient extraction to occur, the solvent must make contact with the target analytes. The more surface area that can be exposed in a sample, the faster the extraction will occur. Therefore, samples with large particle sizes should be ground prior to extraction. Often a large, representative sample can be ground, after which weighed portions of the ground sample can be used for extraction.
- Soil and sediment samples usually do not need to be ground, but you may need to remove stones or sticks from the samples prior to extraction.
- Polymer samples must be in a ground state for an efficient extraction of additive compounds. Materials such as polymers and rubbers are best ground at reduced temperatures (for example, while chilling with liquid nitrogen).
- Animal or plant tissue samples can be homogenized using any procedure, including a blender or tissue homogenizer.

Sample Preparation by Grinding

Grind the sample with one of the following tools: a conventional mortar and pestle, an electric grinder, or an electric mill. For the most efficient extraction, grind until particles are smaller than 1 mm.

3.1.4 Selecting the Sample Cell

Sample cells are available in the following sizes: 1 mL, 5 mL, 10 mL, 22 mL, 34 mL, 66 mL, and 100 mL. All cells are available in stainless steel. Two cell sizes (66 mL and 100 mL) are available in zirconium, also.

The cell size does not affect the extraction time, but it does determine how much solvent is used; because the cell is filled with solvent during the extraction, larger cells require more solvent. Also, a cell that is partially filled with sample requires more solvent than a full cell.

In general, when choosing a cell size:

- Select the smallest cell that holds enough sample to produce accurate extraction results.
- Take into account any drying or dispersing agents, because these increase the volume of the sample.

3.1.5 Installing the Sample Cell Filter

A disposable filter must be installed in the bottom end of the sample cell before sample is loaded. The filter prevents blockage of the frit in the bottom end cap. Review the information here to determine the appropriate filter material and size.

Cell filters are available in two materials:

- *Cellulose* filters are appropriate for most extraction methods that use organic solvents.
- *Glass-fiber* filters are recommended for aqueous extractions, where cellulose may provide inadequate filtration or may interfere with the analytical technique.

Cell filters are available in two sizes:

- Use 27-mm filters for smaller cells (1 mL, 5 mL, 10 mL, or 22 mL).
- Use 30-mm filters for larger cells (34 mL, 66 mL, or 100 mL).

Each Dionex ASE 350 Startup Kit includes a package of 100 cellulose filters in the size required for the sample cell to be used. For information about reordering filters (or ordering filters in a different material or size), see [Appendix D](#).

To install a 27-mm cell filter in a 1-, 5-, 10-, or 22-mL sample cell:

1. Unscrew the bottom end cap from the cell body.

To identify the cell top and bottom, designate the end of the cell with the grooved band around the body as the cell top and the end with the Thermo Scientific logo, serial number, and size as the cell bottom (see [Figure 3-3](#)).



Figure 3-3. Sample Cell Orientation (Smaller Cells)

2. Place a 27-mm filter in the center of the cell bottom end cap (see [Figure 3-4](#)).
3. To prevent leaks, check that no part of the filter overlaps onto the PEEK seal.



Figure 3-4. Installing a 27-mm Cell Filter

4. Carefully place the cell body over the bottom end cap, screw the body onto the end cap, and hand-tighten.

IMPORTANT

Always tighten the cell end caps by hand. Use of a wrench or other tool can damage the cell, as well as the seals inside the cell end caps, and will void the product warranty.

To install a 30-mm cell filter in a 34-, 66-, or 100-mL sample cell:

IMPORTANT

Do not place the 30-mm filter in the bottom end cap before installing the cap; this creates an improper seal and allows leaks.

1. Unscrew the top end cap from the cell body. Verify that an end cap is installed on the bottom of the cell.

To identify the cell top and bottom, designate the end of the cell with the grooved band around the body as the cell top and the end with the Thermo Scientific logo, serial number, and size as the cell bottom (see [Figure 3-5](#)).



Figure 3-5. Sample Cell Top (Larger Cells)

IMPORTANT

Always tighten the cell end caps by hand. Use of a wrench or other tool can damage the cell, as well as the seals inside the cell end caps, and will void the product warranty.

2. Follow the steps in [Figure 3-6](#) to install the 30-mm filter.

NOTE The filter installation procedure is the same for both zirconium and stainless steel cells.



Figure 3-6. Installing a 30-mm Cell Filter in a 34-mL, 66-mL, or 100-mL Cell (Zirconium Cell Shown)

3.1.6 Filling the Sample Cell

Precautions

- When filling the cell with sample, keep the cell body threads as clean as possible. This will prevent thread fouling and extend the life of the cell.
- Also make sure that the ends of the cell body and the seals in the end caps are clean. If debris is allowed to remain here, it will damage the cell body and/or allow leaks during the run.

To fill a sample cell:

1. Carefully load the sample into the top of the cell, using the funnel provided in the Dionex ASE 350 Startup Kit. To accommodate different sample cell sizes, three funnels (with different inner diameters) are available (see [Figure 3-7](#)).

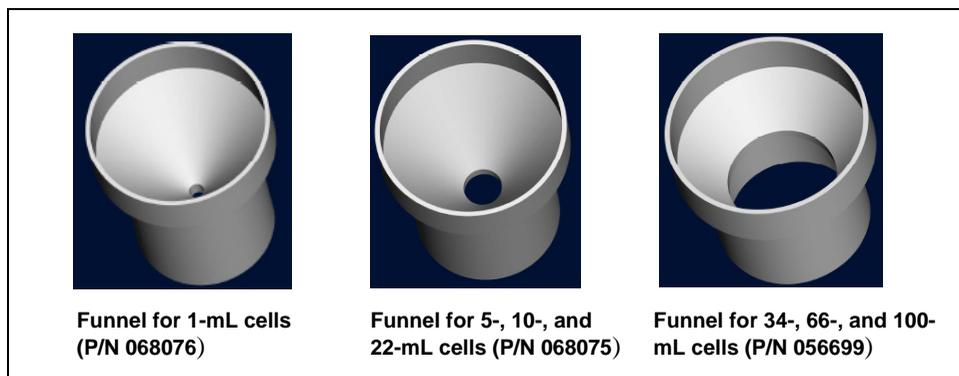


Figure 3-7. Sample Funnels

2. (Optional) To reduce the amount of solvent used during the extraction, fill any void volume in the cell with an inert material such as Ottawa sand (Fisher S23-3).
3. Using a soft brush or cloth, wipe all debris off the threads.
4. Screw the top end cap onto the cell body. Hand-tighten the end cap.

IMPORTANT

Always tighten the cell end caps by hand. Use of a wrench or other tool can damage the cell, as well as the seals inside the cell end caps, and will void the product warranty.

5. Check the ends of each cell end cap to verify that the O-rings are in place and are in good condition (see [Figure 3-8](#)).

- If an O-ring is dislodged, press it into place, using the O-ring insertion tool (P/N 049660) provided in the Dionex ASE 350 Ship Kit (P/N 066060).

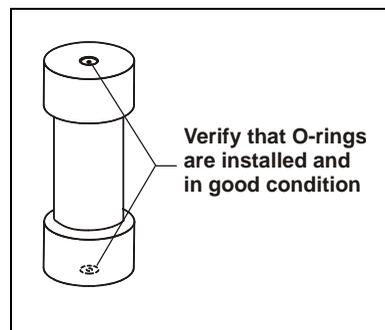


Figure 3-8. Inspecting Cell O-Rings

- If an O-ring is missing, place a new O-ring over the opening in the end of the cell end cap. Press the O-ring into place, using the O-ring insertion tool (see [Figure 3-9](#)). For part numbers for O-rings, see [Appendix D](#).
- If an O-ring has a hole size of less than 0.5 mm, replace it. If a white PTFE O-ring is discolored, replace it (see [page 64](#)).

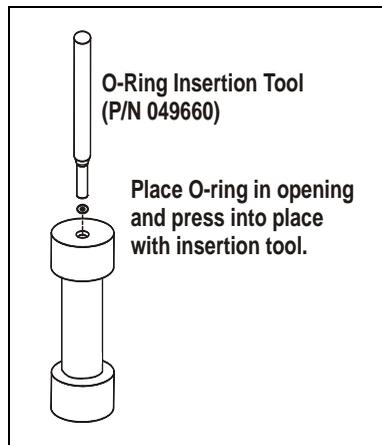


Figure 3-9. Installing a Cell End Cap O-Ring

IMPORTANT

Do not attach any labels to the cell. The cells fit snugly in the oven during the extraction process and a label may cause misalignment. In addition, the high temperatures used during extraction can damage the label. For sample identification, reference the serial number etched on the cell or write on the cell body with a marking pen.

To replace a cell end cap O-ring:

1. Locate the small flathead screwdriver (P/N 046985) and the O-ring insertion tool (P/N 049660) in the Dionex ASE 350 Ship Kit (P/N 066060).

IMPORTANT

Be careful not to scratch the interior of the cell end cap when installing or removing the cell O-ring. Scratches on the sealing surface will prevent the O-ring from sealing properly and may result in leaks during operation.

2. Insert the tip of the screwdriver into the end cap and *carefully* pick out the O-ring. (This may be easier to do if you first remove the cap from the cell body and place it flat on the workbench.)
3. Place a new O-ring (PTFE: P/N 049457, pkg. of 50; Viton: P/N 056325, pkg. of 50) over the opening in the end of the cell end cap. Press the O-ring into place, using the O-ring insertion tool (see [Figure 3-9](#)).

3.1.7 Loading the Cell Tray



To avoid personal injury, exercise caution whenever the tray is in motion.



Pour éviter de vous blesser, faites bien attention lorsque le plateau est en mouvement.



Um Verletzungen vorzubeugen, seien Sie vorsichtig, wenn sich der Probensteller bewegt.

1. Begin loading filled cells into the tray slots in numerical order. Hang the cells vertically in the tray slots from their top end caps. (The top end cap is the one that *does not* contain a cellulose or glass-fiber filter.)

If you plan to run under *method* control, load cells on the tray in any order desired. If you plan to run under *sequence* control, make sure the cell position matches the position specified in the sequence.

[Section 3.2](#) describes method and sequence modes in detail.

2. Locate the two rinse tubes (P/N 068074) provided in the Dionex ASE 350 Ship Kit (P/N 066060). Check the ends of each rinse tube to verify that the O-rings are in place and in good condition (see [Figure 3-10](#)).

If an O-ring has a hole size of less than 0.5 mm, replace it. If a white PTFE O-ring is discolored, replace it (see [page 66](#)).

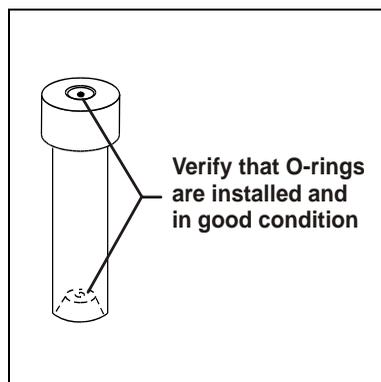


Figure 3-10. Inspecting Rinse Tube O-Rings

3. Load the rinse tubes into the two holes between cell positions 6 and 7 and positions 18 and 19. **Always install rinse tubes in both holes.**

To replace a rinse tube O-ring:

1. Locate the small flathead screwdriver (P/N 046985) and the O-ring insertion tool (P/N 049660) in the Dionex ASE 350 Ship Kit (P/N 066060).

IMPORTANT

Be careful not to scratch the interior of the rinse tube when installing or removing the rinse tube O-ring. Scratches on the sealing surface will prevent the O-ring from sealing properly and may result in leaks during operation.

2. Insert the tip of the screwdriver into the rinse tube and carefully pick out the O-ring.
3. Place a new O-ring (PTFE: P/N 049457, pkg. of 50; Viton: P/N 056325, pkg. of 50) over the opening in the end of the rinse tube. Press the O-ring into place, using the O-ring insertion tool (see [Figure 3-9](#)).

3.1.8 Loading Collection Vessels

Identification of Collection Vessels (Optional)

Before loading collection vessels into the collection tray, you may want to attach a label to a collection vessel or write an identifying name or number on the vessel. [Figure 3-11](#) shows the acceptable locations for this information.

NOTE During the extraction process, sensors determine if a collection vessel is present and, if so, whether it is full. Information outside the approved areas may block the sensors.

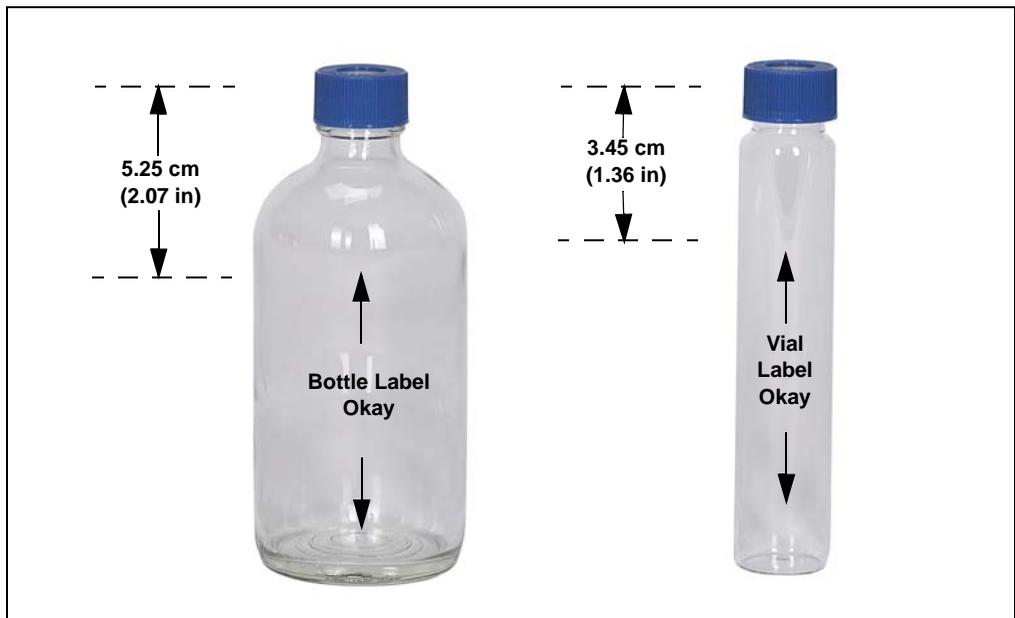


Figure 3-11. Placement of Collection Vessel Labels

To install the vial tray insert (for 60-mL collection vials only):

Before installing collection vials, a vial tray insert (P/N 066176) must be in place on top of the collection tray. The insert resizes the holes in the tray to ensure that the vials, which are smaller than the collection bottles, are secured in place.

When the vial tray insert is installed, the tray can hold up to 28 60-mL collection vials on the outside row, plus five 250-mL collection bottles and two rinse bottles on the inside row.

A vial tray insert is provided in both the Small Volume Startup Kit (P/N 068254) and the 34-mL Volume Startup Kit (P/N 068255).

1. To lock the trays, press the **Trays** button until the right-side LED lights and the collection tray moves to the home position.
2. Open the safety shield.
3. Remove the rinse bottles from the collection tray. Also remove any collection bottles that are present.
4. Orient the vial tray insert so that the indexing feature (the cutout between positions 1 and 2) (see [Figure 3-12](#)) is on the left side of the insert.

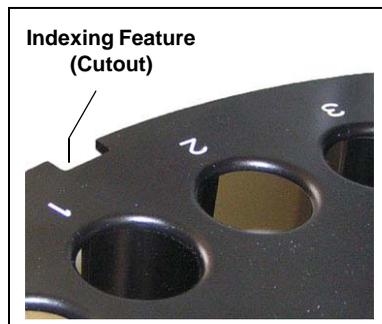


Figure 3-12. Cutout in Vial Tray Insert

5. Move the vial tray insert inside the safety shield and hold the tray insert directly over the collection tray.

6. After verifying that the cutout in the vial tray insert is aligned with the sensor cover on the inside wall (see [Figure 3-13](#)), lower the tray insert onto the collection tray and firmly press it into place.

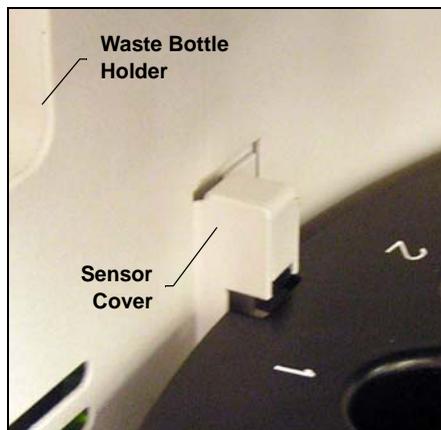


Figure 3-13. Cutout-Sensor Cover Alignment

7. Reinstall the rinse bottles.
8. Load the collection vessels (for instructions, see [page 70](#)).
9. After loading the collection vessels:
 - a. Close the safety shield.
 - b. Press the **Trays** button twice. This enables the Dionex ASE 350 to recognize that the vial tray insert has been installed.

To load the collection vessels:



Before each run, carefully inspect all collection vessels for chips, scratches, or cracks. If a collection vessel shows any sign of damage, do not use it. Use each collection vessel once only.



La présence de fêlure ou d'égratignures sur les flacons de collecte doit être vérifiée avant chaque extraction. N'utilisez jamais un flacon endommagé. Les flacons non gradués sont à usage unique.



Untersuchen Sie vor jedem Lauf alle Sammelgefäße auf Abplatzungen, Kratzer oder Risse. Wenn ein Sammelgefäß eine Beschädigung aufweist, sollten Sie es nicht mehr verwenden. Verwenden Sie nicht graduierte Sammelgefäß nur ein Mal.



Use each collection vessel cap and septum once only. This prevents solvent leaks caused by piercing the septum multiple times.

The collection tray can hold up to 14 collection bottles on the outside row, plus five collection bottles and two rinse bottles on the inside row.

- When running under *method* control: For each sample cell loaded, load a collection vessel into the collection tray. All collection vessels must be the same type (either bottles or vials).
- When running under *sequence* control: If a vial tray insert is installed (see [page 68](#)), you can mix collection bottles and vials in the same run.
- Check that the collection vessels are large enough for the cell used.

If the sample cell is:	Load this collection vessel:
1 mL, 5 mL, 10 mL, 22 mL, or 34 mL	250-mL bottle or 60-mL vial
66 mL or 100 mL	250-mL bottle only

- Load a collection bottle into both rinse positions (labeled R1 and R2).



Check the rinse bottles after each series of runs; if necessary, empty the bottles before starting the next series.

- When you finish loading collection vessels, close the safety shield.

- If you have just installed a vial tray insert (see [page 68](#)), press the **Trays** button twice. This enables the Dionex ASE 350 to recognize the presence of the vial tray insert.

3.1.9 Rinsing (Priming) the System

Pressing the **Rinse** button on the keypad starts up to nine manual rinse (or prime) cycles. During a rinse cycle, the cell tray rotates to the nearest rinse tube, the collection tray rotates to a rinse position, and solvent is pumped through the system. This is followed by a 20-second nitrogen purge step.

Run a manual rinse cycle at the following times:

- After initial setup
- After refilling a solvent reservoir (rinse twice to clear the solvent line of air)
- After changing solvents (rinse twice to remove all of the previous solvent)
- After the Dionex ASE 350 has been shut down for more than one day

In addition:

- When using acids, bases, salts, or buffers as extraction solvents, rinse the system with deionized water at the end of operation.

To select parameters for a manual rinse cycle:

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **5** to display the **SYSTEM SETUP** screen (see [Figure 3-14](#)).

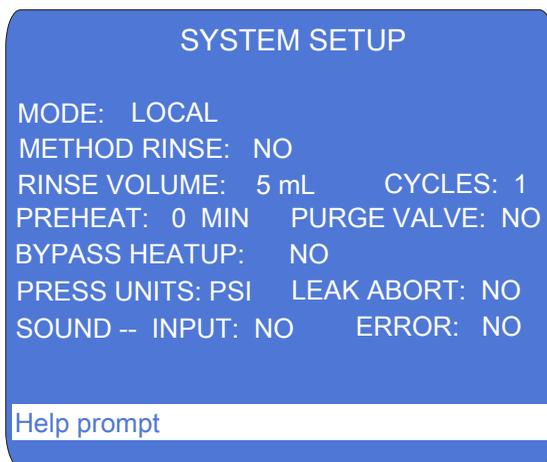


Figure 3-14. System Setup Screen

NOTE For a description of all the parameters on the **SYSTEM SETUP** screen, see [Section B.13](#).

2. Move the cursor to the **RINSE VOLUME** field. Press a numeric button to specify the solvent volume to be pumped through the system during each rinse cycle. Press **Enter** or a cursor arrow button.
3. Move the cursor to the **CYCLES** field. Press a numeric button to specify the number of rinse cycles to run. Press **Enter** or a cursor arrow button.

NOTE For instructions on how to start a rinse cycle from the Dionex ASE 350 ePanel, refer to the Chromeleon software Help (under “Controlling an ASE 350”).

3.1.10 Automatic Rinsing Between Samples

The Dionex ASE 350 can be set up to perform an automatic rinse between sample extractions. The automatic rinse is identical to the manual rinse cycle (see [Section 3.1.9](#)), except that the system can be rinsed only once.

- When running under *method* control, you can set up the Dionex ASE 350 to automatically rinse the system after each sample extraction (see the procedure below).
- When running under *sequence* control, you must specify on each line in the sequence whether an automatic rinse should be performed (see [Section 3.2.3](#)).

To set up an automatic rinse cycle under method control:

1. Verify that the rinse tubes are in place. Verify that empty rinse bottles are in place.
2. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **5** to display the **SYSTEM SETUP** screen (see [Figure 3-15](#)).

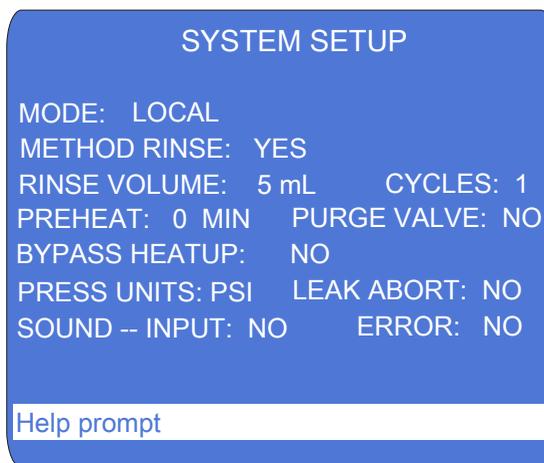


Figure 3-15. System Setup Screen

3. Move the cursor to the **METHOD RINSE** field and press a **Select** button to toggle the field to **YES**. Press **Enter** or a cursor arrow button.
4. Move the cursor to the **RINSE VOLUME** field. Press a numeric button to specify the solvent volume pumped through the system during the rinse. Press **Enter** or a cursor arrow button.

3.1.11 Selecting Local or Remote Mode

When the Dionex ASE 350 is powered up, it is always in Local mode (front panel control) and **LOCAL** is displayed in the operating mode field on the **SYSTEM SETUP**, **SYSTEM STATUS**, and **DETAILED STATUS** screens (for example, see [Figure 3-16](#)).

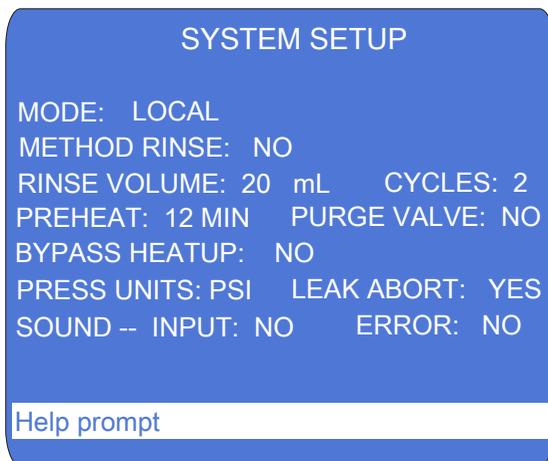


Figure 3-16. System Setup Screen—Local Mode

If you plan to operate in Remote mode (computer control), follow the steps in [“Connecting to Chromeleon Software \(Optional\)”](#) on page 75 to connect the Dionex ASE 350 to the Chromeleon ePanel.

When the Dionex ASE 350 is under computer control, **REMOTE** is displayed in the operating mode field on the **SYSTEM SETUP**, **SYSTEM STATUS**, and **DETAILED STATUS** screens (for example, see [Figure 3-17](#)).

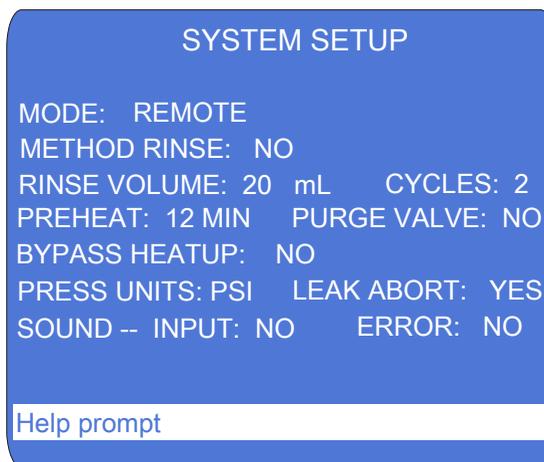


Figure 3-17. System Status Screen—Remote Mode

To return the Dionex ASE 350 to Local mode:

- Click the **Connected** button on the ePanel (see [Figure 3-18](#)). This disconnects the system from Chromeleon software (and changes the button label to **Disconnected**).

–or–

- Turn off the Dionex ASE 350 power briefly.

Connecting to Chromeleon Software (Optional)

1. Start the Chromeleon Instrument Controller Service:
 - a. If you have not already done so, turn on the computer power and log on to Windows as a user with local computer administrator privileges.
 - b. Right-click the Chromeleon software tray icon  in the notification area of the Windows taskbar, and then click **Start Chromeleon Instrument Controller**. The icon changes to gold  to indicate that the Instrument Controller Service is starting. When the Instrument Controller Service is running (idle), the icon changes to gray .

NOTE If the Chromeleon software tray icon is not on the Windows taskbar, click **Start > All Programs > Thermo Chromeleon 7 > Services Manager**, and then click **Start Instrument Controller**.

2. To start the Chromeleon software client, click **Start > All Programs > Chromeleon 7 > Chromeleon 7**.
3. Click the **Instruments** category bar in the Chromeleon Console, and then click the name of your instrument in the Navigation Pane. Chromeleon software will connect to the instrument and display the Dionex ASE 350 ePanel (see [Figure 3-18](#)).

ASE 350 Extractor Audit Queue

Extractor Status
System Status: Idle
Method:
Temperature Set: 40 °C
Temperature Actual: 40 °C
Volume Delivered: 0.0 mL
Time Remaining: 0:01
Step Time: 0:01
Cell Size: ---
Cell Pressure: 0 [psi]

Rinse	Extraction	Method
Idle	Idle	Idle

Manual Rinse
Volume: 1 mL Ratio A: 1
Cycles: 1 Ratio B: 0
Ratio C: 0

Rinse

Legend:
C: Solvent Name
B: Solvent Name
A: Solvent Name

Mode Standard
Connected

Date	Time	Retention Time	Device	Message
12/21/2011	1:42:02 PM		Extractor	Connection established successfully.
12/21/2011	1:42:02 PM		Extractor	Trying to connect ...

Figure 3-18. Chromeleon Software: Example Dionex ASE 350 ePanel

4. Check the lower-left corner of the Dionex ASE 350 graphic. A green **Connected** button should be displayed. If a red **Disconnected** button is currently displayed, click the button to connect the Dionex ASE 350 to Chromeleon software.

3.2 Methods and Sequences

NOTES

- This section describes how to create custom methods and sequences from the Dionex ASE 350 front panel. If you prefer to start operation without creating a new method, run the default method provided with the system (see [Section 3.5.1](#)).
- Most of the information here (including descriptions of method and sequence parameters, guidelines for selecting solvent types, and details about solvent saver modes) applies to all Dionex ASE 350 methods and sequences. Therefore, it is advisable to review this section even if you plan to use Chromeleon software to create methods and sequences (see [Section 3.3](#)).

Each sample extraction occurs according to a predefined set of operating parameters called a *method*. A method specifies the cell heating time, oven temperature, and so on. The Dionex ASE 350 can store up to 24 methods.

When running a series of extractions, you can run each sample using the same method (method control) or customize the series by defining a *sequence* of runs (sequence control). A sequence specifies the method to run on each sample in the sequence, the sample cell assigned to each collection vessel, and the rinse status after each sample run. The Dionex ASE 350 can store up to 24 sequences.

[Table 3-2](#) is an overview of methods, sequences, and control modes.

Feature	Description
Method	Defines the operating parameters for a single run, including oven temperature, time, amount of solvent rinse, nitrogen purge time, solvent saver mode, cell type, and (when more than one solvent reservoir is installed), solvent types and ratios.
Sequence	Defines parameters for a series of runs (cell sequence, method to run on each cell, and rinse status).

Table 3-2. Methods and Sequences Summary

Feature	Description
Method control	Runs the same method on each sample loaded in the tray. By default, a method begins running at collection vessel 1 and cell 1 and continues until completing the entire tray (if it is full) or until reaching an empty collection vessel or cell slot. All collection vessels must be the same type (bottles or vials). Select the method to run on the LOAD METHOD/SEQUENCE screen (see Figure 3-31).
Sequence control	Runs a series of extractions according to the definition of the sequence selected on the LOAD METHOD/SEQUENCE screen (see Figure 3-33). Collection vessels in a run can include a mixture of bottles and vials.

Table 3-2. Methods and Sequences Summary (Continued)

3.2.1 Creating Methods

[Table 3-3](#) describes each method parameter. See [Section 3.2.2](#) for example methods and [Section 3.4](#) for method development guidelines.

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **3** to display the **METHOD EDITOR** screen.

When you open the screen for the first time after power-up, the screen displays the parameters for the default method (see [Figure 3-19](#)). Thereafter, the screen displays the last method that was edited (if any) or the default parameters (if there is no previous method).

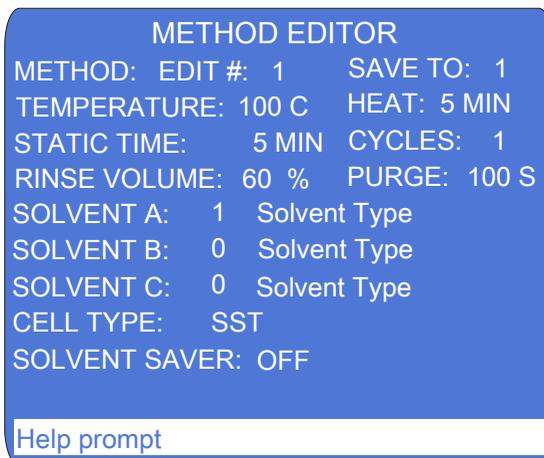


Figure 3-19. Method Editor Screen (Default)

2. To display the default parameters, verify that the cursor is in the **METHOD EDIT #** field and enter zero.
3. To create a method, move the cursor to each field to be changed and enter the preferred value. After entering a new value, save it by pressing **Enter** or a cursor arrow button.

NOTE If you make an invalid selection, an error message appears. Press any key to erase the error message, and then correct the entry.

4. After saving the method parameters, move the cursor to the **METHOD SAVE TO** field. Enter a new (unused) method number, or press **Select** Δ to display the next unused method number. For example, if methods 1 and 3 already exist, pressing **Select** Δ displays 2, the next unused number. Press **Enter** to save the method under the preferred number.

Parameter	Function	Value Range
METHOD EDIT #	The number of the method to edit. Enter the number on the numeric keypad or press a Select button to step through the numbers of all existing methods. To display the default method values, press zero.	0, 1 through 24 (default=1)
METHOD SAVE TO	The method that your editing changes will be saved to. Enter the number on the numeric keypad or press a Select button to step through all unused method numbers.	1 through 24 (default=1)
TEMPERATURE	The temperature set point (the temperature at which the cell is heated). This value determines the duration of the initial heat-up step. Note: Do not select a set point of zero unless you plan to operate at room temperature.	0, 40 to 200 °C (0=off, default=100)

Table 3-3. Method Editor Parameters

Parameter	Function	Value Range
HEAT (read-only)	The time allowed for the sample to reach thermal equilibrium. The duration of the initial heat-up step depends on the temperature set point for the method. Set Point (°C) Heat-up Time (Minutes) 0 (off) 0 40 to 100 5 101 to 125 6 126 to 150 7 151 to 175 8 176 to 200 9	Determined by set point temperature (default=5 min)
STATIC TIME	The duration of the static solvent extraction phase.	0 to 2000 min (default=5)
CYCLES	The number of times the STATIC TIME and RINSE VOLUME steps are performed. When more than one cycle is specified, the RINSE VOLUME is divided by the number of cycles for each rinse step (see Example Method 1 on page 85).	1 to 9 (default=1)
RINSE VOLUME	The amount of solvent flushed through the sample cell after the STATIC TIME step, expressed as a percentage of the cell volume. For example, if the RINSE VOLUME is 50%, 0.5 mL is flushed through a 1-mL cell, 2.5 mL is flushed through a 5-mL cell, and so on.	0 to 150% vol in 5% increments (default=60)
PURGE	The amount of time the cell is purged with nitrogen gas. Thermo Fisher Scientific recommends the following settings: <ul style="list-style-type: none"> • For a 1-mL or 5-mL cell: 20 to 60 seconds • For a 10-mL or 22-mL cell: 40 to 80 (or 90) seconds • For a 34-mL cell: 70 to 110 seconds • For a 66-mL cell: 160 to 200 seconds • For a 100-mL cell: 250 to 290 seconds 	20 to 900 sec (default=100)

Table 3-3. Method Editor Parameters (Continued)

Parameter	Function	Value Range
SOLVENT A, B, C	<p>The solvent ratio and type.</p> <ul style="list-style-type: none"> When more than one solvent reservoir is installed, program the mixture of solvents in ratios. For example, to obtain a mix containing one-sixth solvent A, one-third solvent B, and one-half solvent C, enter ratios of 1, 2, and 3 for solvents A, B, and C, respectively. Solvent types are listed on page 82. 	0 to 5 (default= SOLVENT A, 1; SOLVENT B, 0; SOLVENT C, 0)
CELL TYPE	The cell type: SST (stainless steel) or Zr (zirconium).	SST (default) Zr
CELL SIZE	<p>Specifies the cell size. This field is displayed only when solvent saver flow mode is selected.</p> <p>Note: The cell size determines the flow rate range (for details, see “Notes on Solvent Saver Mode” on page 82).</p>	1, 5, 10, 22, 34, 66, or 100 mL (SST cells); 66 or 100 mL (Zr cells)
SOLVENT SAVER	<p>Selects the solvent saver mode (or turns it off). There are two options:</p> <ul style="list-style-type: none"> Select PRESS (the pressure mode) to maintain a fixed cell pressure and minimize solvent usage by eliminating the introduction of fresh solvent during the heating and static cycles. Select FLOW (the flow mode) to have small aliquots of solvent delivered to the cell at the specified flow rate throughout the static step. 	OFF (default) PRESS, FLOW
FLOW	<p>Selects the solvent flow rate (in increments of 0.5 mL). This field is displayed only when solvent saver flow mode is selected.</p> <p>Note: The flow rate range is determined by the cell size (for details, see “Notes on Solvent Saver Mode” on page 82).</p>	1.0 to 5.0 mL/min (default=1)

Table 3-3. Method Editor Parameters (Continued)

Notes on Solvent Saver Mode

- Selecting solvent saver mode automatically resets the **RINSE VOLUME** field to zero and the **CYCLES** field to 1. You can change the rinse volume, but not the number of cycles.
- When solvent saver flow mode is selected, the cell size determines the flow rate range (see the table below). If a change to the **CELL SIZE** field means that the previously selected flow rate is too high, the flow rate is automatically reset to the maximum flow rate for the new cell size. Conversely, if the previously selected flow rate is now too low, the flow rate is reset to the lowest flow rate for the new cell size.

Cell Size	Flow Rate Range
1 mL, 5 mL	0.5 to 2.0 mL/min
10 mL, 22 mL	1.0 to 2.0 mL/min
34 mL	1.0 to 3.0 mL/min
66 mL	1.0 to 4.0 mL/min
100 mL	1.0 to 5.0 mL/min

- When solvent saver flow mode is selected, the default flow rate is 1.0 mL/min. If you turn off solvent saver mode (or switch to pressure mode), the **FLOW** field is no longer displayed.
- When solvent saver pressure mode is selected (or solvent saver mode is off), the **CELL SIZE** and **FLOW** fields are not displayed. Switching to flow mode automatically displays these fields and resets them as follows:

The **CELL SIZE** field will display the value last selected (or 34 mL, if there is no previous selection).

The **FLOW** field will display the default value of 1.0 mL/min.

Entering Solvent Types and Ratios

The **METHOD EDITOR** screen lists some of the most common solvents for extractions. From the list, select the type(s) and ratio of solvent(s) used in each method. If the solvent you plan to use is not listed, accept the default setting: **Solvent Type**.

When more than one solvent reservoir is installed, these selections determine the type and amount of solvent delivered to the Dionex ASE

350. If only one reservoir is installed, enter 1 for that solvent and enter zero for the other solvents.



Do not use solvents with an autoignition point below 200 °C. If there is a question about solvent suitability, contact Technical Support for Dionex products.



N'utilisez pas de solvants ayant un point éclair inférieur à 200 °C. Le tableau ci-dessous liste quelques solvants qui ne doivent pas être utilisés avec le Dionex ASE 350. Contactez Thermo Fisher Scientific si vous avez un doute concernant l'usage d'un solvant absent de la liste.



Verwenden Sie keine Lösungsmittel, deren Selbstentzündungstemperatur unter 200 °C liegt. Bei Fragen zur Eignung von Lösungsmitteln wenden Sie sich bitte an Thermo Fisher Scientific.

To select a solvent type:

1. Move the cursor to the **SOLVENT** field and press **Select Δ** or **Select ▾** to scroll through a list of solvents that can be used with the Dionex ASE 350. These are the solvent options:

Acetone	Hexane	MeCl ₂	Toluene
Acetonitrile	iso-Octane	Pet Ether	Water
Chloroform	iso-Propanol	PERC	Water/Acid
Ethanol	Methanol	THF	Water/Base

2. After selecting the solvent type, press the left cursor arrow to go to the **RATIO** field for the installed solvent and enter a value from 0 to 5.
3. If more than one solvent will be used, enter the solvent type and ratio in each additional **RATIO** field.

Each solvent will be mixed according to the ratio of the number entered for that solvent divided by the sum of numbers entered for all solvents. For example:

- If you enter a ratio of 1 for all three solvents, the result is a mix containing one-third each of solvents A, B, and C.

- If you enter ratios of 1, 2, and 3 for solvents A, B, and C, respectively, the result is a mix that is one-sixth solvent A, one-third solvent B, and one-half solvent C.

NOTE If you enter a ratio of zero for all three solvents, the Dionex ASE 350 will override these selections and the default setting (SOLVENT A and a ratio of 1) will remain in effect.

Editing Methods

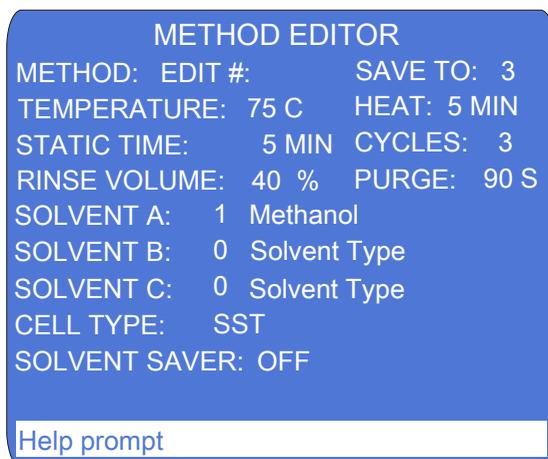
After creating a method, you can modify it by changing parameter values. When you finish, you have two options: you can save the changes to the existing method number or save the modified method to a *new* method number and preserve the original method.

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **3** to display the **METHOD EDITOR** screen.
2. In the **METHOD EDIT #** field, enter the number of the method to be edited (1 through 24), or press a **Select** button to scroll through the numbers of previously stored methods.
3. After selecting the method number, press **Enter**. The method's defined parameters appear.
4. To change a parameter value, move the cursor to the field and enter the new value. This replaces the previous value. [Table 3-3](#) lists the values allowed for each parameter.
5. When changes are complete, move the cursor to the **METHOD SAVE TO** field. Press **Enter** to save the changes to the current method number, or select a new method number and press **Enter**.

3.2.2 Example Methods

Example Method 1: Standard Extraction Mode

[Figure 3-20](#) illustrates the **METHOD EDITOR** screen for Example 1.



```
METHOD EDITOR
METHOD:  EDIT #:          SAVE TO:  3
TEMPERATURE: 75 C      HEAT:  5 MIN
STATIC TIME:   5 MIN   CYCLES:  3
RINSE VOLUME: 40 %    PURGE:  90 S
SOLVENT A:   1  Methanol
SOLVENT B:   0  Solvent Type
SOLVENT C:   0  Solvent Type
CELL TYPE:   SST
SOLVENT SAVER: OFF
Help prompt
```

Figure 3-20. Example Method 1: Standard Mode

- When you load the method (or a sequence using this method), the oven begins heating to 75 °C.
- To start the run, press **Start** any time after the method (or sequence) is loaded.
- The collection vessel is loaded.
- When the oven reaches the set point, the cell is moved into the oven and the pump fills the cell with solvent A. The static valve remains closed, and the pump continues pumping until the pressure reaches 10.34 MPa (1500 psi).
- The initial fill step and 5-minute heat step occur, followed by the first 5-minute static step.
- After the static step, the static valve opens and the pump rinses 13% of the cell volume of fresh solvent through the cell. This amount is approximately one-third of the total of 40% to be rinsed through in the three cycles.
- The static and cell rinse cycles repeat two more times.

- The method ends with a 90-second nitrogen purge step.

To create Example Method 1:

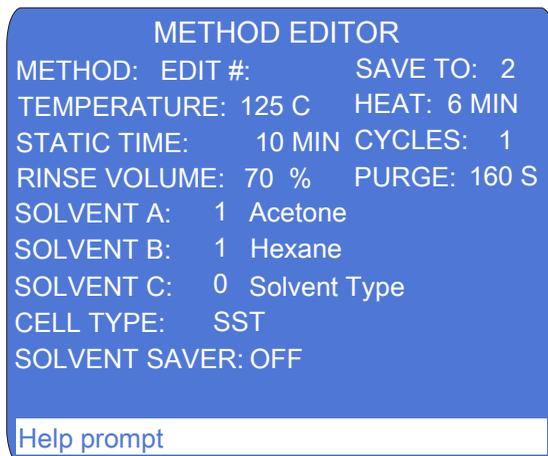
1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **3** to display the **METHOD EDITOR** screen.
2. With the cursor positioned in the **METHOD EDIT** field, enter zero to display the default method parameters.
3. Move the cursor to the following fields and enter the values listed here:

TEMPERATURE	75 °C
CYCLES	3
RINSE VOLUME	40%
PURGE	90 S
SOLVENT A	1
Solvent Type	Methanol

4. Move the cursor to the **SAVE TO** field and enter 3 for the new method. Press **Enter**.

Example Method 2: Standard Extraction Mode

[Figure 3-21](#) illustrates the **METHOD EDITOR** screen for Example 2.



METHOD EDITOR

METHOD: EDIT #:	SAVE TO: 2
TEMPERATURE: 125 C	HEAT: 6 MIN
STATIC TIME: 10 MIN	CYCLES: 1
RINSE VOLUME: 70 %	PURGE: 160 S
SOLVENT A: 1 Acetone	
SOLVENT B: 1 Hexane	
SOLVENT C: 0 Solvent Type	
CELL TYPE: SST	
SOLVENT SAVER: OFF	

Help prompt

Figure 3-21. Example Method 2: Standard Mode

- When you load the method (or a sequence using this method), the oven begins heating to 125 °C.
- To start the run, press **Start** any time after the method (or sequence) is loaded.
- The collection vessel is loaded.
- When the oven reaches the set point, the cell is moved into the oven and the pump fills the cell with equal parts of solvents A and B. The static valve closes and the pump continues pumping until the pressure reaches 10.34 MPa (1500 psi).
- The initial 6-minute heat step occurs, followed by a 10-minute static step.
- After the static step, the static valve opens and the pump rinses 70% of the cell volume of fresh solvent through the cell.
- The method ends with a 160-second nitrogen purge step.

Example Method 3: Solvent Saver Pressure Mode

[Figure 3-22](#) illustrates the **METHOD EDITOR** screen for Example 3.



```
METHOD EDITOR
METHOD:  EDIT #:          SAVE TO:  1
TEMPERATURE: 125 C      HEAT:  6 MIN
STATIC TIME:   15 MIN   CYCLES:  1
RINSE VOLUME:  0 %     PURGE: 120 S
SOLVENT A:    4 Hexane
SOLVENT B:    1 Acetone
SOLVENT C:    0 Solvent Type
CELL TYPE:    SST
SOLVENT SAVER: PRESS

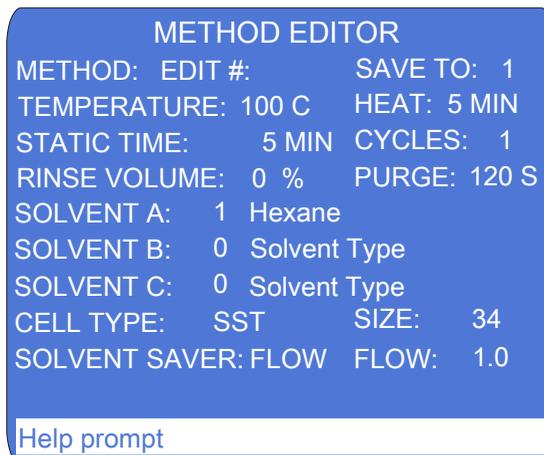
Help prompt
```

Figure 3-22. Example Method 3: Pressure Mode

- When you load the method (or a sequence using this method), the oven begins heating to 125 °C.
- To start the run, press **Start** any time after the method (or sequence) is loaded.
- The collection vessel is loaded.
- When the oven reaches the set point, the cell is moved into the oven. The pump begins filling the cell with solvent at the full flow rate (as in standard mode). When the pressure reaches 10.34 MPa (1500 psi), the pump is turned off.
- The initial 6-minute heat step occurs, followed by a 15-minute static step.
- After the static step, the static valve opens.
- The method ends with a 120-second nitrogen purge step.

Example Method 4: Solvent Saver Flow Mode

[Figure 3-23](#) illustrates the **METHOD EDITOR** screen for Example 4.



```
METHOD EDITOR
METHOD:  EDIT #:          SAVE TO:  1
TEMPERATURE: 100 C      HEAT:  5 MIN
STATIC TIME:   5 MIN    CYCLES:  1
RINSE VOLUME:  0 %     PURGE: 120 S
SOLVENT A:    1 Hexane
SOLVENT B:    0 Solvent Type
SOLVENT C:    0 Solvent Type
CELL TYPE:    SST       SIZE:   34
SOLVENT SAVER: FLOW    FLOW:   1.0
Help prompt
```

Figure 3-23. Example Method 4: Flow Mode

- When you load the method (or a sequence using this method), the oven begins heating to 100 °C.
- To start the run, press **Start** any time after the method (or sequence) is loaded.
- The collection vessel is loaded.
- When the oven reaches the set point, the cell is moved into the oven. The pump begins filling the cell with solvent at the full flow rate (as in standard mode). When the pressure reaches 10.34 MPa (1500 psi), the pump is turned off.
- The initial 5-minute heat step begins. When the pressure reaches 11.72 MPa (1700 psi), the static valve opens to release some solvent.
- This is followed by a 5-minute static step. During the static step, the static valve opens at 9.65 MPa (1400 psi) and the pump delivers flow at 1.0 mL/min.
- After the static step, the static valve opens.
- The method ends with a 120-second nitrogen purge step.

3.2.3 Creating Sequences

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **4** to display the **SEQUENCE EDITOR** screen.

When the screen is opened for the first time after power-up, it displays the default sequence parameters (see [Figure 3-24](#)). Thereafter, the screen displays the last sequence that was edited (if any) or the default parameters (if there is no previous sequence).

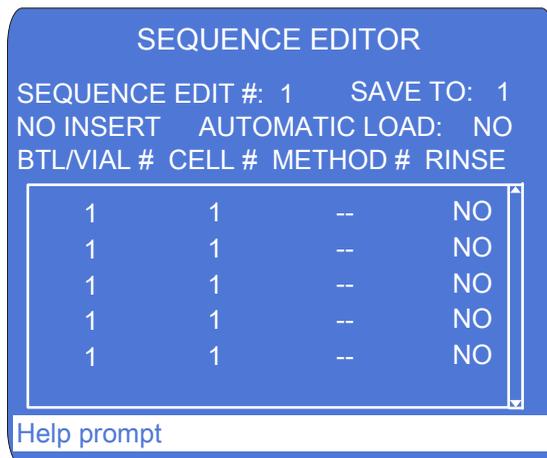


Figure 3-24. Sequence Editor Screen (Default)

Each line in the **SEQUENCE EDITOR** represents one sample extraction run. (The maximum number of runs per sequence is 33.)

To scroll through the sequence, press the down arrow button repeatedly. When the cursor reaches the bottom line on the screen, the next line scrolls up.

2. For each line in a sequence, enter the required sequence information (see [Table 3-4](#)).
3. When you finish editing, move the cursor to the **SEQUENCE SAVE TO** field. Enter a number for the new sequence (1 through 24), or press **Select** Δ to display the next unused sequence number and press **Enter** to save the sequence.

Sequence Field	Description	Values
SEQUENCE EDIT #	The number of the sequence to edit. Enter the number on the numeric keypad, or press a Select button to step through the numbers of all existing sequences. To display the default sequence values, press zero.	0, 1 through 24 (default=1)
SEQUENCE SAVE TO	The sequence number that your editing changes will be saved to. Enter the number on the numeric keypad, or press a Select button to step through all of the unused sequence numbers.	1 through 24 (default=1)
NO INSERT	Indicates whether a vial tray insert (P/N 066176) is installed. The insert is required to accommodate collection vials (see page 68).	NO INSERT, INSERT (default= NO INSERT)
AUTOMATIC LOAD	Selecting YES automatically loads the sequence currently displayed and turns on the top plate and oven heaters. This eliminates the need to exit the SEQUENCE EDITOR and load the sequence from the LOAD METHOD/SEQUENCE screen (see Section 3.5.2). To run the sequence immediately, press Start .	YES =Load sequence, NO =Do not load sequence
BTL/VIAL #	The number of the collection bottle or vial to be used. Note: Any line in the sequence that includes a zero in the BTL/VIAL # field is defined as the last line of the sequence. The Dionex ASE 350 will ignore any parameters on this line, as well as all lines after this.	No vial tray insert: 1–19 (bottles only) With a vial tray insert: 1–28 for vials; 29–33 for bottles (default=1)

Table 3-4. Sequence Editor Parameters

Sequence Field	Description	Values
CELL #	<p>The sample cell used for the run. You can use the default cell or enter a different cell number. For example, when performing multiple extractions on the same sample, you can assign one sample cell to multiple collection vessels (see Example Sequence 3 on page 97).</p> <p>Note: Any line in the sequence that includes a zero in the CELL # field is defined as the last line of the sequence. The Dionex ASE 350 will ignore any parameters on this line, as well as all lines after this.</p>	1 through 24 (default=1)
METHOD #	<p>The number of the method to run. If necessary, a different method can be run on each sample. Thermo Fisher Scientific recommends entering methods in a sequence from coolest to hottest. Because the oven heats up faster than it cools down, this minimizes the oven wait time between methods.</p> <p>Note: Any line in the sequence that includes a zero in the METHOD # field is defined as the last line of the sequence. The Dionex ASE 350 will ignore any parameters on this line, as well as all lines after this.</p>	1 through 24 (default= --)

Table 3-4. Sequence Editor Parameters (Continued)

Sequence Field	Description	Values
RINSE	<p>Determines whether the system is rinsed after each extraction:</p> <ul style="list-style-type: none"> • When YES is selected, a post-extraction rinse is performed with the solvent used in the method just completed. • When NO is selected, a post-extraction rinse is not performed. • When ON A, ON B, or ON C is selected, a post-extraction rinse is performed using solvent from reservoir A, B, or C, respectively. <p>If the next method in the sequence uses a different solvent than the method just completed, or a different solvent than the one used for the post-extraction rinse, the Dionex ASE 350 will automatically perform a pre-extraction rinse with the solvent specified in the method in the next line of the sequence. The automatic pre-extraction rinse occurs even if NO is selected here.</p>	<p>YES=rinse with last solvent used; NO=no rinse; ON A, ON B, ON C=rinse with specified solvent (default=NO)</p>

Table 3-4. Sequence Editor Parameters (Continued)

3.2.4 Example Sequences

Example Sequence 1

Run a different method (using 250-mL collection bottles) every six extractions. Do not rinse between extractions.

BTL/VIAL #	CELL #	METHOD #	RINSE
1	1	1	NO
2	2	1	NO
3	3	1	NO
4	4	1	NO
5	5	1	NO
6	6	1	NO
7	7	2	NO
8	8	2	NO
9	9	2	NO
10	10	2	NO
11	11	2	NO
12	12	2	NO
13	13	3	NO
14	14	3	NO
15	15	3	NO
16	16	3	NO
17	17	3	NO
18	18	3	NO
19	19	4	NO

To create Example Sequence 1:

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **5** to display the **SYSTEM SETUP** screen.
2. Verify that the **METHOD RINSE** field is **NO**. If necessary, move the cursor to the field and press **Select Δ** to toggle to **NO**. Press **Enter**.
3. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **4** to display the **SEQUENCE EDITOR** screen.
4. Verify that **NO INSERT** is displayed on the second line. If necessary, move the cursor to the field and press **Select Δ** to toggle to **NO INSERT**. Press **Enter**.
5. With the cursor positioned in the **SEQUENCE EDIT #** field, enter zero to display the default sequence parameters (see [Table 3-4](#)).
6. For the first line in the sequence, move the cursor to the **METHOD #** field and enter 1. This is the only parameter you need to enter here; all other parameters remain at their default values.

NOTE It is not necessary to change the **RINSE** field on any line of Example Sequence 1.

7. For the second line in the sequence, move the cursor to the following fields and enter the values listed here:

BTL/VIAL #	2
CELL #	2
METHOD #	1

8. For the third line in the sequence, move the cursor to the following fields and enter the values listed here:

BTL/VIAL #	3
CELL #	3
METHOD #	1

9. Repeat the steps above until all parameters have been selected. When you finish, move the cursor to the **SEQUENCE SAVE TO** field and enter a number for the new sequence. Press **Enter**.

Example Sequence 2

Run a different method every six extractions. Run three extractions on the last cell. Rinse between extractions.

BOTTLE #	CELL #	METHOD #	RINSE
1	1	1	YES
2	2	1	YES
3	3	1	YES
4	4	1	YES
5	5	1	YES
6	6	1	YES
7	7	2	YES
8	8	2	YES
9	9	2	YES
10	10	2	YES
11	11	2	YES
12	12	2	YES
13	13	3	YES
14	14	3	YES
15	15	3	YES
16	16	3	YES
17	17	3	YES
18	17	3	YES
19	17	4	YES

Example Sequence 3

Run three extractions on each cell. Run a different method every six extractions. Rinse between samples.

BOTTLE #	CELL #	METHOD #	RINSE
1	1	1	YES
2	1	1	YES
3	1	1	YES
4	2	1	YES
5	2	1	YES
6	2	1	YES
7	3	2	YES
8	3	2	YES
9	3	2	YES
10	4	2	YES
11	4	2	YES
12	4	2	YES
13	5	3	YES
14	5	3	YES
15	5	3	YES
16	6	3	YES
17	6	3	YES
18	6	3	YES

3.3 Methods and Sequences in Chromeleon Software

NOTES

- This section provides a brief description of the steps required to set up and run the Dionex ASE 350 with Chromeleon software. For detailed instructions, refer to the Chromeleon software Help.
- Most of the information in [Section 3.2](#) (including descriptions of method and sequence parameters, guidelines for selection of solvent types, and details about solvent saver modes) applies to all Dionex ASE 350 methods and sequences. Therefore, it is advisable to review this information before using Chromeleon software to create methods and sequences.

3.3.1 Overview

1. Create an instrument method that specifies the functions the Dionex ASE 350 will perform. You can create methods automatically (with the help of the Instrument Method Wizard) or manually (by editing an existing method).
 - When the Dionex ASE 350 is configured in Chromeleon software, the Instrument Method Wizard includes pages (**Rinse Settings** and **Extraction Method**) specifically for the Dionex ASE 350.
 - After creating the instrument method, specify options related to the preheating and initial heat-up steps on the **System Setup** page in the Instrument Method Editor.
2. Create a sequence (a list of extractions). For each extraction, specify the starting cell and vial positions and the number of extractions to be performed. You can create sequences automatically (with the help of the Sequence Wizard) or manually (by editing an existing sequence).

NOTE Chromeleon software can run up to 456 extractions per sequence. In Local mode, the Dionex ASE 350 can run up to 33 extractions per sequence.

3. Load the sequence into a queue by clicking **Start** on the Sequence Control Bar (above the extraction list).
 - If this is the only sequence in the queue, it will start immediately.
 - If the queue already includes one or more sequences, the new sequence will be appended to the queue.
4. When a queue starts, Chromeleon software automatically performs a Ready Check procedure to verify that the instrument is ready for operation and that the methods used to run the sequences are error-free.
 - If no problems are detected, the following message is displayed:
Ready Check result: Successful.
 - If a problem is detected, an error message is displayed. See [Section 4.1](#) for a list of possible error messages, as well as the appropriate corrective action to take. Most problems can be resolved by the user.

NOTE For instructions on how to run a Ready Check manually, refer to the Chromeleon software Help.

3.3.2 Example Methods

Example Method 5: Solvent Saver Fixed Volume Mode

NOTE Chromeleon software control is required to run fixed volume methods.

[Figure 3-25](#) illustrates the **Extraction Method** page for Example 5. In this method, the solvent volume is less than the cell volume.

Instrument Method Wizard - Extractor (ASE 350: Extractor): Extraction Method

Extraction Method

Mode: FixedVolume Cell Type: SST

Oven Temperature

Off

On 150 [40...200 °C]

At this temperature, the cell will be heated for 7 minutes to ensure that the sample reaches thermal equilibrium.

Static Cycle

Time: 10 [0...2000 min]

Cycles: 1 [1...9]

Rinse Volume: 0 [0...150 %]

Purge

Time: 100 [20...900 s]

Solvents

Type	Ratio
A: Solvent_A	1 [0...5]
B: Solvent_B	0 [0...5]
C: Solvent_C	0 [0...5]

Cell Size: 66mL [1mL...100mL]

Volume: 20 [2...175 mL]

< Back Next > Cancel Help

Figure 3-25. Example Method 5: Fixed Volume Mode

- After creating a sequence that contains this method, click **Start** on the Sequence Control Bar (above the extraction list) to start the run.
- The collection vessel is loaded.
- The oven begins heating to 150 °C.
- When the oven reaches the set point, the cell is moved into the oven. The pump begins filling the cell with solvent A; when 20 mL of solvent A has been delivered, the pump is turned off.
- The extraction proceeds through the remaining fill time, the 5-minute heat step, and the 20-minute static step. Because the solvent volume is less than the cell volume, the pressure will not reach 11.72 MPa (1700 psi) and the static valve will remain closed.
- After the static step, the static valve opens.
- The method ends with a 100-second nitrogen purge step.

Example Method 6: Solvent Saver Fixed Volume Mode

NOTE Chromeleon software control is required to run fixed volume methods.

[Figure 3-26](#) illustrates the **Extraction Method** page for Example 6. In this method, the solvent volume is greater than the cell volume.

Instrument Method Wizard - Extractor (ASE 350: Extractor): Extraction Method

Extraction Method

Mode: FixedVolume Cell Type: SST

Oven Temperature

Off

On 125 [40...200 °C]

At this temperature, the cell will be heated for 6 minutes to ensure that the sample reaches thermal equilibrium.

Static Cycle

Time: 5 [0...2000 min]

Cycles: 1 [1...9]

Rinse Volume: 0 [0...150 %]

Purge

Time: 60 [20...900 s]

Solvents

Type	Ratio
A: Solvent_A	0 [0...5]
B: Solvent_B	1 [0...5]
C: Solvent_C	0 [0...5]

Cell Size: 34mL [1mL...100mL]

Volume: 50 [2...175 mL]

< Back Next > Cancel Help

Figure 3-26. Example Method 6: Fixed Volume Mode

- After creating a sequence that contains this method, click **Start** on the Sequence Control Bar (above the extraction list) to start the run.
- The collection vessel is loaded.

- When the oven reaches the set point, the cell is moved into the oven and the pump fills the cell with solvent B. The static valve remains closed, and the pump continues pumping until the pressure reaches 10.34 MPa (1500 psi).
- The initial fill step and the 6-minute heat step occur, followed by the 5-minute static step.
- After the static step, the static valve opens and the pump continues until it has pumped a total of 50 mL.
- The method ends with a 60-second nitrogen purge step.

Example Method 7: Solvent Saver Gradient Mode

NOTE Chromeleon software control is required to run gradient methods.

Figure 3-27 illustrates the **Extraction Method** page for Example 7. In this method, a mixture of two solvents is delivered.

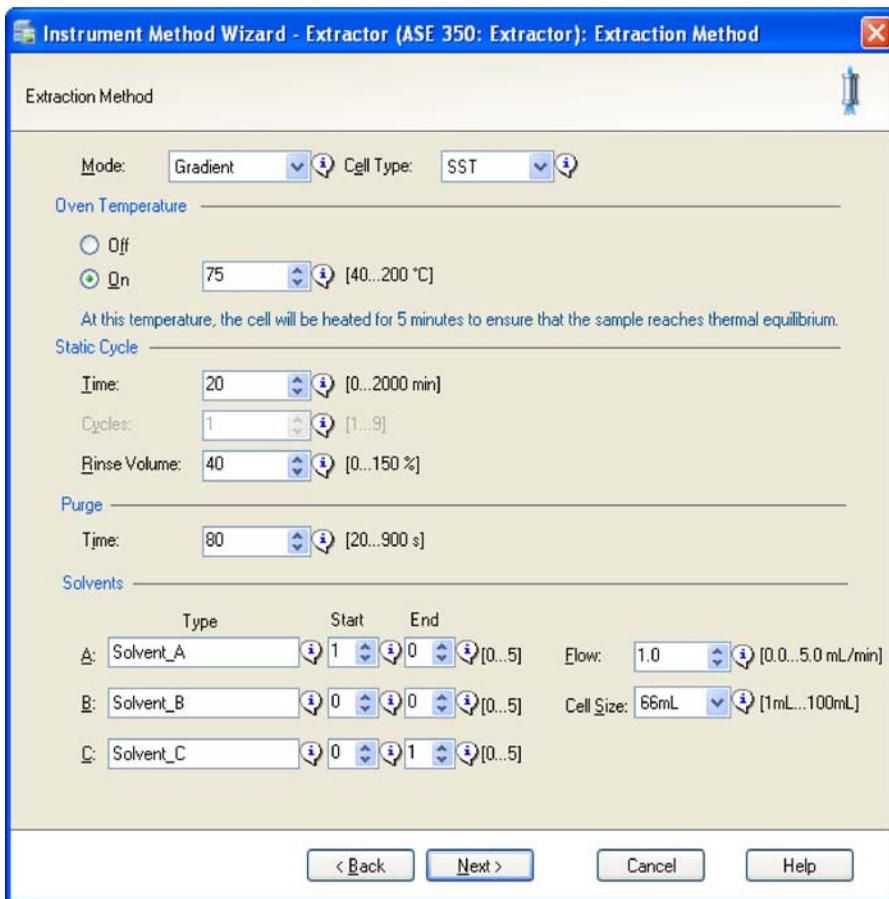


Figure 3-27. Example Method 7: Gradient Mode

- After creating a sequence that contains this method, click **Start** on the Sequence Control Bar (above the extraction list) to start the run.
- The collection vessel is loaded.

- When the oven reaches the set point, the cell is moved into the oven and the pump fills the cell with solvent A. The static valve remains closed, and the pump continues pumping until the pressure reaches 10.34 MPa (1500 psi).
- The initial fill step and the 5-minute heat step occur.
- During the static step, the pump delivers 20 mL of solvent (20 minutes \times 1.0 mL/min). At the beginning of the static step, the gradient flow consists of 100% solvent A. At 10 minutes into the static step, the pump delivers equal amounts of solvent A and solvent C. At the end of the static step, the pump is delivering solvent C only.
- After the static step, the static valve opens and the pump delivers about 26 mL (40% of the 66-mL cell) of solvent C.
- The method ends with an 80-second nitrogen purge.

Example Method 8: Solvent Saver Gradient Mode

NOTE Chromeleon software control is required to run gradient methods.

Figure 3-27 illustrates the **Extraction Method** page for Example 8. In this method, a mixture of three solvents is delivered.

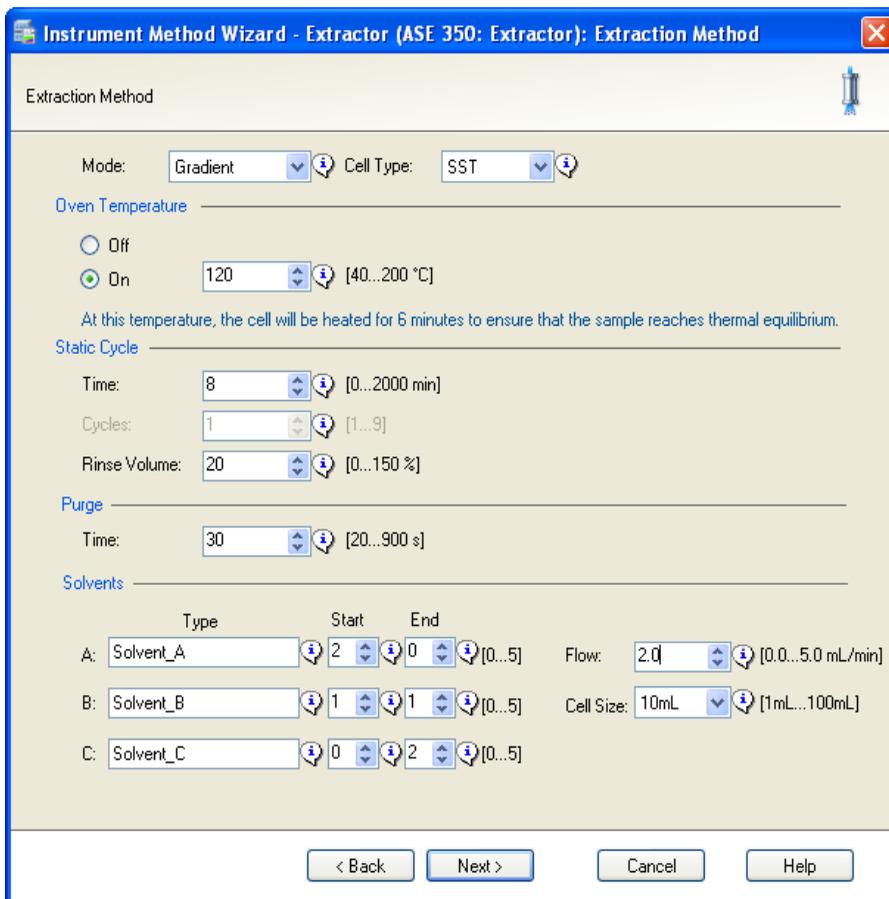


Figure 3-28. Example Method 8: Gradient Mode

- After creating a sequence that contains this method, click **Start** on the Sequence Control Bar (above the extraction list) to start the run.
- The collection vessel is loaded.

- When the oven reaches the set point, the cell is moved into the oven and the pump fills the cell with solvents A and B at a ratio of 2:1. The static valve remains closed, and the pump continues pumping until the pressure reaches 10.34 MPa (1500 psi). The initial fill step and the 6-minute heat step occur.
- During the static step, the pump delivers 16 mL of solvent (8 minutes \times 2.0 mL/min). At the beginning of the static step, the gradient flow consists of solvents A and B at a ratio of 2:1. At 4 minutes into the static step, the pump delivers equal amounts of solvents A, B, and C at a ratio of 1:1:1. At the end of the static step, the pump is delivering solvents B and C at a ratio of 1:2.
- After the static step, the static valve opens and the pump delivers about 2 mL (20% of the 10-mL cell) of solvents B and C at a ratio of 1:2.
- The method ends with a 30-second nitrogen purge step.

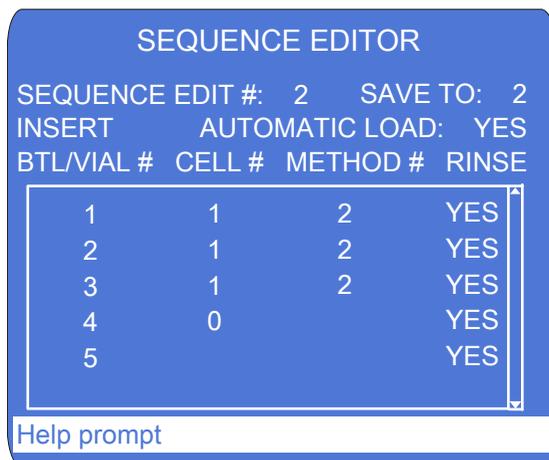
3.4 Method Development Guidelines

Follow the procedure here when developing a method for a new sample type. For more details about method development, see [Appendix E](#) of this manual.

In addition, you may want to refer to Thermo Scientific Dionex Technical Notes 208, 209, and 210 (available on the Thermo Fisher Scientific website).

1. Select the solvent(s) (see [Section 3.1.1](#)).
2. Prepare the sample (see [Section 3.1.3](#)).
3. Select the cell size (see [Section 3.1.4](#)).
4. Create a new method based on the default operating parameters.
 - a. On the **METHOD EDITOR** screen, position the cursor in the **METHOD EDIT #** field and enter zero to display the default parameters.
 - b. Move the cursor to the **METHOD SAVE TO** field and select a new method number (2, for example).
 - c. Press **Enter** to save the method.

5. Create a sequence that extracts the same sample three times into separate collection vessels (see the example in [Figure 3-29](#)). Specify the method created in [Step 4](#) for each extraction.



SEQUENCE EDITOR			
SEQUENCE EDIT #:	2	SAVE TO:	2
INSERT		AUTOMATIC LOAD:	YES
BTL/VIAL #	CELL #	METHOD #	RINSE
1	1	2	YES
2	1	2	YES
3	1	2	YES
4	0		YES
5			YES

Help prompt

Figure 3-29. Example Sequence for Method Development

6. Run the sequence and analyze the extracts.
7. If target analytes are present in collection vessel 2 or 3, make the following adjustments (*one at a time*) to the method:
 - a. Raise the temperature. In general, raising the temperature increases the efficiency of the extraction process. However, because compounds can degrade at high temperatures, it is advisable to raise the temperature in small increments (10 to 20 °C). The maximum allowable temperature of the Dionex ASE 350 is 200 °C. If oxidation is a concern, degas the solvent before use.
 - b. Run two static cycles. Extending the static time enhances diffusion of the analytes into the extraction fluid. Separating the static time into two cycles, rather than using one longer cycle, allows the introduction of fresh solvent midway through. The fresh solvent helps maintain a favorable solvent/sample equilibrium for samples that are heavily loaded, or otherwise difficult to extract.

- c. Increase the rinse volume to allow more solvent to pass through the sample. The rinse volume can be up to 150% of the cell volume.

NOTE To prevent overflowing of the 34-mL and 100-mL cells or the collection bottles or vials, it may be necessary to balance the number of static cycles and the rinse volume.

8. After each adjustment, repeat the sequence and analyze the extract. When target analytes no longer appear in the extract from the second collection vessel, the method is complete for this sample type.
9. If analytes still appear in the extract from the second collection vessel, make the following adjustments (*one at a time*) to the method:
 - Run three static cycles.
 - Raise the temperature again.
 - Extend the static time.
 - Select a different solvent.

When target analytes no longer appear in the extract from the second collection vessel, the method is complete for this sample type.

3.5 Operating the Dionex ASE 350

IMPORTANT

Before beginning a run, check that the door to the service area (see [Figure 2-1](#)) is closed. If the door is open during operation, the oven may not reach and maintain the set temperature.

3.5.1 Operating Under Method Control

Before starting a run, decide whether to create a new method to define the operating conditions (see [Section 3.2.1](#)) or use the default method provided with the Dionex ASE 350.

The default method enables you to get started running quickly, without having to create a custom method. However, before you can run a default method, be sure to assign a new method number to it (see below).

To renumber the default method:

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **3** to display the **METHOD EDITOR** screen.
2. With the cursor in the **METHOD EDIT #** field, enter zero to display the default parameters (see [Figure 3-30](#)).
3. For each solvent selected, check that the corresponding solvent reservoir is installed.
4. Move the cursor to the **METHOD SAVE TO** field, press **Select Δ** to display the next unused method number, and press **Enter**. The new method can now be loaded and run (see below).

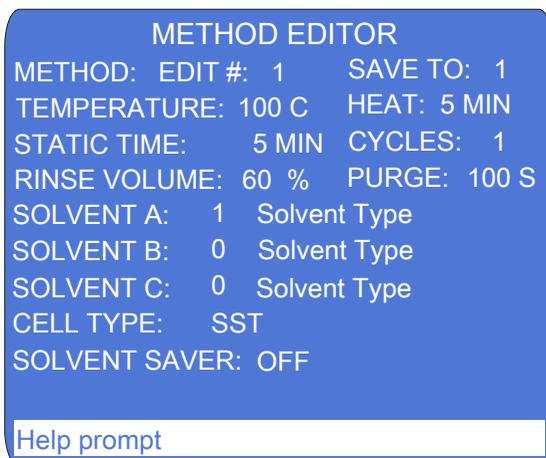
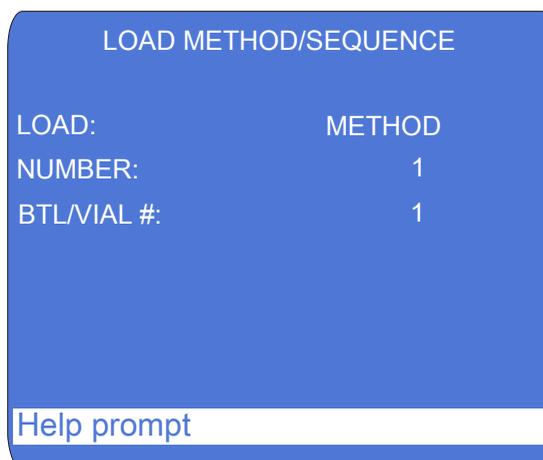


Figure 3-30. Method Editor Screen (Default)

To load a method and start the run:

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **1** to display the **LOAD METHOD/SEQUENCE** screen (see [Figure 3-31](#)).



LOAD:	METHOD
NUMBER:	1
BTL/VIAL #:	1

Help prompt

Figure 3-31. Load Method/Sequence Screen

2. Verify that **METHOD** appears in the **LOAD** field. If it does not, press a **Select** button to toggle to **METHOD**.
3. Move the cursor to the **NUMBER** field and enter the number of the method. Press **Enter** or a cursor arrow.
4. The number selected in the **BTL/VIAL #** field applies to both the collection vessel and the sample cell. By default, the method begins running at collection vessel 1 and cell 1.
 - To start the run at a different collection vessel and cell, move the cursor to the **BTL/VIAL #** field, enter the preferred number, and press **Enter**. This closes the **LOAD METHOD/SEQUENCE** screen and opens the **SYSTEM STATUS** screen.
 - If you do not need to edit the **BTL/VIAL #** field, open the **SYSTEM STATUS** screen by moving the cursor to the **BTL/VIAL #** field and pressing **Enter**.

The **SYSTEM STATUS** screen displays the method number, as well as the status of several other Dionex ASE 350 operating parameters (see [Figure 3-32](#)).

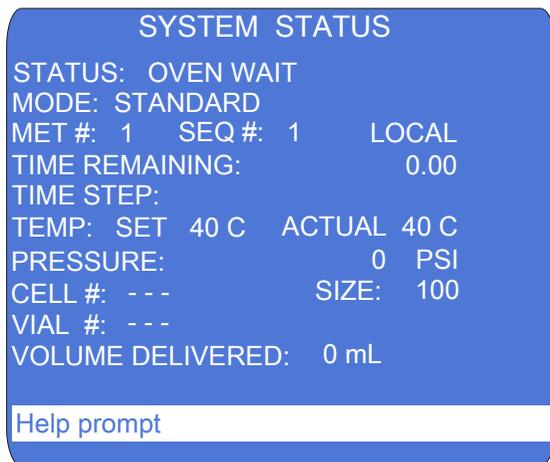


Figure 3-32. System Status Screen

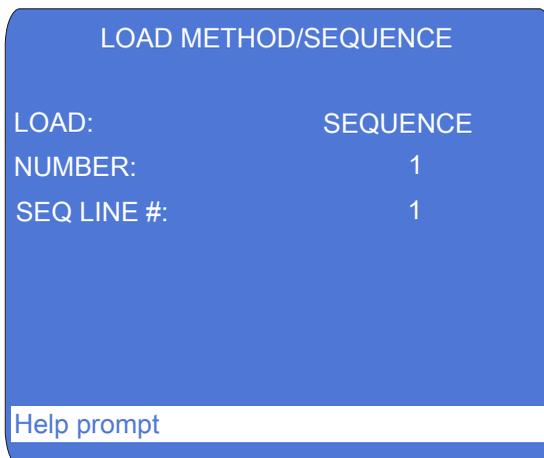
NOTE The temperature set point is activated immediately after the method is loaded (before you press **Start**). If the current oven temperature is below the set point, the oven begins heating.

5. Press **Start** to begin the run.
 - If the oven has not reached the set point, there is a delay while the oven heats up or cools down. During this time, the **SYSTEM STATUS** screen displays **OVEN WAIT**.
 - When the oven is within 1 °C of the set point, the cell and collection trays rotate to the first positions and the Dionex ASE 350 begins running the first sample.
 - When running under method control, the Dionex ASE 350 runs samples sequentially until it reaches an empty cell or collection vessel position or, if the trays are full, until all samples have been run and the trays have returned to positions 1. For a detailed description of each step of the extraction process, see [Section 2.6](#).

- The status of the operating parameters displayed on the **SYSTEM STATUS** screen is updated every 1 second during the run.

3.5.2 Operating Under Sequence Control

1. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **1** to display the **LOAD METHOD/SEQUENCE** screen (see [Figure 3-33](#)).



LOAD:	SEQUENCE
NUMBER:	1
SEQ LINE #:	1

Help prompt

Figure 3-33. Load Method/Sequence Screen

2. Verify that **SEQUENCE** is displayed in the **LOAD** field. If necessary, press a **Select** button to toggle to **SEQUENCE**.
3. Move the cursor to the **NUMBER** field and enter the number of the sequence to run. Press **Enter** or a cursor arrow.
4. By default, the sequence begins running at the first line of the sequence.
 - To start the run at a different line, move the cursor to the **SEQ LINE #** field and enter the preferred number. Press **Enter**. This closes the **LOAD METHOD/SEQUENCE** screen and opens the **SYSTEM STATUS** screen.
 - If you do not need to edit the **SEQ LINE #** field, open the **SYSTEM STATUS** screen by moving the cursor to the **SEQ LINE #** field and pressing **Enter**.

The **SYSTEM STATUS** screen displays the method number, as well as the status of several other Dionex ASE 350 operating parameters (see [Figure 3-32](#)).

NOTE The temperature set point for the first method in the sequence is activated immediately after the sequence is loaded (before you press **Start**). If the current oven temperature is below the set point, the oven begins heating.

5. Press **Start**. This initiates the following series of events:
 - The Dionex ASE 350 begins the first run according to the parameters in the first method in the sequence.
 - When the first extraction run is complete, the Dionex ASE 350 proceeds to the next line in the sequence, and then continues until all sequenced runs are completed.
 - When the methods in a sequence have different temperature set points or use cells of different sizes, the sequence is delayed each time the oven is required to heat up before the cell is moved into the oven. During this delay, the **STATUS** field on the **SYSTEM STATUS** screen displays **OVEN WAIT**.

NOTE Any line in the sequence that includes a zero in the **BTL/VIAL #**, **CELL #**, or **METHOD #** field is defined as the last line of the sequence. The Dionex ASE 350 will ignore any parameters on this line and all lines after this.

3.5.3 Stopping a Run

The appropriate method of stopping a run depends on the Dionex ASE 350 operating mode. When you stop a run (regardless of the method used), the pump turns off, valves close, and all flow stops.

To stop a run while in Local mode:

To interrupt the current run when the Dionex ASE 350 is in Local mode, press the **Stop** button on the front panel. The **ABORT REQUEST** screen appears (see [Figure 3-34](#)).

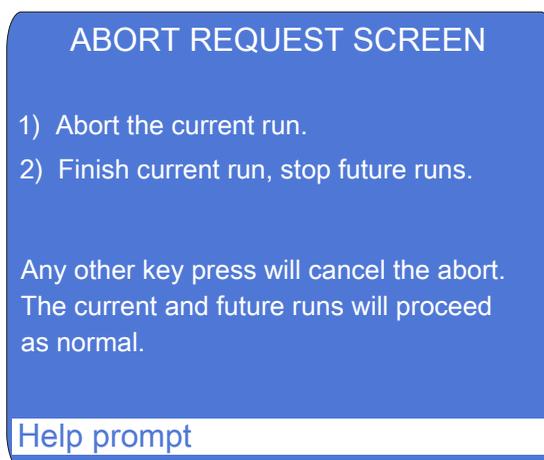


Figure 3-34. Abort Request Screen

Press the button that selects the preferred option:

- 1 Abort the current run.** This option cancels the method or sequence. If necessary, a purge step is performed to remove solvent from the sample cell. Then, residual pressure is relieved from the system and the current cell is returned to the tray.
 - 2 Finish current run, stop future runs.** This option cancels the method or sequence after the current run (and rinse, if programmed) is completed.
- Any other key press will cancel the abort process. The current and future runs will proceed as usual.** Pressing any keypad button other than **1** or **2** resumes the run at the point at which you pressed **Stop**.

To stop a run while in Remote mode:

There are two ways to interrupt the current run when the Dionex ASE 350 is in Remote mode:

- Press the **Stop** button on the Dionex ASE 350 ePanel in Chromeleon software. If an extraction or a rinse is in progress, the function is aborted and the Dionex ASE 350 returns to the idle state. If the Dionex ASE 350 is already in the idle state, pressing the button has no effect.

NOTE When you press the front panel Stop button, Chromeleon software logs the event in the audit trail.

- Press the **Stop** button on the Dionex ASE 350 front panel. The **ABORT REQUEST** screen appears (see [Figure 3-34](#)). Select the preferred option.

3.6 Performing Post-Run Procedures

3.6.1 Cleaning the Sample Cells



Cells are extremely hot after an extraction. Allow cells to cool for at least 15 minutes before handling. Be especially cautious with cells that have been heated over 50 °C.



Les cellules sont extrêmement chaudes après une extraction. Laissez-les refroidir pendant au moins 15 minutes avant de les manipuler. Faites particulièrement attention aux cellules qui ont été chauffées à plus de 50 °C.



Die Zellen sind nach ihrer Entnahme sehr heiß. Lassen Sie die Zellen mindestens 15 Minuten abkühlen. Seien Sie besonders vorsichtig, wenn Zellen über 50 °C erhitzt wurden.

After use, empty the cells and rinse the cell bodies and end caps with water or organic solvent.

- Both stainless steel and zirconium cell bodies (but not the cell end caps) can be cleaned in a dishwasher or high temperature cleaning

unit. Do not exceed 200 °C when performing high temperature cleaning.

- For most applications, simply rinsing the end caps is sufficient. If necessary, disassemble the end caps (see [Section 5.2](#)) and sonicate or soak in solvent to clean them.
- Do not use detergent to clean the end cap frits.

3.6.2 Processing Extracts

The composition of the extracts generated by the Dionex ASE 350 is identical or equivalent to that generated by Soxhlet and other standard solid-liquid extraction techniques when using the same solvent.

Therefore, you may use the same analytical method for Dionex ASE 350 extracts that was employed for extracts obtained from other techniques.

3.7 Performing Routine Maintenance

This section describes routine Dionex ASE 350 maintenance procedures that users can perform. All procedures not included here must be performed by qualified Thermo Fisher Scientific personnel.

3.7.1 Daily Maintenance

- Check that the nitrogen gas and compressed air supplies are adequate for the day.
- Fill the solvent reservoirs, if needed.
- After filling a reservoir, run two rinse cycles to clear the solvent line of air (see [Section 3.1.9](#)).
- When using acids, bases, salts, or buffers as solvents, rinse the system with deionized water at the end of daily operation.
- Empty the waste bottle, if needed.
- Empty both rinse bottles.
- Check for leaks from the solvent reservoirs, pump, valves, and other components in the flow path. Wipe up liquids, and tighten or replace leaking fittings (see [Section 4.2](#) and [Section 5.1](#)).

- If the septum in a rinse bottle cap is pierced during a run, install a new septum before starting the next series. The rinse bottle septum is pierced during rinse cycles (manual and automatic) and whenever the collection vessel is full.

3.7.2 Periodic Maintenance

- Replace the external O-rings (PTFE: P/N 049457, pkg. of 50; Viton: P/N 056325, pkg. of 50) in the cell end caps and rinse tubes approximately every 50 to 75 extractions.

NOTE The number of extractions the Dionex ASE 350 has performed is displayed on the USAGE LOG screen (see [Section C.4](#)) and the Dionex ASE 350 Wellness panel in Chromeleon software (see [Section 2.5.3](#)).

- Replace the PEEK seals (P/N 061687, pkg. of 50) inside the cell end caps after approximately every 50 extractions (see [Section 5.2](#)).
- Verify that the three needles on the needle mechanism are straight. To replace the source needle, follow the instructions in [Section 5.8](#). To replace the vent needles, contact Technical Support for Dionex products.
- Replace the O-ring, filter, and seals in the static valve every 6 to 12 months (see [Section 5.7](#)).
- Clean the exterior of the system with water and a mild detergent.

3.7.3 Annual Maintenance

- Thermo Fisher Scientific recommends performing preventive maintenance annually. The Dionex ASE 150/Dionex ASE 350 Preventive Maintenance Kit (P/N 068954) contains all the required parts for the procedure.
- If you frequently extract acidic or basic samples, consider having the solvent lines replaced at the same time the annual preventive maintenance is performed. The solvent lines must be replaced by qualified Thermo Fisher Scientific personnel.

3.8 Shutting Down

Overnight Shutdowns

- If acids, bases, salts, or buffers are used as extraction solvents, rinse the system with 100% polar organic solvent (for example, acetone or methanol) or distilled water before turning off the power.
- After extracting with a 100% organic solvent, you may turn off the power immediately (no system rinse is required).

Longer Shutdowns

Before shutting down the system for more than one night:

- After extracting with a solvent that contains acids, bases, or other strong additives, rinse the system with either 100% organic solvent or distilled water (see [Section 3.1.9](#)) before turning off the power. Also turn off the nitrogen gas and compressed air supplies.
- After extracting with a 100% organic solvent, you may turn off the power immediately. Also turn off the nitrogen gas and compressed air supplies.

Before Shipping the Dionex ASE 350

1. After extracting with a solvent that contains acids, bases, or other strong additives, rinse the system with either 100% organic solvent or distilled water (see [Section 3.1.9](#)) before turning off the power.
2. Empty all solvent reservoirs, reconnect the reservoirs to the system, and run one or more rinse cycles to ensure that all solvent is removed from the lines.
3. Remove the solvent reservoirs.

NOTE If there is no input to the Dionex ASE 350 for four hours (that is, no control panel buttons are pressed) and no method or sequence is running, the oven is automatically shut off.

4 • Troubleshooting

This chapter is a guide to troubleshooting minor issues that may arise during operation of the Dionex ASE 350:

- [Section 4.1](#) describes error messages and how to troubleshoot them.
- The remaining sections describe operating problems and how to resolve them.

If you are unable to resolve a problem by following the instructions here, contact Technical Support for Dionex products:

- In the U.S. and Canada, call 1-800-532-4752 and select **option 2**.
- Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

When talking with Technical Support personnel, please have this chapter of the manual at hand, as well as the following information:

- The Dionex ASE 350 method parameters
- The sample matrix

4.1 Error Messages

The Dionex ASE 350 firmware periodically checks the status of certain parameters. When a problem is detected, an error message is displayed on the Dionex ASE 350 screen. The message remains until you clear it (by pressing any button) or until another error message appears.

When the Dionex ASE 350 is in Remote mode, the firmware reports errors to Chromeleon software and error messages are logged in the audit trail.

There are two ways to review the errors that occurred during a given run:

- On the Dionex ASE 350 front panel, go to the **ERROR LOG** screen (see [Section C.5](#)).
- In Chromeleon software, click the **Audit** tab on the Dionex ASE 350 ePanel Set (see [Figure 3-18](#)).

Each error message is identified by a number. This section lists the error messages and explains how to respond if an error occurs. Most problems can be resolved by the user.

Error 1 Abort: Nitrogen compression pressure low. The run will abort.

Cause: Insufficient nitrogen gas pressure applied to the oven compression system during a run.

Action: Check the **PRESSURE REGULATORS** screen (see [Section C.3](#)). When the oven is compressed, the **COMPRESSION** field should read $0.90 \text{ MPa} \pm 0.03 \text{ MPa}$ ($130 \pm 5 \text{ psi}$). If the **COMPRESSION** reading is low, check the nitrogen gas pressure supplied to the Dionex ASE 350. It should be 0.97 to 1.38 MPa (140 to 200 psi); 1.03 MPa (150 psi) is recommended. If necessary, replace the gas cylinder.

Error 2 Abort: System air pressure low.

Cause: Insufficient air pressure applied to the system.

Action: Check the **PRESSURE REGULATORS** screen (see [Section C.3](#)). The **SYSTEM AIR** field should read $0.34 \pm 0.02 \text{ MPa}$ ($50 \pm 3 \text{ psi}$). If the **SYSTEM AIR** reading is low, check the air pressure supplied to the Dionex ASE 350. The recommended pressure is 0.69 MPa (100 psi). The allowed range when an inlet filter is *not* installed is 0.41 to 1.38 MPa (60 to 200 psi). When an inlet filter is installed, do not exceed 0.86 MPa (125 psi). If necessary, replace the air cylinder or make sure your air compressor is in working order.

Error 3 Abort: Cell pressure high.

Cause: A blockage in the system; the cell or lines may be plugged.

Action: Run a rinse cycle (see [Section 3.1.9](#)) to ensure that the pump, valves, and solvent lines are clear. If the rinse cycle runs with no errors, the cell may be plugged. Replace the cell filter (see [Section 3.1.5](#)) and clean (or replace) the cell frit (see [Section 5.2](#)). Then, mix the sample with a dispersing agent (see [Section 3.1.3](#)) and restart the run.

Action: If an error occurs during the rinse cycle, there may be a blockage in the rinse tube, solvent lines, source needle, or static valve. To determine the source of the high pressure, isolate segments of the flow path. One at a time, reconnect each component of the flow path. If reconnecting a component causes an increase in pressure, replace the component. It is not necessary to replace the static valve; it can be rebuilt (see [Section 5.7](#)).

Error 5 Abort: Rinse volume time-out.

Cause: The pump was unable to deliver the specified rinse volume within the allotted time; the cell or lines may be plugged.

Action: Run a rinse cycle (see [Section 3.1.9](#)) to ensure that the pump, valves, and solvent lines are clear. If the rinse cycle runs with no errors, the cell may be plugged. Replace the cell filter (see [Section 3.1.5](#)) and clean (or replace) the cell frit (see [Section 5.2](#)). Then, mix the sample with a dispersing agent (see [Section 3.1.3](#)) and restart the run.

Action: If an error occurs during the rinse cycle, there may be a blockage in the rinse tube, solvent lines, source needle, or static valve. To determine the source of the high pressure, isolate segments of the flow path. One at a time, reconnect each component of the flow path. If reconnecting a component causes an increase in pressure, replace the component. It is not necessary to replace the static valve; it can be rebuilt (see [Section 5.7](#)).

Error 6 Abort: Collection vessel loading time-out.

Cause: A collection vessel was not loaded within the allotted time; the collection tray may be blocked.

Action: Find and remove any obstructions; for example, check that all collection vessels are completely seated in the collection tray. Then, press the **Trays** button to return the collection tray to the home position.

Action: If the needle assembly hits the top of the collection vessel when it is lowered, the collection tray may need to be aligned. Contact Technical Support for Dionex products for assistance.

Error 7 **Abort: Cell loading time-out.**

Cause: A sample cell was not loaded within the allotted time; the AutoSeal arm or cell tray may be blocked.

Action: Find and remove any obstructions. Then, press the **Trays** button to return the collection tray to the home position.

Action: If the AutoSeal arm did not pick up the cell correctly, the cell tray may need to be aligned. Contact Technical Support for Dionex products for assistance.

Error 8 **Error: Rinse bottle full.**

Cause: One (or both) rinse bottles is full.

NOTE: When the Dionex ASE 350 is in Remote mode, this error message appears only when both rinse bottles are full.

Action: Empty the rinse bottles. Press **Enter** to continue the run.
Tip: In future, check the rinse bottles after each series of runs. If necessary, empty the bottles before starting the next series.

Error 9 **Error: Rinse bottle missing error.**

Cause: No rinse bottle is installed in the collection tray, or a rinse bottle is present but the sensor failed to detect it.

NOTE: When the Dionex ASE 350 is in Remote mode, this error message appears only when both rinse bottles are missing.

Action: Check that rinse bottles are installed in both rinse positions (labeled R1 and R2).

Action: Check that caps are installed on both rinse bottles.

Action: Find and remove any obstructions between the rinse bottle and the needle.

Error 10 Abort: Plate high temperature error.

Cause: The temperature of the top plate heater exceeded the maximum allowable temperature.

Action: The heater control needs to be calibrated. Contact Technical Support for Dionex products for assistance.

Error 11 Error: Plate low temperature error.

Cause: The temperature of the top plate heater is less than zero.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: The heater control needs to be calibrated. Contact Technical Support for Dionex products for assistance.

Error 12 Abort: Oven high temperature error.

Cause: The oven temperature exceeded the maximum allowable temperature.

Action: The heater control needs to be calibrated. Contact Technical Support for Dionex products for assistance.

Error 13 Error: Oven low temperature error.

Cause: The oven heater temperature is less than zero.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: The heater control needs to be calibrated. Contact Technical Support for Dionex products for assistance.

Error 14 Abort: Temperature RTD error.

Cause: The temperature reading from the RTD (Resistive Temperature Device) is inconsistent with normal operation.

Action: The RTD should be replaced. Contact Technical Support for Dionex products for assistance.

Error 15 Error: Liquid leak alarm.

Cause: The leak sensor detected liquid in the bottom of the drip tray.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode and the **LEAK ABORT** option on the **SYSTEM SETUP** screen (see [Figure 3-14](#)) is set to **YES**, Chromeleon software will abort the running sequence. When **NO** (the default setting) is selected, Chromeleon software will display an error message in the audit trail but will not interrupt the current run.

Action: Open the door to the service area (see [Section 2.1.8](#)). Clean and dry the drip tray thoroughly. Then, inspect the following for leaks (see [Figure 4-3](#)): pump check valves, pump fittings, pump seal in between the head and pump cylinder, solvent line fittings, transducer fittings, and pressure relief valve fittings. Also check for solvent leaks from the drain tubes connected to the solvent reservoirs and static valve. Tighten or replace any leaking fittings (see [Section 5.1](#)).

Error 16 Error: Hydrocarbon vapor alarm.

Cause: The hydrocarbon (vapor) level exceeds the threshold; this may indicate a leak from the cell, liquid components, or fittings.

Action: Check for foreign material on the threads of the cell end cap, the seal surface, and the cell body. If necessary, replace the seals (see [Section 5.2](#)) and/or O-rings (see [Section 5.3](#)). Tighten the cell end caps hand-tight. **Do not use a wrench or other tool.**

- Action:** Open the door to the service area (see [Section 2.1.8](#)). Clean and dry the drip tray thoroughly. Then, inspect the following for leaks (see [Figure 4-3](#)): pump check valves, pump fittings, pump seal in between the head and pump cylinder, solvent line fittings, transducer fittings, and pressure relief valve fittings. Also check for solvent leaks from the drain tubes connected to the solvent reservoirs and static valve. Tighten or replace any leaking fittings (see [Section 5.1](#)).
- Action:** Go to the **HYDROCARBON STATUS** screen (see [Section C.7](#)) and reset the **THRESHOLD**.
- Action:** If the hydrocarbon sensor is tripped repeatedly, you may want to disable the sensor. Contact Technical Support for Dionex products for assistance.
NOTE: If the sensor is disabled, be sure to monitor the system for leaks that may cause an excessive hydrocarbon vapor level.
- Action:** If the hydrocarbon sensor is tripped repeatedly, you may have to replace the sensor (see [Section 5.9](#)).

Error 17 Error: Cell size error.

- Cause:** The AutoSeal arm or sensors have malfunctioned.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, a message is displayed in the Chromeleon software audit trail, but the current run is not interrupted.
- Action:** Contact Technical Support for Dionex products for assistance.

Error 18 Error: AutoSeal close command was not successful.

- Cause:** The air supply is not reaching the AutoSeal arm.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.
- Action:** Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.
- Action:** Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

- Action:** Check the **AUTOSEAL** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); when the AutoSeal is closed, it should be 172.37 ± 14 kPa (25 ± 2 psi).
- Action:** Find and remove any obstructions that may be preventing AutoSeal arm movement.
- Action:** If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 19 Abort: AutoSeal open command was not successful.

- Cause:** The air supply is not reaching the AutoSeal arm.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.
- Action:** Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.
- Action:** Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).
- Action:** Check the **AUTOSEAL** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); when the AutoSeal is closed, it should be 172.37 ± 14 kPa (25 ± 2 psi).
- Action:** Find and remove any obstructions that may be preventing AutoSeal arm movement.
- Action:** If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 20 Error: AutoSeal go to tray command was not successful.

- Cause:** The air supply is not reaching the AutoSeal arm.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.
- Action:** Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.

Action: Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

Action: Find and remove any obstructions that may be preventing AutoSeal arm movement.

Action: If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 21 Error: AutoSeal go to oven command was not successful.

Cause: The air supply is not reaching the AutoSeal arm.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.

Action: Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

Action: Find and remove any obstructions that may be preventing AutoSeal arm movement.

Action: If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 22 Error: AutoSeal command rejected due to unsafe condition.

Cause: The cell tray sensor was unable to detect the tray at a cell position (not aligned with a cell).
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: The cell tray may not be in the correct position. Abort the run (see [Section 3.5.3](#)), and then restart the run.

Action: The cell tray sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Cause: The AutoSeal arm is hitting the cell tray.

Action: The AutoSeal arm or cell tray is out of alignment. Contact Technical Support for Dionex products for assistance.

Error 23 Error: Needle up command was not successful.

Cause: Insufficient air pressure supplied to the system.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended. Check the system air regulator pressure (see [Section B.7](#)). Check the delivery of air to the needle assembly.

Cause: Movement of the needle assembly is blocked.

Action: Find and remove any obstructions.

Cause: The needle sensor was unable to detect the needle in the up position.

Action: The sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Error 24 Error: Needle down command was not successful.

Cause: Insufficient air pressure supplied to the system.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.

Action: Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

Action: The collection tray may not be in the correct position. Abort the run (see [Section 3.5.3](#)), and then restart the run.

Cause: Movement of the needle assembly is blocked.

Action: Find and remove any obstructions.

Cause: The needle sensor was unable to detect the needle in the down position.

Action: The sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Cause: The collection tray is out of alignment.

Action: Contact Technical Support for Dionex products for assistance.

Error 25 Error: Needle in command was not successful.

Cause: Insufficient air pressure supplied to the system.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.

Action: Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

Action: The collection tray may not be in the correct position. Abort the run (see [Section 3.5.3](#)), and then restart the run.

Cause: Movement of the needle assembly is blocked.

Action: Find and remove any obstructions.

Cause: The needle sensor was unable to detect the needle in the in position.

Action: The sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Error 26 Error: Needle out command was not successful.

Cause: Insufficient air pressure supplied to the system.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check the air supply pressure. The air source should be between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.

Action: Check the **SYSTEM AIR** reading on the **PRESSURE REGULATORS** screen (see [Section C.3](#)); it should be 0.34 ± 0.02 MPa (50 ± 3 psi).

Action: The collection tray may not be in the correct position. Abort the run (see [Section 3.5.3](#)), and then restart the run.

Cause: Movement of the needle assembly is blocked.

Action: Find and remove any obstructions.

Cause: The needle sensor was unable to detect the needle in the out position.

Action: The sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Error 27 Error: Needle command rejected due to unsafe condition.

Cause: The collection tray sensor was unable to detect the tray at a position that contains a collection vessel.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: The collection tray may not be in the correct position. Abort the run (see [Section 3.5.3](#)), and then restart the run.

Action: The sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Cause: The needle assembly is hitting the collection tray.

Action: The collection tray is out of alignment. Contact Technical Support for Dionex products for assistance.

Error 28 Abort: Cell tray cannot find the requested position.

Cause: The cell tray is either physically blocked or is out of position.

Action: Find and remove any obstructions. Then, manually check the tray movement, using the **Trays** button. If the tray does not move freely, contact Technical Support for Dionex products for assistance.

Error 29 Abort: Cell tray command rejected due to unsafe condition.

Cause: The AutoSeal arm is either in motion or is in an invalid position.

Action: Wait until the AutoSeal arm stops moving.

Action: If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 31 Abort: Collection tray cannot find the requested position.

Cause: The collection tray is either physically blocked or is out of position.

Action: Find and remove any obstructions. Then, manually check the tray movement, using the **Trays** button. If the tray does not move freely, contact Technical Support for Dionex products for assistance.

Error 32 Abort: Collection tray command rejected due to unsafe condition.

Cause: The needle assembly is either in motion or is in an invalid position.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Wait until the needle assembly stops moving.

Action: If the error message appears again, contact Technical Support for Dionex products for assistance.

Error 35 Error: You forgot to save your method after editing it.

Cause: You tried to load a new method or exit the **METHOD EDITOR** screen before saving changes to the method currently being edited.

Action: If you do not want to save the editing changes, press **Enter**. This retains the method in its previously saved version. If you want to continue editing the method, press any key. When you finish editing, you have two options:

- To save the changes to the current method number, move the cursor to the **SAVE TO** field and press **Enter**.
- To save the changes to a different method number, move the cursor to the **SAVE TO** field and either enter a new (unused) method number or press **Select** Δ to display the next unused method number. Press **Enter** to save the method.

Error 36 Error: You forgot to save your sequence after editing it.

Cause: You tried to load a new sequence or exit the **SEQUENCE EDITOR** screen before saving changes to the sequence currently being edited.

Action: If you do not want to save the editing changes, press **Enter**. This retains the sequence in its previously saved version. If you want to continue editing the sequence, press any key. When you finish editing, you have two options:

- To save the changes to the current sequence number, move the cursor to the **SAVE TO** field and press **Enter**.
- To save the changes to a different sequence number, either move the cursor to the **SAVE TO** field and enter a new (unused) sequence number or press **Select** Δ to display the next unused sequence number. Press **Enter** to save the sequence.

Error 37 Error: Operator input value is not in range. Check prompt line at the bottom of the screen for the range of values allowed.

Cause: While editing a method or sequence, you entered an invalid parameter.

Action: Enter an acceptable value.

Error 38 **Error: Usage Log screen, press Enter button to continue resetting parameter. Any other keypad entry will cancel process.**

Cause: This error message requests confirmation that you want to reset a counter on the **USAGE LOG** screen (see [Section C.4](#)).

Action: To reset the counter, press **Enter**. To cancel the change, press any other keypad button.

Error 39 **Error: Method was not saved; there are one or more invalid fields. Enter zero in edit # field for a default method and re-enter desired conditions.**

Cause: A method contains parameters that are in conflict with each other. For example, if you change the cell type from stainless steel to zirconium, a cell size that was previously valid may now be out of range.

Action: Enter zero in the **METHOD EDIT #** field to display the default method parameters. Make any changes required, and then save the method to a new method number (see [Section 3.2.1](#)). [Table 3-3](#) lists the values allowed for each parameter.

Error 40 **Error: Sequence was not saved; there are one or more invalid fields. Methods in the sequence were also checked for valid fields.**

Cause: A method in the sequence was not saved.

Action: Check that all methods have been saved.

Cause: A method contains an invalid parameter.

Action: Check for method parameters that are in conflict with each other. For example, if you change the cell type from stainless steel to zirconium, a cell size that was previously valid may now be out of range. [Table 3-3](#) lists the values allowed for each parameter.

Error 43 Abort: The collection tray type (60-mL vials or 250-mL bottles) does not match the sequence entry.

Cause: The collection tray type specified in the sequence does not correspond to the tray type currently installed.

Action: If the collection vessels are 250-mL bottles:

- Go to the **SEQUENCE EDITOR** screen (see [Section 3.2.3](#)) and check that **NO INSERT** is displayed.
- If a vial tray insert (P/N 066176) is installed in the collection tray, remove it.

Action: If the collection vessels are 60-mL vials:

- Go to the **SEQUENCE EDITOR** screen (see [Section 3.2.3](#)) and check that **INSERT** is displayed.
- If a vial tray insert P/N 066176) is not installed in the collection tray, install it now (see [page 68](#)).

Error 44 Error: Rinse tube is missing.

Cause: One (or both) of the rinse tubes is missing from the cell tray, or a rinse tube is present but the sensor failed to detect it.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, a message is displayed in the Chromeleon software audit trail, but the current run is not interrupted.

Action: Check that rinse tubes are installed between positions 6 and 7 and positions 18 and 19 of the cell tray.

Error 45 Error: There are no rinse tubes.

Cause: The cell tray does not contain any rinse tubes, or a rinse tube is present but the sensor failed to detect it.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Check that rinse tubes are installed between positions 6 and 7 and positions 18 and 19 of the cell tray.

Action: If the error message appears again, the sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Action: The cell tray needs to be aligned. Contact Technical Support for Dionex products for assistance.

Error 46 **Error: The rinse bottles are either missing or full. Please place an empty rinse bottle in R1 or R2. Then press the Enter button to continue the rinse process.**

Cause: One (or both) rinse bottles is missing.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Place a collection bottle in positions R1 and R2 in the collection tray. Press **Enter** to continue the run.

To avoid having this error message reappear: Check the rinse bottles after each series of runs; if necessary, empty the bottles before starting the next series.

Cause: One (or both) rinse bottles is full.

Action: Empty the rinse bottles. Press **Enter** to continue the run.

Tip: In future, check the rinse bottles after each series of runs; if necessary, empty the bottles before starting the next series.

Error 47 **Error: The cell size is too large for the collection vessel capacity. Use 250-mL bottles for this cell size.**

Cause: Although the cell tray contains 66-mL and/or 100-mL sample cells, 60-mL vials are installed as collection vessels.

Action: When using 66-mL or 100-mL sample cells, always install 250-mL collection bottles as the collection vessels.

Error 48 **Abort: Pump error detected. Check pump seals, check valves, and check cell seals.**

Cause: The expected pressure was not reached during the fill step.

Action: Replace the pump check valve cartridges (see [Section 5.4](#)).

Action: Check for foreign material on the threads of the cell end cap, the seal surface, and the cell body. If necessary, replace the seals (see [Section 5.2](#)) and/or O-rings (see [Section 5.3](#)). Tighten the cell end caps hand-tight. **Do not use a wrench or other tool.**

Action: Open the door to the service area (see [Section 2.1.8](#)). Inspect the following for leaks (see [Figure 4-3](#)): pump check valves, pump fittings, pump seal in between the head and pump cylinder, solvent line fittings, transducer fittings, and pressure relief valve fittings. Tighten or replace any leaking fittings (see [Section 5.1](#)).

Error 49 **Abort: For this run, the method parameter cell type is zirconium. Only 66- and 100-mL cell sizes are allowed for zirconium. Sensors detected a smaller cell.**

Cause: Only 66-mL and 100-mL cells are available in zirconium. The installed cell is smaller than this, indicating that the cell material is not zirconium.

Action: Either use the cell specified in the method or edit the method to match the cell type currently installed.

Error 50 **Caution: The LOAD METHOD/SEQUENCE screen is not accessible during a run or rinse.**

Cause: You tried to display the **LOAD METHOD/SEQUENCE** screen while a run or rinse was in progress.

Action: Wait until the task underway has finished before selecting this screen.

Error 51 **Error: The safety shield is not in a safe position. Please close the safety shield and restart the run.**

Cause: You tried to start a run while the safety shield was open.
NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: Close the safety shield and restart the run.

Error 54 **Error: For solvent saver mode, the cell size detected should match the cell size in the method.**

Cause: The cell size installed in the tray does not match the cell size specified in the method.

Action: If the correct cells are installed, edit the method to specify this cell size. If the wrong cells are installed, replace them.

Error 60 **Error: Changing this setting will cause sequence lines 20–33 to be deleted. Do you want to continue? Press Enter to continue. Press any other key to go back.**

Cause: You changed the setting from **INSERT** to **NO INSERT** on the **SEQUENCE EDITOR** screen (see [Section 3.2.3](#)). Because collection vessels cannot be loaded in positions 20–33 of the collection tray unless a vial tray insert (P/N 066176) is installed, these sequence lines are now invalid.

NOTE: If this error occurs when the Dionex ASE 350 is in Remote mode, Chromeleon software will abort the running sequence.

Action: If you want to delete lines 20–33 from the sequence, press **Enter**.

Action: If you do not want to delete lines 20–33 from the sequence, press any key other than **Enter**.

Error 63 **Warning: Invalid entry while in remote control.**

Cause: With the Dionex ASE 350 under Chromeleon software control, you either tried to edit a field on the front panel display or pressed a front panel button other than **Trays**, **Stop**, **Contrast** Δ , or **Contrast** ∇ (the only buttons that are functional in Remote mode).

Action: Before trying to make any changes from the Dionex ASE 350 front panel, wait until Chromeleon software has finished the function in process and then disconnect from Chromeleon software control.

Error 64 Error: Collection vessel full.

- Cause:** The collection vessel is full.
- Action:** Empty the collection vessel, if needed. If there is a label or written information on the collection vessel, make sure it is not blocking the area read by the sensor (see [Figure 3-11](#)).
- Action:** Check the cell size and rinse volume specified in the method; if it exceeds the volume of the collection vessel, reduce the rinse volume.
- Action:** If liquid drips continually into the collection vessel during the heating and static steps, it indicates a leak from the static valve. Rebuild the static valve (see [Section 5.7](#)).
- Action:** Viscous samples and plant samples may repeatedly trip the sensor. If you routinely extract these samples, you may want to disable the sensor. Contact Technical Support for Dionex products for assistance.
NOTE: If the sensor is disabled, be sure to monitor collection vessels to check for overfilling.

Error 65 Error: Collection vessel missing error.

- Cause:** No collection vessel is installed in the collection tray, or a collection vessel is present but the sensor failed to detect it.
- Action:** Check that a collection vessel is installed in the position specified in the method.
- Action:** Find and remove any obstructions between the collection vessel and the needle.

Error 66 Error: Cell is missing.

- Cause:** No cell is installed, or a cell is present but the sensor failed to detect it.
- Action:** Check that a cell is installed in the position specified in the sequence.
- Action:** If the error message appears again, the sensor has malfunctioned. Contact Technical Support for Dionex products for assistance.

Action: The cell tray needs to be aligned. Contact Technical Support for Dionex products for assistance.

Error 67 **Error: There was a problem in reducing the cell pressure to a safe level under 15 psi before returning the cell to the carousel. The cell pressure may need to be calibrated or check for obstruction.**

Cause: A blockage in the system; the cell or lines may be plugged.

Action: Run a rinse cycle (see [Section 3.1.9](#)) to ensure that the pump, valves, and solvent lines are clear. If the rinse cycle runs with no errors, the cell may be plugged. Replace the cell filter (see [Section 3.1.5](#)) and clean (or replace) the cell frit (see [Section 5.2](#)). Then, mix the sample with a dispersing agent (see [Section 3.1.3](#)) and restart the run.

Action: If an error occurs during the rinse cycle, there may be a blockage in the rinse tube, solvent lines, source needle, or static valve. To determine the source of the high pressure, isolate segments of the flow path. One at a time, reconnect each component of the flow path. If reconnecting a component causes an increase in pressure, replace the component. It is not necessary to replace the static valve; it can be rebuilt (see [Section 5.7](#)).

Cause: If the error message appears again, the cell pressure needs to be calibrated.

Action: The cell pressure cannot be calibrated by the user. Contact Technical Support for Dionex products for assistance.

4.2 Liquid Leaks

- **Missing or worn-out cell or rinse tube O-rings**

Check the ends of the cell or rinse tube to verify that the O-rings are in place and in good condition (see [Figure 4-1](#)). If an O-ring has a hole size of less than 0.5 mm, replace it. If a white PTFE O-ring is discolored, replace it.

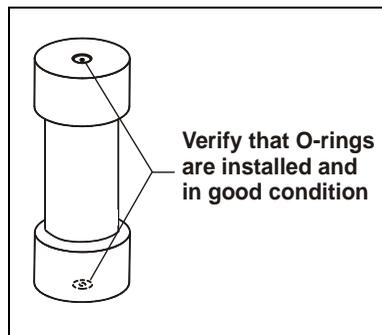


Figure 4-1. Inspecting O-Rings

Remove O-rings with a small flathead screwdriver (P/N 046985). Using the tool (P/N 049660) provided in the Dionex ASE 350 Ship Kit (P/N 066060), place a new O-ring (PTFE: P/N 049457, pkg. of 50; Viton: P/N 056325, pkg. of 50) over the opening in the end of the cell end cap and press it into place (see [Figure 4-2](#)).

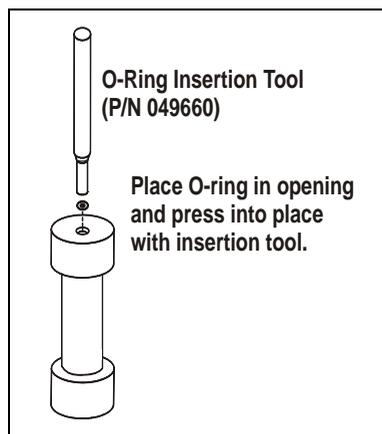


Figure 4-2. Installing a Cell End Cap O-Ring

- **Worn-out PEEK seal in cell end cap**

Replace the seal (see [Section 5.2](#)).

- **Leak in solvent flow path**

Open the door to the service area (see [Figure 2-1](#)). (Rotate the three quarter-turn screws along the edge of the door and swing the door open.) Inspect the following for leaks (see [Figure 4-3](#)): pump check valves, pump fittings, solvent line fittings, transducer fittings, and pressure relief valve fittings. Tighten or replace any leaking fittings (see [Section 5.1](#)).

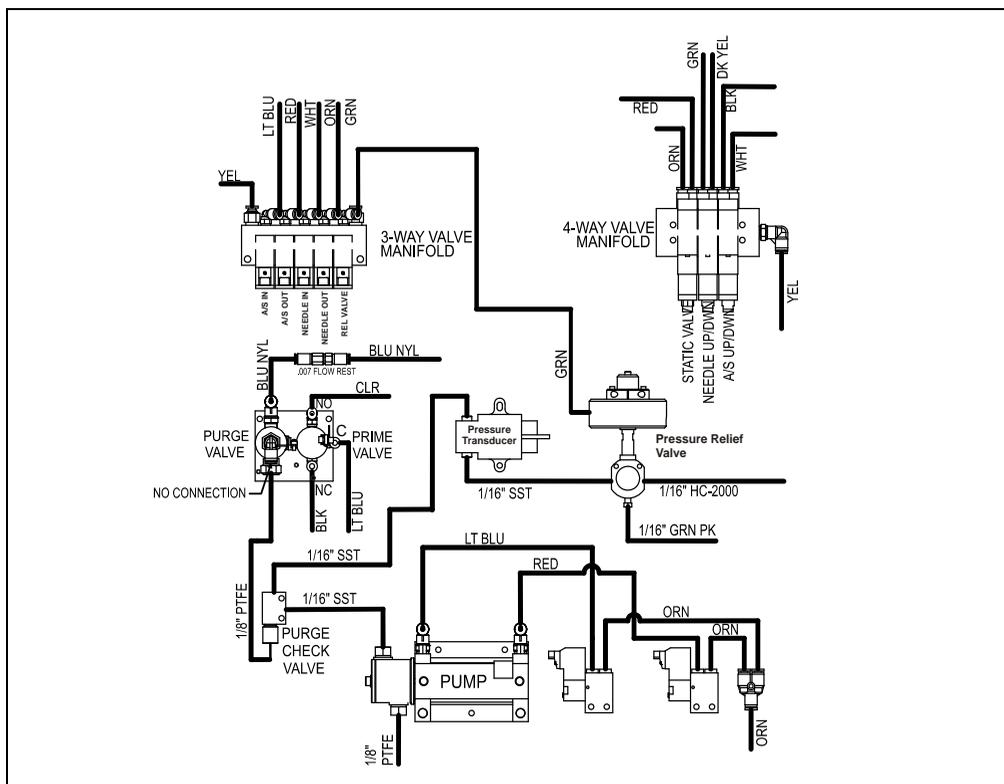


Figure 4-3. Dionex ASE 350 Solvent and Gas Flow Schematic

If the pump head is leaking, replace the piston seals (see [Section 5.5](#)).

If the drip tray contains liquid, it indicates leakage from the static valve O-ring. Use a 5/8-inch open-end wrench to tighten the 5/8-inch nut on the valve body. If this does not stop the leak, rebuild the static valve (see [Section 5.7](#)).

- **Leaking into waste bottle during static cycle**

If liquid drips from the green tubing into the waste bottle when the cell is under pressure and the static valve is closed, the pressure relief valve is worn and must be replaced (see [Section 5.6](#)).

- **Leaking into collection vessel during static cycle**

Check the tip of the source needle (see [Figure 5-25](#)) in the collection vessel. If liquid drips from the source needle into the collection vessel during the static cycle (when the static valve should be closed), the static valve seals are worn and the valve must be rebuilt (see [Section 5.7](#)).

4.3 Gas and Air Leaks

Gas and air leaks are usually audible. In addition, excessive gas or air consumption often indicates a leak. If you suspect a leak, check the following locations: the rear panel, the solvent reservoirs, and the connections inside the service area (see [Figure 2-1](#)). Listen for leaks, and run your hand over connections to feel for escaping gas.

If you find a loose connection, push the tubing firmly onto its fitting. If the fitting or tubing continues to leak, replace it (see [Section 5.1](#)).

4.4 Stopped System

- **Operating error occurred**

Check the display screen or Chromeleon software audit trail for error messages. If a message is displayed, note the error number and refer to [Section 4.1](#) for troubleshooting guidance.

- **Method or sequence complete**

Check the display screen or Chromeleon software audit trail for error messages. If no messages are displayed, the Dionex ASE 350 probably stopped because the method or sequence was finished.

When running under *method* control, the Dionex ASE 350 runs samples sequentially until it reaches an empty cell or collection vessel position or, if the trays are full, until all samples have been run and the trays have returned to positions 1.

When running under *sequence* control, the Dionex ASE 350 runs according to each line defined in the sequence. A sequence is complete when no more lines are defined *or* when one of these fields contains a zero: **BTL/VIAL #**, **CELL #**, or **METHOD #**.

- **Electrical cables improperly installed**

Open the door to the service area (see [Figure 2-1](#)). (Rotate the three quarter-turn screws along the edge of the door and swing the door open.) Check that all electrical cables are seated properly in the connectors on the printed circuit boards.

This chapter describes Dionex ASE 350 service and repair procedures that users can perform. All procedures not included here, including electronics-related repair procedures, must be performed by qualified Thermo Fisher Scientific personnel. For assistance, contact Technical Support for Dionex products:

- In the U.S. and Canada, call 1-800-532-4752 and select **option 2**.
- Outside the U.S. and Canada, call the nearest Thermo Fisher Scientific office.

Before replacing any parts, review the troubleshooting information in [Chapter 4](#) to isolate the cause of the problem.

IMPORTANT

Substituting non-Thermo Scientific/Dionex parts (including extraction cells, extraction cell components, and valves) may impair the performance of the Dionex ASE 350, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

5.1 Replacing Tubing and Fittings

Use a 1/4-inch open-end wrench (P/N 014605) to tighten any leaking fittings. **Do not overtighten!**

If tightening a fitting does not stop a leak, the tubing and fitting assembly must be replaced. Contact Technical Support for Dionex products for assistance.

5.2 Replacing the Cell End Cap Seal

A worn PEEK seal is deeply grooved and will fail to form a tight seal between the cell cap and body during a run, causing leaks.

1. Unscrew the cell end cap from the cell body (see [Figure 5-1](#)).



Wear safety glasses when removing the old snap ring from the cell end cap or installing a new snap ring.



Le port de lunettes de sécurité est requis lors du changement de l'anneau de serrage sur le bouchon des cellules d'extraction.



Tragen Sie eine Schutzbrille, wenn Sie den alten Sicherungsring von der Verschlusskappe der Zelle entfernen oder wenn Sie einen neuen Sicherungsring installieren.

IMPORTANT

Do not disassemble the cell end cap after each use. This can cause the seal to fail prematurely.

2. Remove the snap ring from the end cap, using the snap ring tool (P/N 056684) provided in the Dionex ASE 350 Ship Kit (P/N 066060). (The procedure for stainless steel and zirconium caps is identical. The photos here show a stainless steel cap.)
 - a. Insert the pointed ends of the tool into the two holes in the snap ring (see [Figure 5-1](#)).
 - b. Squeeze the handles of the tool together to reduce the diameter of the ring. At the same time, carefully pull the ring out of the cap.
 - c. Carefully release the handles of the tool and remove the ring from the tool.



Figure 5-1. Removing the Snap Ring

3. Remove the cap insert (see [Figure 5-2](#)).
Remove the PEEK seal from the groove in the bottom of the cap insert.
4. Remove the frit from the bottom of the end cap.
5. Clean the frit by sonicating it in solvent (or replace it).

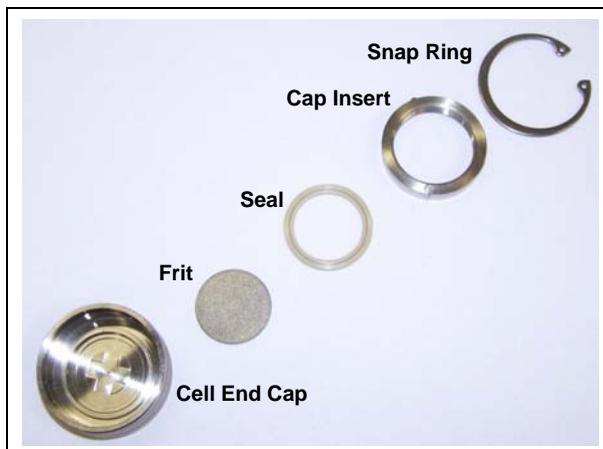


Figure 5-2. Cell End Cap Disassembled

6. Refer to [Figure 5-3](#) and the following steps to reassemble the sample cell.
 - a. Place the frit (cleaned or new) into the bottom of the end cap.
 - b. Press a new PEEK seal (P/N 061687, pkg. 50) into the bottom of the cap insert.
 - c. Align the pins in the cap insert with the grooves in the end cap, and then place the insert, with the seal facing down, into the end cap.
7. With the cap assembly upright on the workbench, install the snap ring.
 - a. Insert the snap ring tool into the holes on the ring.
 - b. Squeeze the tool handles to bring the ends of the ring together.
 - c. Insert the ring into the cap. Using your fingers, push the ring under the lip of the end cap. After verifying that the entire ring is under the lip, release the tension on the tool and remove the tool from the ring.
 - d. Screw the cap back onto the cell body and hand-tighten.

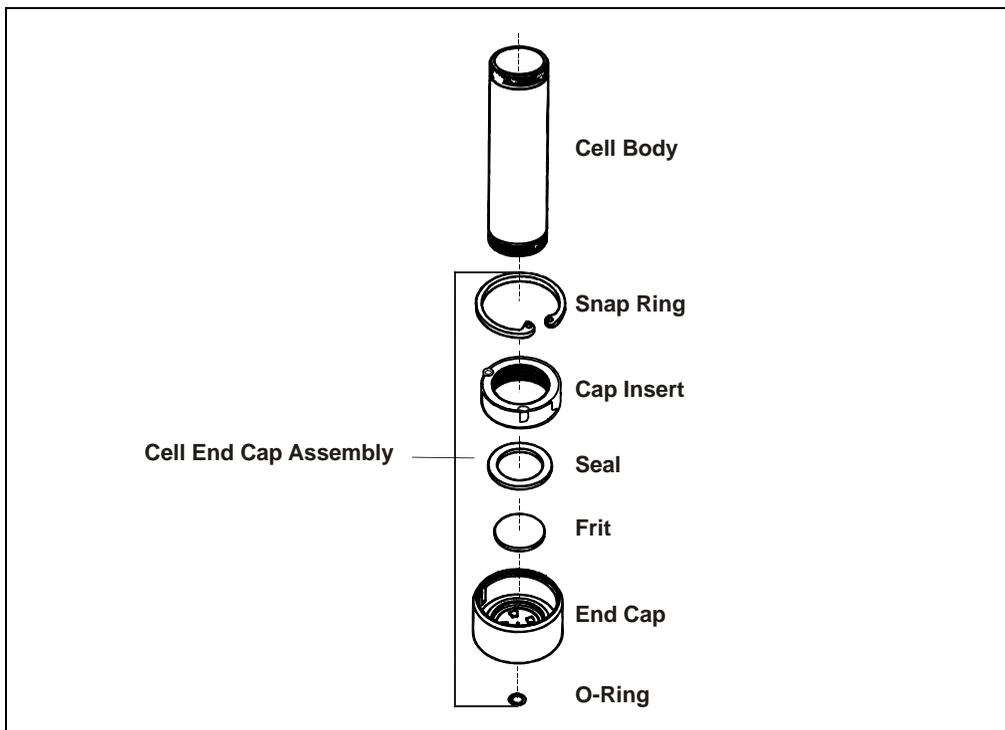


Figure 5-3. Sample Cell Assembly

5.3 Replacing the Cell End Cap O-Ring

IMPORTANT

Be careful not to scratch the interior of the cell end cap when installing or removing the cell O-ring. Scratches on the sealing surface will prevent the O-ring from sealing properly and may result in leaks during operation.

1. Locate the small flathead screwdriver (P/N 046985) and the O-ring insertion tool (P/N 049660) provided in the Dionex ASE 350 Ship Kit (P/N 066060).
2. Insert the tip of the screwdriver into the end cap and *carefully* pick out the O-ring. (This may be easier to do if you first remove the cap from the cell body and place it flat on the workbench.)
3. Place a new O-ring (PTFE: P/N 049457, pkg. of 50; Viton: P/N 056325, pkg. of 50) over the opening in the end of the cell end cap. Press the O-ring into place, using the O-ring insertion tool (see [Figure 5-4](#)).

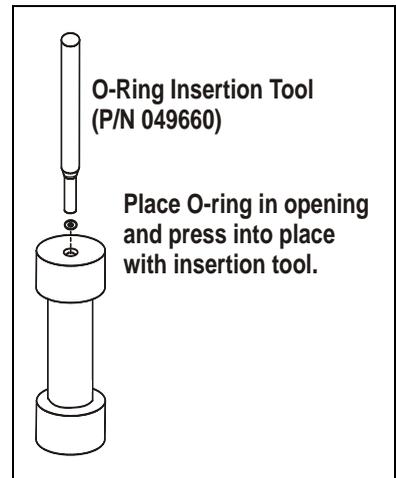


Figure 5-4. Installing a Cell End Cap O-Ring

5.4 Replacing the Pump Check Valve Cartridges

5.4.1 Before Beginning

Disconnect all solvent reservoirs and remove them from the solvent reservoir tray. Press **Rinse** to run a rinse cycle (see [Section 3.1.9](#)). This prevents siphoning of solvent when the inlet tubing is disconnected.

5.4.2 Removing the Pump

1. Turn off the gas and air supplies. Disconnect the gas and air sources at the Dionex ASE 350 rear panel.
2. Turn off the Dionex ASE 350 power switch.
3. To access the service area, rotate the three quarter-turn screws along the edge of the door and swing the door open.
4. The pump is installed near the bottom of the component mounting panel on the inside of the door (see [Figure 5-5](#), **1**).

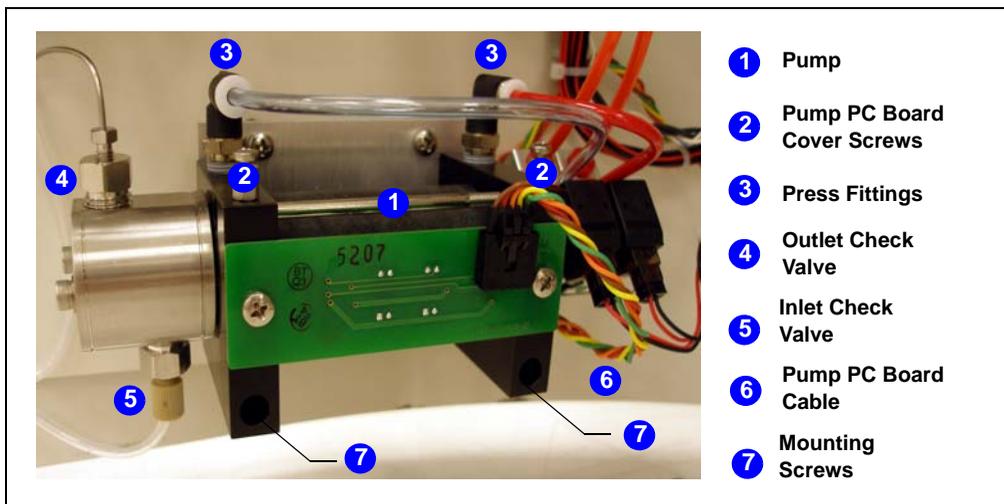


Figure 5-5. Dionex ASE 350 Pump (Shown with PC Board Cover Removed)

5. Use a Phillips screwdriver to loosen the two screws (**2**) that attach the pump PC board cover (black) to the pump. Remove the cover.

6. Disconnect the red tubing and the transparent blue tubing from the two black press fittings (3) on top of the pump.
- Tip:** To disconnect a press fitting, use your fingers (or a small open-end wrench) to press the ring on the fitting *in*, while at the same time pulling the tubing *out*.
7. Use a 1/4-inch open-end wrench (P/N 014605) to disconnect the stainless steel fitting from the outlet check valve (4).
 8. By hand, disconnect the PEEK fitting from the inlet check valve (5).
 9. Disconnect the multicolored cable from the pump PC board (6).
 10. Use a 3-mm hex-screw driver (P/N 060154) to remove the screws in the left and right end plates (7) on the pump. These screws secure the pump to the component panel.

5.4.3 Removing the Check Valves and Cartridges

1. Use a 1/2-inch open-end wrench to loosen the *inlet* check valve housing. Remove the housing, and then remove the check valve cartridge from the housing (see [Figure 5-6](#)).

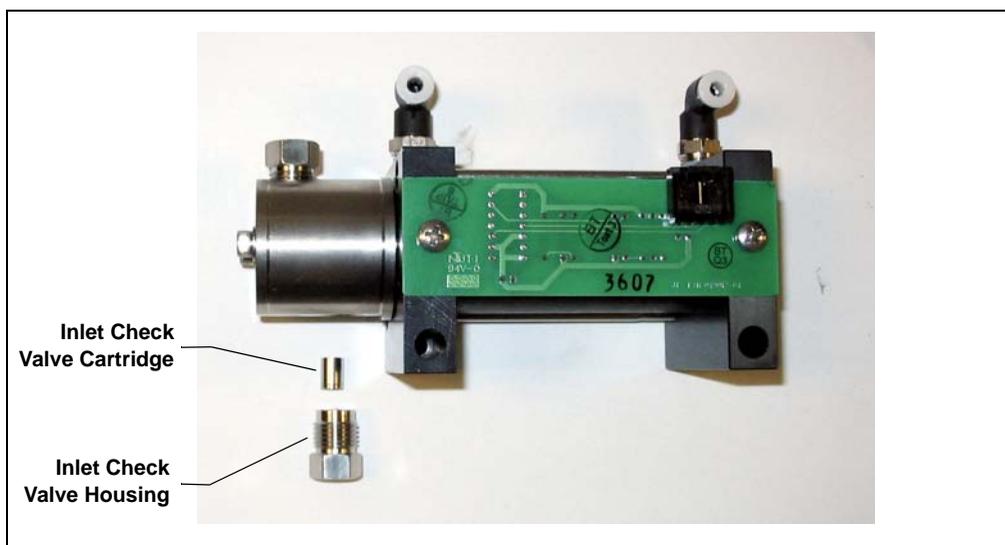


Figure 5-6. Disassembled Dionex ASE 350 Inlet Check Valve

2. Turn the pump over, so that the outlet check valve is facing down.
3. Use a 1/2-inch open-end wrench to loosen the *outlet* check valve housing. Remove the housing, and then remove the check valve cartridge from the housing.

NOTE The housing for the *inlet* check valve assembly has a 1/4-28 port. The housing for the *outlet* check valve assembly has a 10-32 port (see [Figure 5-7](#)).

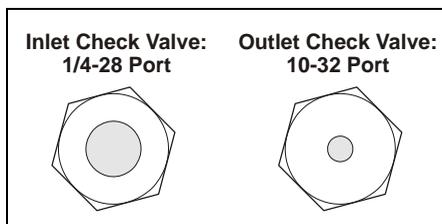


Figure 5-7. Check Valve Housings

5.4.4 Cleaning the Check Valve Housings

1. Place the check valve housings in a beaker with methanol. Sonicate or agitate for several minutes.
2. Rinse each check valve housing thoroughly with filtered, deionized water.

5.4.5 Installing New Check Valve Cartridges

1. Install the new inlet cartridge (P/N 047755) in the inlet check valve housing so that the double-hole end of the cartridge is visible (see [Figure 5-8](#)).
2. Install the new outlet cartridge (P/N 057346) in the outlet check valve housing so that the arrow on the cartridge points down.

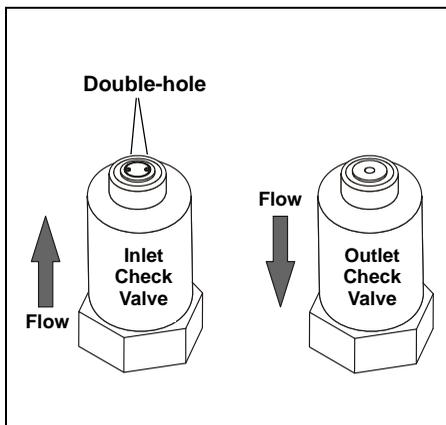


Figure 5-8. Check Valve Assemblies

5.4.6 Reinstalling the Check Valves

1. Install the inlet check valve assembly on the bottom of the pump head.
2. Install the outlet check valve assembly on the top of the pump head.
3. Tighten the check valves finger-tight, and then use a wrench to tighten an additional one-eighth to one-quarter turn.

IMPORTANT

Overtightening may damage the pump head and the check valve housing and crush the check valve seats.

5.4.7 Reinstalling the Pump

1. Reinstall the pump on the component panel.
2. Reconnect the two air lines to the press fittings on top of the pump.
3. Reconnect the solvent lines to the inlet and outlet check valve housings.
4. Reconnect the multicolored cable to the pump PC board.
5. Reinstall the pump PC board cover.
6. Close the door to the service area.

5.4.8 Completing the Procedure

1. Reinstall the solvent reservoirs.
2. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
3. Turn on the Dionex ASE 350 power switch.

5.5 Replacing the Pump Seals

5.5.1 Before Beginning

Disconnect all solvent reservoirs and remove them from the solvent reservoir tray. Press **Rinse** to run a rinse cycle (see [Section 3.1.9](#)). This prevents siphoning of solvent when the inlet tubing is disconnected.

5.5.2 Removing the Pump

Follow the instructions in [“Removing the Pump”](#) on page 152.

5.5.3 Replacing the Piston High-Pressure Seal

1. Use a 3-mm hex driver (P/N 060154) or wrench to loosen and remove the two screws securing the pump head to the body.
2. Slide the pump head straight off the pump (see [Figure 5-9](#)).

IMPORTANT

Lateral motion while disengaging the pump head from the pump may break the piston.

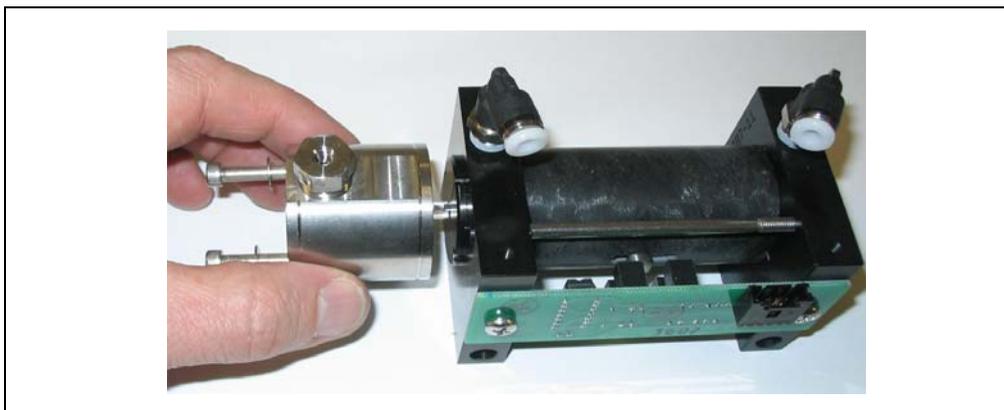


Figure 5-9. Removing the Pump Head

3. Turn the pump head over so that the open end is facing down. The tan piston guide (see [Figure 5-10](#)) should drop out of the head; if it does not, gently tap the head on the workbench to dislodge the guide. Save the piston guide (it will be reinstalled in [Step 7](#)).

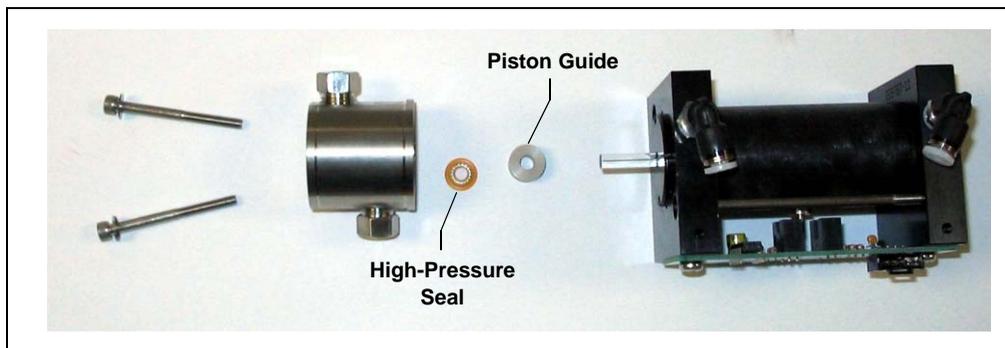


Figure 5-10. Replacing the Dionex ASE 350 Piston High-Pressure Seal

4. If tapping the head on the bench does not remove the piston guide, use the wooden end of a cotton-tipped swab to pry the guide out of the pump head. Discard the used piston guide.
5. To remove the orange high-pressure seal from the pump head, insert the wooden end of a cotton-tipped swab and pry out the seal. Discard the seal.

IMPORTANT

Do not use a sharp tool (such as tweezers) to remove or install the piston high-pressure seal. This will scratch the inside of the pump housing; these scratches will prevent a proper seal and cause leakage.

6. Hold the new high-pressure seal (P/N 066162), with the smaller diameter (ringed) side of the seal facing down, and drop it into the pump head cavity.
7. If the piston guide was easily removed from the pump head ([see Step 3](#)), place the original piston guide on top of the high-pressure seal and firmly press it into place. This also seats the high-pressure seal in the pump head.



If you had to pry the piston guide out of the pump head ([Step 4](#)), install a new piston guide (P/N 066109) as described above. The original piston guide may have been scratched during removal, so do not use it again. A scratched guide will not seal properly.

8. Reinstall the pump head onto the pump. Make sure the outlet check valve is oriented upward.

5.5.4 Replacing the Piston Air Seal

1. Remove the PC board from the side of the pump (see [Figure 5-11](#), view A).
2. Loosen the two bolts that hold the two pump end plates together (see [Figure 5-11](#), view B).

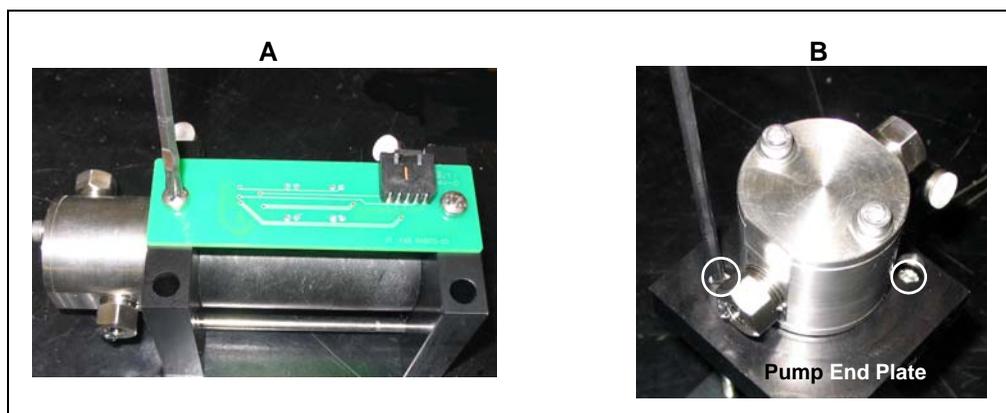


Figure 5-11. Removing the Pump PC Board and End Plate Bolts

3. Remove the right end plate from the pump cylinder (see [Figure 5-12](#)).

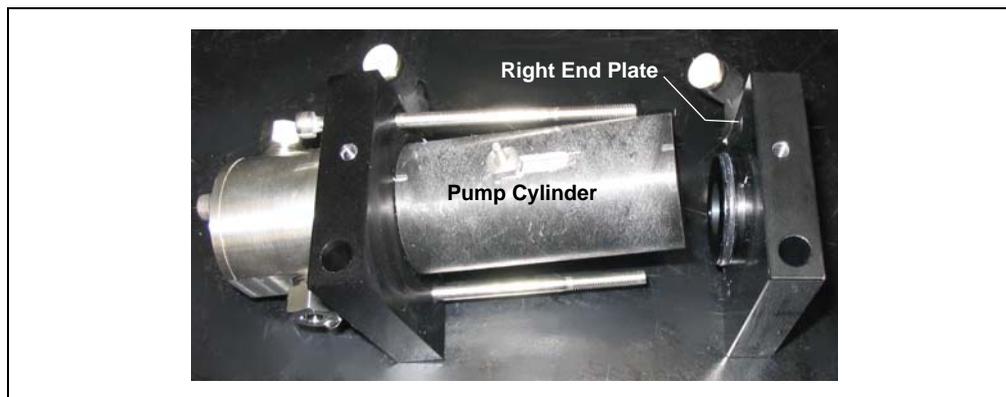


Figure 5-12. Removing the Pump Right End Plate

4. Remove the left end plate from the cylinder, exposing the piston (see [Figure 5-13](#)).

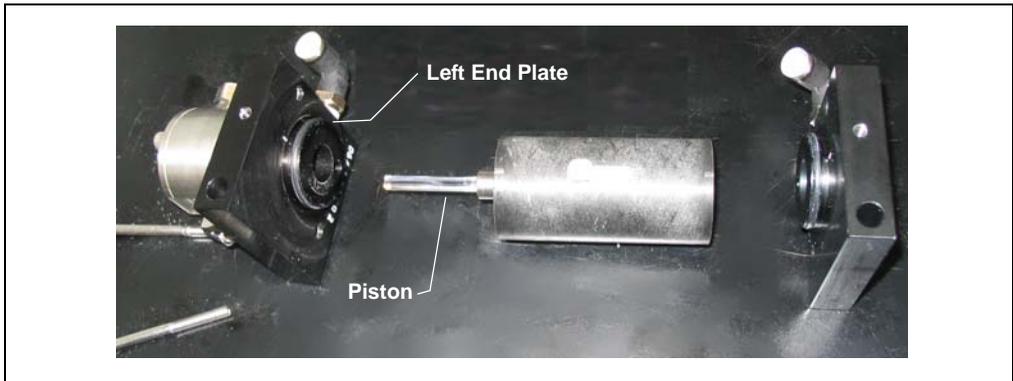


Figure 5-13. Removing the Pump Left End Plate

5. To remove the piston air seal from the left end plate (see [Figure 5-14](#)), insert the wooden end of a cotton-tipped swab and pry out the seal. Discard the seal.

IMPORTANT

Do not use a sharp tool (such as tweezers) to remove or install the piston air seal. This will scratch the seal and the inside of the pump housing; these scratches will prevent a proper seal and cause leakage.

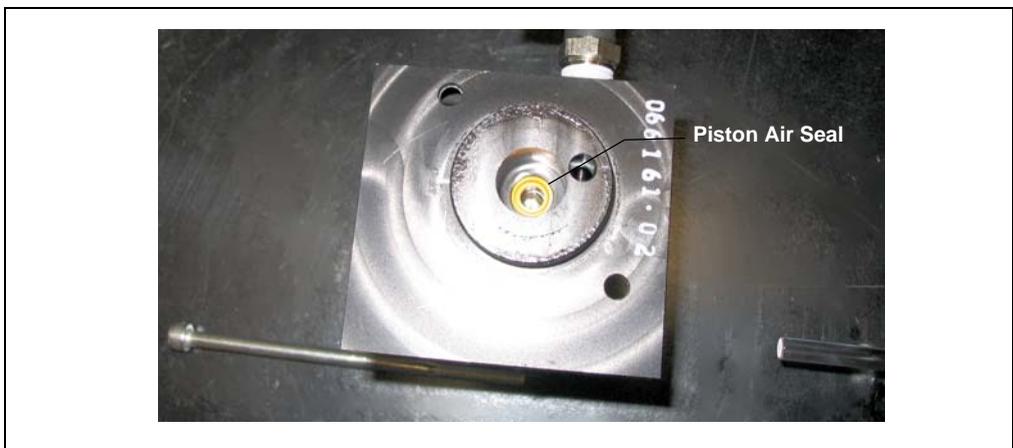


Figure 5-14. Removing the Piston Air Seal

6. Insert the new piston air seal (P/N 066163), with the closed end facing down, into the end plate. Gently press the seal into place with a cotton-tipped swab (or other tool with a soft tip).
7. Gently insert the piston into the new seal in the left end plate. Push the pump cylinder over the O-ring on the left end plate. Push the pump cylinder over the O-ring on the right end plate.
8. Loosely secure the pump cylinder to the end plates with the two bolts removed previously.
9. Place the assembled pump on a flat surface. Make sure all four alignment marks on the pump cylinder and the end plates are aligned (see [Figure 5-15](#)). Press down on the end plates to square them with each other. The pump should sit evenly on the flat surface without rocking.



Figure 5-15. Alignment Marks on Pump Cylinder and End Plates

10. Tighten the two bolts evenly.
11. Reattach the PC board to the side of the pump.

5.5.5 Reinstalling the Pump

1. Reinstall the pump on the component panel.
2. Reconnect the red gas line to the left press fitting on top of the pump. Reconnect the transparent blue gas line to the right press fitting.
3. Connect the solvent lines to the inlet and outlet check valve housings.

4. Reconnect the multicolored cable to the pump PC board.
5. Reinstall the pump PC board cover.

5.5.6 Completing the Procedure

1. Close the door to the service area.
2. Reinstall the solvent reservoirs.
3. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
4. Turn on the Dionex ASE 350 power switch.
5. Reset the **PUMP SEAL STROKES** counter on the front panel **USAGE LOG** screen (see [Section C.4](#)).

–or–

If the Dionex ASE 350 is running under Chromeleon software control, the **TotalPumpSealStrokes** counter on the Wellness panel (see [Section 2.5.3](#)) must be reset. Contact Technical Support for Dionex products for assistance.

5.6 Replacing the Pressure Relief Valve

1. Turn off the gas and air supplies. Disconnect the gas and air sources at the Dionex ASE 350 rear panel.
2. Turn off the Dionex ASE 350 power switch.
3. To access the service area, rotate the three quarter-turn screws along the edge of the door and swing the door open.
4. The pressure relief valve is installed (see [Figure 5-16](#), ①) directly above the pump on the component mounting panel on the inside of the door. Disconnect the green gas tubing (②) from the press fitting on the valve.

Tip: To disconnect a press fitting, use your fingers (or a small open-end wrench) to press the ring on the fitting *in*, while at the same time pulling the tubing *out*.

5. Use a 1/4-inch open-end wrench (P/N 014605) to disconnect the stainless steel line (③) from the left side of the valve. (This tubing connects to the pump transducer.)
6. Use the 1/4-inch open-end wrench to disconnect the Hastelloy™ line (④) from the right side of the valve. (This tubing connects to the upper AutoSeal arm.)

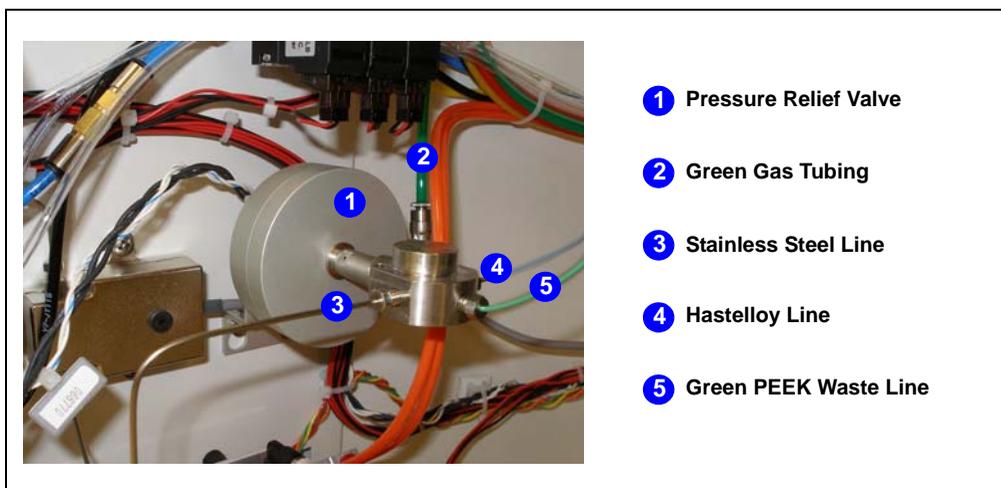


Figure 5-16. Connections to the Pressure Relief Valve

7. Use the 1/4-inch open-end wrench to disconnect the green PEEK waste line (5) from the front of the valve.
8. Use a 9/64-inch Allen wrench to loosen the hex screw (black) on the right side of the clamp ring, behind the valve base (see [Figure 5-17](#)).

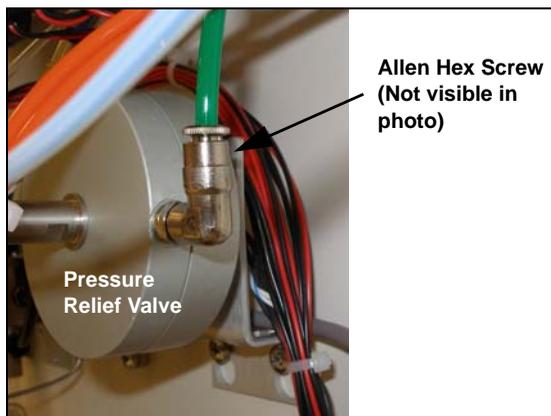


Figure 5-17. Removing the Allen Hex Screw

9. Pull the old valve off the valve base.
10. Insert the new pressure relief valve (P/N 048889) onto the valve base. Tighten the Allen hex screw on the clamp ring.
11. Reconnect all of the lines.
12. Close the door to the service area.
13. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
14. Turn on the Dionex ASE 350 power switch.

5.7 Rebuilding the Static Valve

Leaking from the source needle into the collection vessel when the static valve is closed indicates that the static valve is worn and must be rebuilt. The rebuild procedure consists of replacing a few parts (an O-ring, a filter, and two seals) and cleaning the inside of the valve body.

Before proceeding, check that a Static Valve Repair Kit (P/N 068115) is on hand. The kit contains replacement parts and special tools required to rebuild the valve (see [Table 5-1](#) and [Table 5-2](#)). A Static Valve Repair Kit is included in the Dionex ASE 150/ASE 350 Preventive Maintenance Kit (P/N 068954); it can also be ordered separately.

Part Number	Item	Quantity
065254	Dionex ASE 150/ASE 350 Static Valve Repair Kit Instructions	1
066165	High-pressure flangeless seal	2
067326	Perlast™ O-ring	1
067327	Zirconium filter	1
068116	Static Valve Tool Kit*	1

*A Static Valve Tool Kit is also provided in the Dionex ASE 350 Ship Kit (P/N 066060).

Table 5-1. Static Valve Repair Kit Contents

Part Number	Item	Quantity
049293	Tubing, orange (extension tubing)	60.96 cm (24 in)
057395	Union (for extension tubing)	1
067394	Piston-side seal insertion tool	1
067395	Inlet-side seal insertion tool	1
068117	Alignment tool	1
068118	Spacer tool	1
068119	Push screw tool	1
068245	Seal removal tool	1

Table 5-2. Static Valve Tool Kit Contents

[Figure 5-18](#) illustrates the tools in the Static Valve Tool Kit.



Figure 5-18. Static Valve Tools

Additional Required Items

- #2 Phillips screwdriver
- 1/4-inch open-end wrench (P/N 014605)*
- 2.5-mm hex wrench
- 5/8-inch open-end wrench
- Large flathead screwdriver
- Cotton-tipped swabs (or other tool with a soft tip)
- Isopropyl alcohol (IPA) or acetone
- Tie wraps (P/N 013331)*

* Provided in the Dionex ASE 350 Ship Kit (P/N 066060)

5.7.1 Removing the Static Valve from the System

1. Turn off the gas and air supplies. Disconnect the gas and air sources at the Dionex ASE 350 rear panel.
2. Turn off the Dionex ASE 350 power switch.
3. Close the safety shield.
4. Use a Phillips screwdriver to remove the four screws along the top of the safety shield (see [Figure 5-19](#)). (The screws attach the shield to the sliders.) Remove the safety shield and set it aside. **Do not move the sliders.**

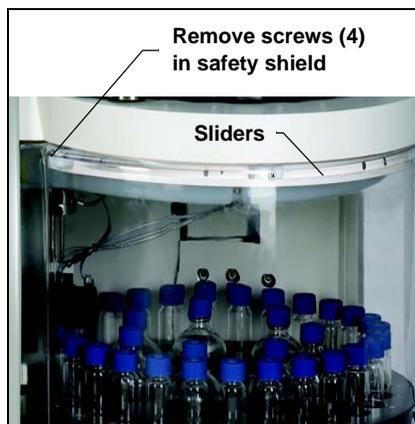


Figure 5-19. Removing the Safety Shield

- Note the three pieces of tubing routed underneath the upper drip tray and held in place with tie wraps. Cut off the tie wrap indicated in [Figure 5-20](#).



Figure 5-20. Tie Wrap Removal

- Remove the three screws that hold the drip tray in place under the cell tray. (There is a screw in the back of the tray, in the front, and on the right.)
- Slowly move the drip tray down and to the right; this will pull the drain tube connected to the drip tray through the hole in the wall that separates the service area from the trays.
- Gently pull just enough of the drain tube through the hole to allow you to place the drip tray on top of the collection tray.

NOTE The drain tube is attached to the drip tray via a barbed fitting. Be careful not to break the barb or kink the drain tube.

9. The static valve is located above the drip tray. Using a 1/4-inch open-end wrench (P/N 014605), disconnect the two Hastelloy lines from the valve (see [Figure 5-21](#), ①).

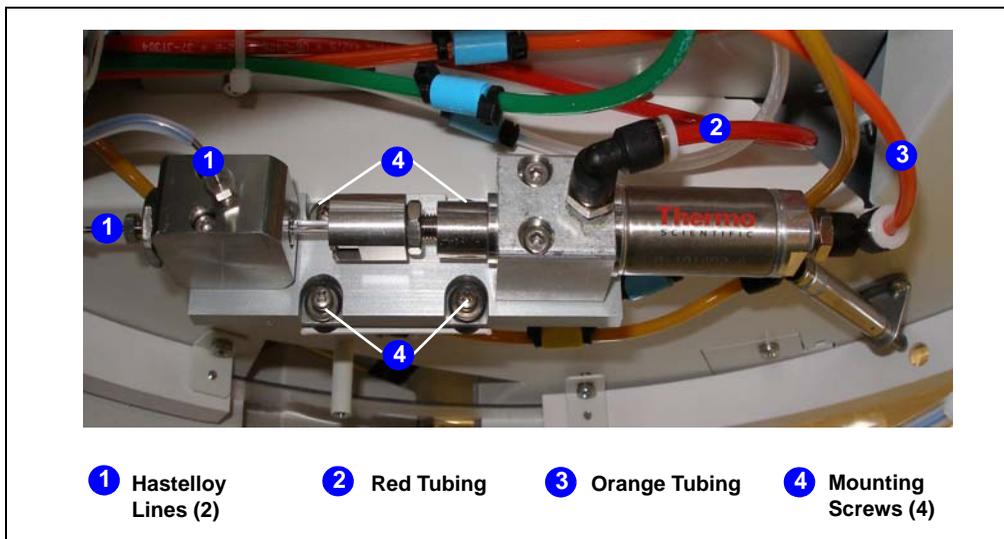


Figure 5-21. Removing the Static Valve

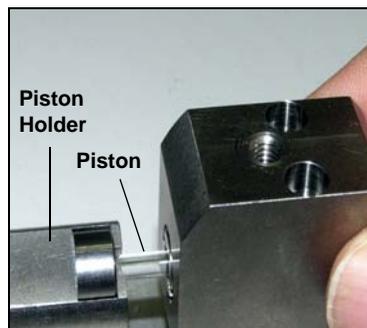
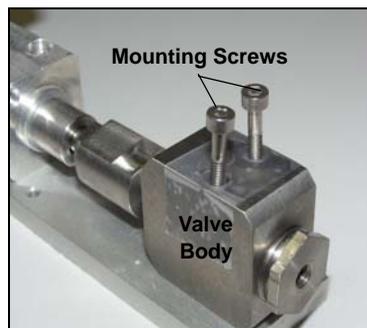
10. Disconnect the red tubing (②) and the orange tubing (③) from the press fittings on the valve.
Tip: To disconnect a press fitting, use your fingers (or a small open-end wrench) to press the ring on the fitting *in*, while at the same time pulling the tubing *out*.
11. Using a 2.5-mm hex wrench, remove the four screws (④) that secure the static valve to the Dionex ASE 350.
12. Remove the static valve assembly from the system and place it on the workbench (see [Figure 5-22](#)).



Figure 5-22. Static Valve

5.7.2 Disassembling the Static Valve Body

1. Using a 2.5-mm hex wrench, remove the two mounting screws that secure the valve body to the base plate of the valve assembly.
2. Remove the piston from the static valve.
 - a. Locate the slot in the piston holder. If necessary, rotate the piston holder so that the opening in the slot faces up.
 - b. Guide the piston out of the piston holder by lifting the valve body straight up. (This removes both the piston and the valve body.) **Do not drop the piston.**



IMPORTANT

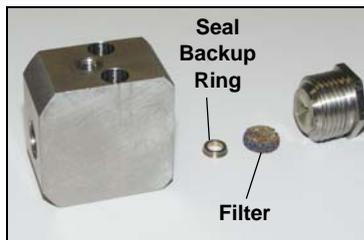
Dropping the piston may cause scratches or breakage. If the piston is damaged, it cannot be used again and must be replaced.

Dionex ASE 350 Solvent Extraction System

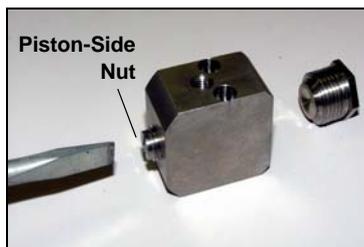
- c. Pull the piston out of the valve body and place in a secure location.
3. Remove the nut installed in each side of the valve body.
 - a. Using a 5/8-inch open-end wrench, remove the nut on the inlet side of the valve body.



- b. Remove the seal backup ring and the filter from the inlet side (they may fall out on their own). **Save the seal backup ring.** You can discard the filter.



- c. Using a large flathead screwdriver, remove the nut on the piston side of the valve body.



IMPORTANT

Do not use a sharp object inside the valve body. Any scratches on the valve body will prevent a proper seal and cause leakage.

4. Insert the flat end of the seal removal tool (P/N 068245) into the piston side of the valve body and push the inlet-side seal out of the valve body.



5. Pull the seal removal tool out of the valve body.
6. Insert the flat end of the seal removal tool into the inlet side of the valve body and push the seal out of the piston side of the valve body.



7. Insert the pointed end of the seal removal tool into the valve body. Slide the tool between the valve body and O-ring and pull the O-ring out of the valve body.



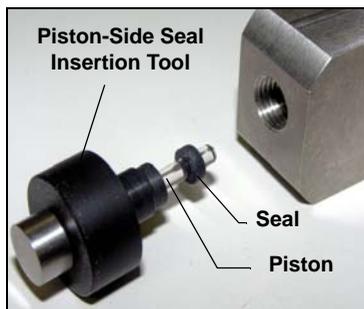
8. Using a cotton-tipped swab (or other tool with a soft tip), thoroughly clean the inside of the valve body. If necessary, moisten the area with IPA (isopropyl alcohol) or acetone to remove all debris, sediment, and precipitates.

IMPORTANT

Do not use a sharp object to clean the inside of the valve body. Any scratches on the valve body will prevent a proper seal and cause leakage.

5.7.3 Installing the New Seals

1. Push the piston through the piston-side seal insertion tool (P/N 067394).
2. Wet a new seal (P/N 066165) with IPA (isopropyl alcohol) for lubrication.



3. Note that one side of the seal is flat, while the other side contains an O-ring. Orient the O-ring side of the seal so that it faces away from the tool, and then slide the seal onto the piston.
4. Place the valve body, with the piston side (small opening) facing up, on the workbench.



5. Rest your thumbs on the back of the tool and press down firmly (with equal pressure) on both the left and right sides of the tool. You should hear a snap as the new seal is seated in the valve body. Remove the piston and the tool from the valve body.



NOTE: If there is no audible snap, check the seal for damage. An undamaged seal will lie completely flat in the valve body, with no visible edges. If the seal is damaged, remove it from the valve body and order a replacement seal.

IMPORTANT

Do not use a sharp object inside of the valve body. Any scratches on the valve body will prevent a proper seal and allow leakage.

6. Place a new seal (P/N 066165), with the O-ring side facing up, into the inlet gland (large opening) of the valve body.



7. Use the inlet-side seal insertion tool (P/N 067395) to press the new seal into place.



5.7.4 Reassembling the Static Valve

1. Place the seal backup ring, with the flat side facing up, on top of the seal in the inlet gland. When correctly installed, the backup ring completely covers the mounting surface.

NOTE: The seal backup ring was removed from the valve body in [Step 1](#) on [page 169](#).



2. Place the new filter (P/N 067327) on top of the backup ring. Check that the filter is seated, lies flat, and is centered in the inlet gland.



3. Insert the new O-ring (P/N 067326) into the cavity and press it all the way into the cavity, using the flat end of the seal removal tool.



IMPORTANT

When completing the valve reassembly, make sure the valve body remains upright to avoid dislodging the backup ring, filter, and O-ring.

4. Reinstall the nut on the inlet side of the valve body and tighten with a 5/8-inch open-end wrench. Use another open-end wrench to hold the valve body in place while tightening the nut.



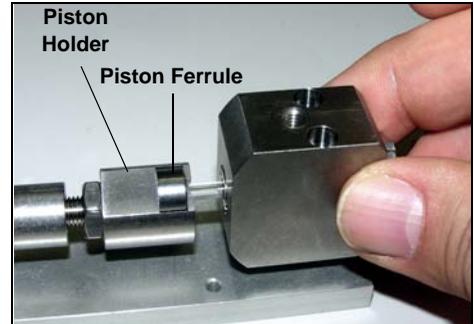
5. Reinstall the nut on the piston side of the valve body and tighten with a flathead screwdriver.



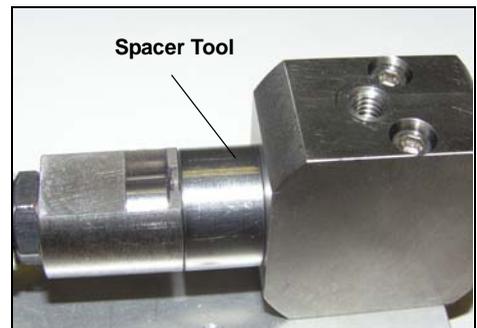
6. Insert the piston into the valve body.



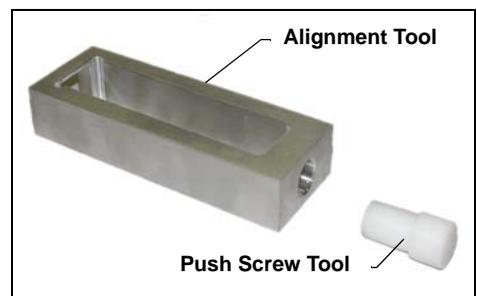
- Slide the piston ferrule onto the piston holder at the end of the cylinder.



- Replace the two mounting screws in the valve body and screw them lightly into the valve body to secure the body to the base plate. **Do not fully tighten the screws yet.**
- Slide the spacer tool (P/N 068118) between the valve body and the piston ferrule.



- Screw the push screw tool (P/N 068119) into the opening in the end of the alignment tool (P/N 068117).



11. Place the alignment tool on top of the static valve.



12. Reconnect the gas source and turn on the gas supply. Check the red and orange lines to see which one is dispensing gas; connect this line to the press fitting on the back of the valve cylinder.

If the gas line is too short to reach the valve, use the union (P/N 057395) to connect the piece of orange extension tubing (P/N 049293) to the end of the gas line.

13. The gas pressure will extend the cylinder shaft fully and hold the spacer tool firmly in place between the valve body and the piston holder.



14. Start turning the push screw tool clockwise until the opposite side of the alignment tool touches the back of the cylinder. With the cylinder shaft still extended, fully tighten the valve body mounting screws that you partially tightened in [Step 8](#).



15. Turn the push screw tool counterclockwise. Remove the alignment tool from the valve.

16. Turn off the gas supply and disconnect the gas line from the valve. Remove the orange extension tubing, if used.
17. Slide the cylinder shaft toward the cylinder. Remove the spacer tool from between the valve body and the piston ferrule.

5.7.5 Reinstalling the Static Valve

1. Reattach the static valve to the Dionex ASE 350, using the four mounting screws removed in [Section 5.7.1](#). Use a 2.5-mm hex wrench to tighten the screws. Tighten until the screw head touches the rubber bumper on the mounting bracket, and then turn an additional one-half turn. **Do not overtighten!**
2. Reconnect all fluid and gas connections to the static valve.
3. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
4. Turn on the Dionex ASE 350 power switch.
5. Test the system for leaks.
 - a. Install one of the following in the cell tray: a “blank” sample cell of any size filled with Ottawa sand (Fisher S23-3) *or* an empty sample cell with a volume of less than 34 mL.

NOTE A “blank” cell contains no sample.

- b. Install a collection vessel in position 1 of the collection tray.
- c. Check that the waste bottle is installed.
- d. Press **Menu** to display the **MAIN MENU OF SCREENS** and press **3** to display the **METHOD EDITOR** screen.

- e. Create a method with the following values:

TEMPERATURE	0 °C
STATIC TIME	1 min
CYCLES	1
RINSE VOLUME	0%
PURGE	60 s (If the test solvent is water, set the purge time to 120 s.)
SOLVENT A	1
SOLVENT B	0
SOLVENT C	0
CELL TYPE	SST
SOLVENT SAVER	OFF

- f. When you finish selecting parameters, save the method.
- g. Press **Menu** to return to the **MAIN MENU OF SCREENS** and press **1** to display the **LOAD METHOD/SEQUENCE** screen.
- h. Verify that **METHOD** appears in the **LOAD** field. If it does not, press **Select** Δ to toggle to **METHOD**.
- i. Move the cursor to the **NUMBER** field and enter the number of the method you created for the leak test. Press **Enter** or a cursor arrow.
- j. Move the cursor to the **BTL/VIAL #** field, press **1**, and press **Enter**. This closes the **LOAD METHOD/SEQUENCE** screen and opens the **SYSTEM STATUS** screen.
- k. Press **Start** to begin the run.
- l. During the static cycle, check the 5/8-inch nut connection on the inlet side of the valve body for leaks. If there is a leak, use a 5/8-inch open-end wrench to tighten the connection. Use another wrench to hold the valve body in place while tightening the nut.

During the static cycle, also check the tip of the source needle (the needle with the smaller diameter, near the front of the needle assembly) in the collection vessel. If liquid drips from the source needle into the collection vessel during the static cycle (when the static valve should be closed), it indicates that the valve was not

rebuilt correctly. Press **STOP** to stop the run (or wait for the method to finish), and then reassemble the valve correctly.

- m. When the system passes the leak test, go on to [Step 6](#).
6. Gently feed the drain tube connected to the upper drain tray through the hole in the side wall and back into the service area. Replace the three screws that hold the drip tray in place under the cell tray.

NOTE The drain tube is attached to the drip tray via a barbed fitting. Be careful not to break the barb or kink the drain tube.

7. Replace the tie wrap removed from the tubing underneath the drip tray (see [Figure 5-20](#)) with a new tie wrap (P/N 013331).
8. Reinstall the safety shield.
9. Reset the **STATIC TOTAL CYCLE** and **STATIC SEAL CYCLES** counters on the front panel **USAGE LOG** screen (see [Section C.4](#)).

–or–

If the Dionex ASE 350 is running under Chromeleon software control, the **TotalStaticValveCycle** and **TotalStaticValveSealCycle** counters on the Wellness panel (see [Section 2.5.3](#)) must be reset. Contact Technical Support for Dionex products for assistance.

5.8 Replacing the Source Needle

1. Turn off the Dionex ASE 350 power switch.
2. Disconnect the compressed air or nitrogen gas connections at the Dionex ASE 350 rear panel. (This allows you to move the needle assembly manually.)
3. Open the safety shield.
4. Remove the collection bottles from the tray. With the needle assembly positioned over the outside row of the tray, push the needle assembly down.

5. Use a 2.5-mm hex wrench to remove the two screws on the top of the needle block (see [Figure 5-23](#)).

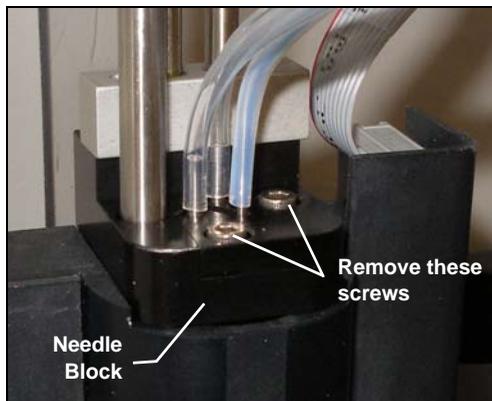


Figure 5-23. Removing the Needle Block Screws

6. Slide the cover of the needle block to the front and right, away from the needle (see [Figure 5-24](#)). Remove the cover.

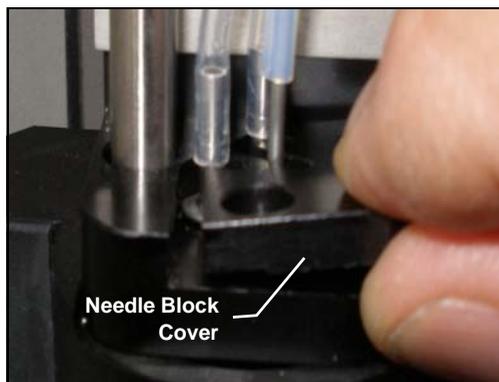


Figure 5-24. Removing the Needle Block Cover

7. Lift up the source needle to remove it from the needle block (see [Figure 5-25](#)).

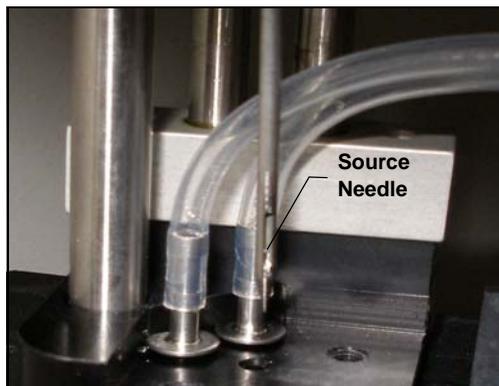


Figure 5-25. Removing the Source Needle from the Needle Block

8. Note the three pieces of tubing routed underneath the upper drip tray (see [Figure 5-26](#)). Cut off the tie wraps that hold the tubing together. Then, cut off the tie wrap that attaches all of this tubing to the upper drip tray.

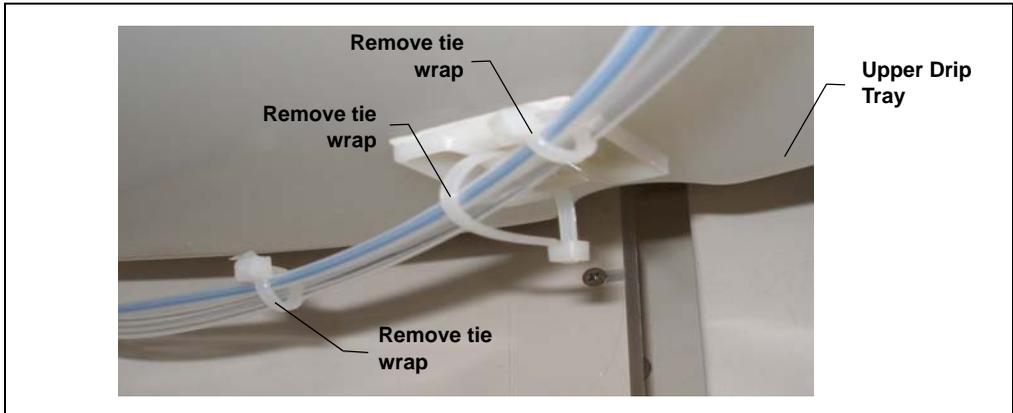


Figure 5-26. Removing Tie Wraps (Typical Installation Points)

9. Remove the three screws that hold the drip tray in place under the cell tray. (There is a screw in the back of the tray, in the front, and on the right.)
10. Slowly move the drip tray down and to the right; this will pull the drain tube connected to the drip tray through the hole in the wall that separates the service area from the trays.
11. Gently pull just enough of the drain tube through the hole to allow you to place the drip tray on top of the collection tray.

NOTE The drain tube is attached to the drip tray via a barbed fitting. Be careful not to break the barb or kink the drain tube.

12. Use a 1/4-inch open-end wrench to disconnect the fitting on the side of the static valve (see [Figure 5-27](#)).



Figure 5-27. Disconnecting the Source Needle Fitting from the Static Valve

13. Remove the source needle and tubing from the Dionex ASE 350.

IMPORTANT The new source needle is pre-bent at the angle required for operation. Do not straighten the needle (or bend it more).

14. Insert the new source needle assembly (P/N 068955) into the middle opening in the needle block (see [Figure 5-28](#)) just until the washer meets the top surface.

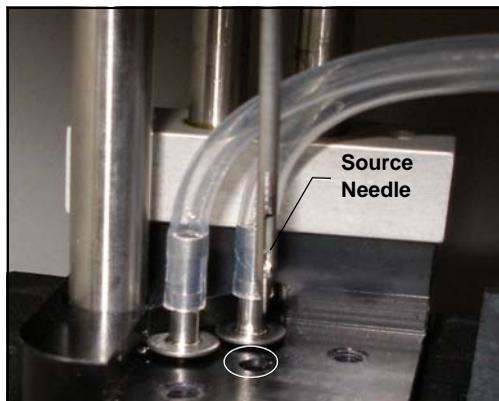


Figure 5-28. Installing the New Source Needle

15. Replace the cover on the needle block and reinstall the two screws.
16. Connect the free end of the source needle tubing to the side of the static valve.

17. Gently feed the drain tube connected to the upper drip tray through the hole in the side wall and back into the service area. Replace the three screws that hold the drip tray in place under the cell tray.

NOTE The drain tube is attached to the drip tray via a barbed fitting. Be careful not to break the barb or kink the drain tube.

18. Locate the tie wraps (P/N 013331) provided in the Dionex ASE 350 Ship Kit (P/N 066060). Install new tie wraps to support the three pieces of tubing.
19. Install an additional tie wrap to attach the pieces of tubing to the upper drip tray. Leave some slack in this tie wrap (see [Figure 5-26](#)).
20. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
21. Turn on the Dionex ASE 350 power switch.

5.9 Replacing the Hydrocarbon Sensor

A failed (or failing) hydrocarbon sensor can have reduced sensitivity.

1. Turn off the gas and air supplies. Disconnect the gas and air sources at the Dionex ASE 350 rear panel.
2. Turn off the Dionex ASE 350 power switch. Disconnect the power cord from both its source and the Dionex ASE 350 right-side panel.



HIGH VOLTAGE—Disconnect the main power cord from its source, as well as from the right-side panel of the Dionex ASE 350.



HAUTE TENSION—Débranchez le cordon d'alimentation principal du panneau latéral droit de le Dionex ASE 350.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der rechten Seite des Dionex ASE 350.

Dionex ASE 350 Solvent Extraction System

3. Use a #1 Phillips screwdriver to remove all of the screws in the rear cover of the Dionex ASE 350. Remove the rear cover and set it aside.
4. The hydrocarbon sensor (see [Figure 5-29](#), ①) is installed in the upper-right corner, behind the fan (②).

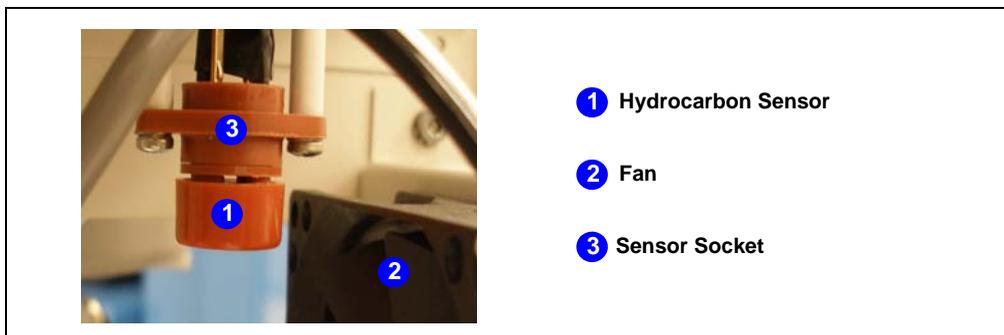


Figure 5-29. Hydrocarbon Sensor

5. The sensor is plugged into a socket (③) that is connected to an electrical cable. To remove the sensor from the socket, wiggle it and gently pull it out of the socket.
6. Plug the new hydrocarbon sensor (P/N 938914) into the socket.
7. Replace the rear cover of the Dionex ASE 350.
8. Reconnect the gas and air sources at the Dionex ASE 350 rear panel. Turn on the gas and air supplies.
9. Reconnect the main power cord and turn on the Dionex ASE 350 power switch.
10. Thermo Fisher Scientific recommends equilibrating the sensor overnight. If this is not possible, wait at least two hours for the signal to stabilize before resuming operation.

5.10 Replacing the Main Power Fuses

1. Turn off the Dionex ASE 350 power switch. Disconnect the power cord from both its source and from the Dionex ASE 350 right-side panel.



HIGH VOLTAGE—Disconnect the main power cord from its source, as well as from the right-side panel of the Dionex ASE 350.



HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau latéral droit de le Dionex ASE 350.



HOCHSPANNUNG—Ziehen Sie das Netzkabel aus der Steckdose und der Netzbuchse auf der rechten Seite des Dionex ASE 350.

2. The fuse drawer is located above the main power switch (see [Figure 5-30](#)). A small tab locks the fuse drawer in place. Using a small screwdriver, press the tab *in* and then *up* to release the fuse drawer.
3. Pull the fuse drawer out of the rear panel and remove the old fuses. Thermo Fisher Scientific recommends always replacing *both* fuses.
4. Insert two new 10 amp IEC127 fast-blow fuses (P/N 954746) into both of the springs in the fuse drawer. Press gently to fully insert the fuses into the drawer.

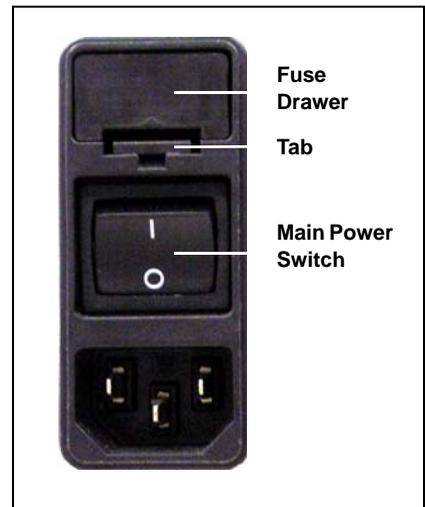


Figure 5-30. Fuse Drawer

5. Insert the fuse drawer into the right-side panel and press until the drawer snaps into place.
6. Reconnect the main power cord and turn on the Dionex ASE 350 power switch.

A.1 Electrical

Power Recommendations	100 to 120 VAC <i>or</i> 220 to 240 VAC; 50/60 Hz Note: The voltage range is configured at the factory to meet installation site requirements.
Power Limitations	The Dionex ASE 350 is functional at a low of 90 VAC and a high of 264 VAC.
Typical Operating Power	500 W
Maximum Operating Power	1000 W
Maximum Line Draw	8 A at 110 VAC
Oven Power Requirements	Either 120 VAC <i>or</i> 240 VAC (depending on the factory-configured voltage range)
Fuse Requirements	Two IEC127 fast-blow fuses (P/N 954746) rated at 10 A

A.2 Environmental

Operating Temperature	4 to 40 °C (39.2 to 104 °F) Note: The Dionex ASE 350 is intended for indoor operation only.
Operating Humidity	5% to 95% relative humidity (noncondensing)

A.3 Physical

Dimensions	Height: 69.3 cm (27.3 in)
	Width: 67.3 cm (26.5 in)
	Depth: 61.7 cm (24.3 in)
Weight	65 kg (140 lb)

A.4 Pneumatic

Laboratory Air	0.41 to 1.38 MPa (60 to 200 psi) Note: If an inlet filter assembly (P/N 049492) is installed, do not exceed 0.86 MPa (125 psi).
Nitrogen	0.97 to 1.38 MPa (140 to 200 psi)

A.5 Front Panel Display and Keypad

Display	Liquid crystal with adjustable contrast
Keypad	26-button keypad for entering commands and programming methods and sequences

A.6 Sample Cells and Tray

Stainless Steel Sample Cells	Stainless steel cell bodies and end caps with PEEK seals and stainless steel frits; cells are available in the following sizes: <ul style="list-style-type: none">• 1 mL (actual volume: 1.0 mL)• 5 mL (actual volume: 5.0 mL)• 10 mL (actual volume: 10.0 mL)• 22 mL (actual volume: 22.0 mL)• 34 mL (actual volume: 34.0 mL)• 66 mL (actual volume: 66.0 mL)• 100 mL (actual volume: 99.8 mL)
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Zirconium Sample Cells	Zirconium cell bodies and end caps with PEEK seals and zirconium frits; cells are available in the following sizes: <ul style="list-style-type: none"> • 66 mL (actual volume: 66.0 mL) • 100 mL (actual volume: 99.8 mL)
Cell Tray	24 sample cell positions and 2 rinse positions

A.7 Collection Vessels and Tray

Collection Bottles	250-mL bottle; the bottle septum is PTFE-coated on the solvent side
Collection Vials	60-mL vial; the vial septum is PTFE-coated on the solvent side
Collection Tray	Holds up to 14 collection bottles on the outside row, plus five collection bottles and two rinse bottles on the inside row. When a vial tray insert (P/N 066176) is installed: Holds up to 28 60-mL collection vials on the outside row, plus five 250-mL collection bottles and two rinse bottles on the inside row.

A.8 Interior Components

Oven	Accommodates one cell at a time in a vertical position; heats from 40 to 200 °C (104 to 392 °F) Note: The oven turns off automatically after four hours of no system activity, provided that no method or sequence is running.
Pump	Operating pressure of 10.34 MPa (1500 psi)
Valves	High-pressure valves: pressure relief and static Low-pressure valves: purge, prime, and pneumatics
Collection Vessel Sensors	Sensors detect whether a collection vessel is present and whether it is full
Leak Sensors	Sensors detect hydrocarbon vapor and liquid leaks
Cell Sensors	Sensors detect cell size

B.1 Facility Requirements

Provide the following installation site facilities for the Dionex ASE 350:

- A sturdy table or workbench in a location that minimizes Dionex ASE 350 exposure to direct sunlight. Allow at least 6 cm (2.5 in) of free space behind the Dionex ASE 350 for connections and ventilation.



Use caution when lifting the Dionex ASE 350. Two or more persons should lift the module, which weighs 65 kg (140 lb). Lift only from the bottom surface.



Soyez prudent lorsque vous soulevez le Dionex ASE 350. Au moins deux personnes devraient soulever le module qui pèse 65 kg. Soulevez seulement du fond.



Seien Sie vorsichtig, wenn Sie den Dionex ASE 350 anheben. Das Gerät wiegt 65 kg. Wenn Sie den Dionex ASE 350 anheben oder bewegen möchten, greifen Sie bitte immer unter den Boden.

- A source of laboratory compressed air (if possible):
 - If the air source does not contain VOCs or oil, regulate the pressure to between 0.41 and 1.38 MPa (60 and 200 psi); 0.69 MPa (100 psi) is recommended.
 - If the air source might contain VOCs or oil, install an inlet filter assembly (P/N 049492) (see [Section B.3](#)). When the filter is installed, do not exceed 0.86 MPa (125 psi); 0.69 MPa (100 psi) is recommended.
- If compressed air is unavailable, substitute nitrogen (see [Section B.3](#)).
- A source of 99.9% pure nitrogen gas, regulated to between 0.97 and 1.38 MPa (140 and 200 psi); 1.03 MPa (150 psi) is recommended. Ultra-high purity (UHP) gas may be required for applications that require a very clean baseline from an electron capture detector (ECD).
- A grounded, single-phase power source of either 120 VAC or 240 VAC, depending on the voltage configuration set at the factory. A label on the right-side panel (see [Section 2.2](#)) indicates the voltage configuration.

B.2 Getting Ready to Install the Dionex ASE 350

Before beginning the installation procedure, check that the Dionex ASE 350 Ship Kit (P/N 066060) is on hand. Also locate the Dionex ASE 350 Startup Kit and verify that the kit is appropriate for the sample cell size you plan to use (see below). Both kits contain several items that are required to install the system.

Small Volume Startup Kit

Use the Small Volume Startup Kit (P/N 068254) with the following sample cell sizes: 1 mL, 5 mL, 10 mL, and 22 mL.

Part Number	Item	Quantity
048784	Collection vials, 60 mL	Pkg. 72
049482	Cell End Cap PTFE O-Ring Kit	Pkg. 10
061687	PEEK seals	Pkg. 50
066176	Vial tray insert	1
068075	Sample funnel, 5 to 22 mL	1
068076	Sample funnel, 1 mL	1
068093	Cellulose filters, 27 mm	Pkg. 100

Table B-1. Dionex ASE 350 Small Volume Startup Kit Contents

34-mL Volume Startup Kit

Use the 34-mL Volume Startup Kit (P/N 068255) with 34-mL sample cells only.

Part Number	Item	Quantity
048784	Collection vials, 60 mL	Pkg. 72
049482	Cell End Cap PTFE O-Ring Kit	Pkg. 10
056699	Sample funnel, 34 to 100 mL	1
056780	Cellulose filters, 30 mm	Pkg. 100
061687	PEEK seals	Pkg. 50
066176	Vial tray insert	1

Table B-2. Dionex ASE 350 34-mL Volume Startup Kit Contents

Large Volume Startup Kit

Use the Large Volume Startup Kit (P/N 068256) with the following sample cell sizes: 66 mL and 100 mL.

Part Number	Item	Quantity
049482	Cell End Cap PTFE O-Ring Kit	Pkg. 10
056284	Collection bottles, 250 mL	Pkg. 12
056699	Sample funnel, 34 to 100 mL	1
056780	Cellulose filters, 30 mm	Pkg. 100
061687	PEEK seals	Pkg. 50

Table B-3. Dionex ASE 350 Large Volume Start-Up Kit Contents

B.3 Connecting the Air and Nitrogen Sources

This section contains two installation procedures:

- To connect compressed air and nitrogen gas sources to the Dionex ASE 350, go to [page 194](#).
- To configure the system when compressed air is not available, go to [page 196](#).

The air and nitrogen connections on the Dionex ASE 350 rear panel (see [Figure B-1](#)) are press fittings.

- To *connect* a press fitting, firmly push the tubing into the fitting until it is seated.
- To *disconnect* a press fitting, use your fingers (or a small open-end wrench) to press the ring on the fitting *in*, while at the same time pulling the tubing *out*.

Compressed Air and Nitrogen Connections

Refer to [Figure B-1](#) when making air and nitrogen connections.

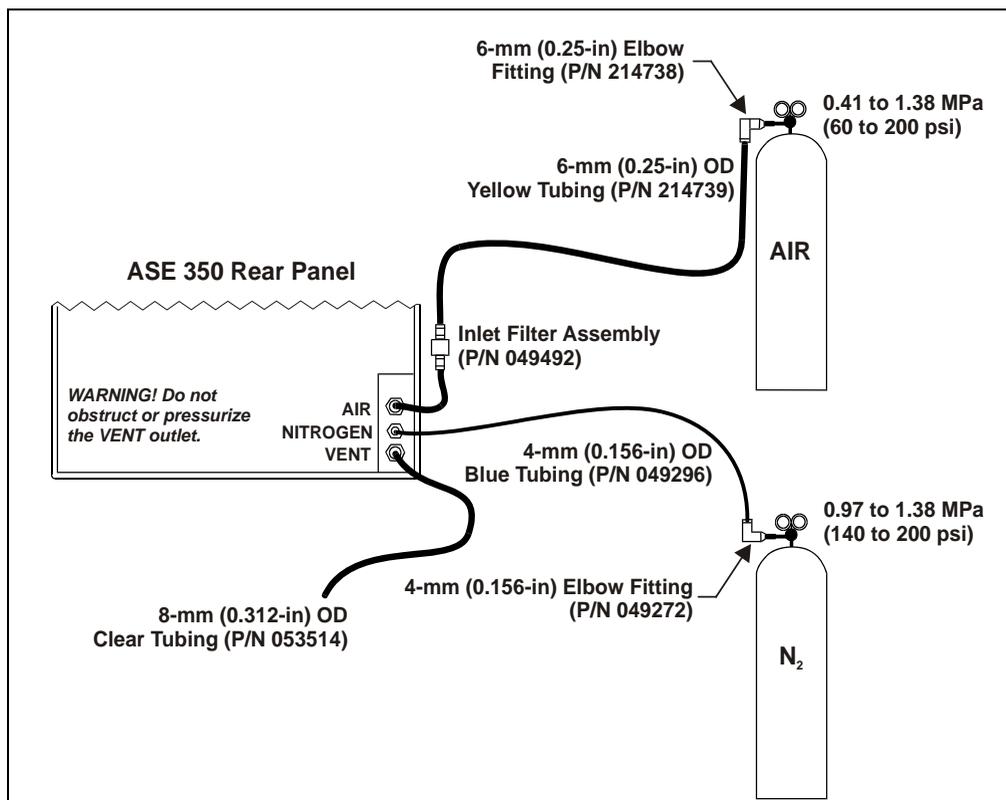


Figure B-1. Dionex ASE 350 Rear Panel Connections

1. (Optional) If the air supply is suspected of containing VOCs or oil, connect an inlet filter assembly (P/N 049492) between the air supply and the Dionex ASE 350 air inlet.
 - a. Check that the arrow on the filter points in the direction of the air flow (toward the Dionex ASE 350).
 - b. Push the tubing on the end of the filter assembly into the **AIR** connector on the Dionex ASE 350 rear panel.
2. Connect the 6-mm (0.25-in) elbow fitting (P/N 214738) to the regulator on the compressed air source.

3. Push one end of the yellow 6-mm (0.25-in) OD tubing (P/N 214739) into the 6-mm (0.25-in) elbow fitting. Push the other end of the tubing into the **AIR** connector on the Dionex ASE 350 rear panel (or into the inlet filter assembly, if installed).
4. Adjust the compressed air source. The recommended pressure is 0.69 MPa (100 psi); the allowed range is 0.41 to 1.38 MPa (60 to 200 psi) unless an inlet filter assembly is installed. If a filter is installed, do not exceed 0.86 MPa (125 psi).
5. Connect the 4-mm (0.156-in) elbow fitting (P/N 049272) to the regulator on the nitrogen gas source.
6. Push one end of the blue 4-mm (0.156-in) OD tubing (P/N 049296) into the 4-mm (0.156-in) elbow fitting; push the other end into the **NITROGEN** connector on the Dionex ASE 350 rear panel.
7. Adjust the nitrogen pressure source to between 0.97 and 1.38 MPa (140 and 200 psi); 1.03 MPa (150 psi) is recommended.
8. Push one end of the 3.05-meter (10-ft) section of clear 8-mm (0.312-in) OD tubing (P/N 053514) into the **VENT** connector on the Dionex ASE 350 rear panel (see [Figure B-1](#)).



Make sure the vent tubing runs downhill from the Dionex ASE 350 rear panel. This prevents formation of a trap, which would prevent vapors from being vented through the tubing.

9. Route the tubing from the **VENT** connector to a fume hood.



Do not obstruct or pressurize the vent outlet.



Ne bouchez ni ne mettez sous pression la sortie d'aération.



Halten Sie den Lüftungsauslaß frei, setzen Sie ihn nicht unter Druck.

10. Push one end of the clear corrugated drain hose (P/N 055075) on to the drip tray drain port on the Dionex ASE 350 rear panel (see [Figure 2-7](#)). If necessary, use a hose clamp (P/N 067402) to attach the hose to the drain port.
11. Place the free end of the drain hose into a waste container. To maintain a positive siphon, position the waste container below the level of the Dionex ASE 350.

IMPORTANT For correct drainage, make sure the drain hose is not bent, pinched, or elevated at any point. Do not allow the end of the drain hose to be submerged in waste liquid.

Substituting Nitrogen for Compressed Air

This section describes how to connect the nitrogen gas source to the **NITROGEN** and **AIR** connectors on the Dionex ASE 350 rear panel. Refer to [Figure B-2](#) when making these connections.

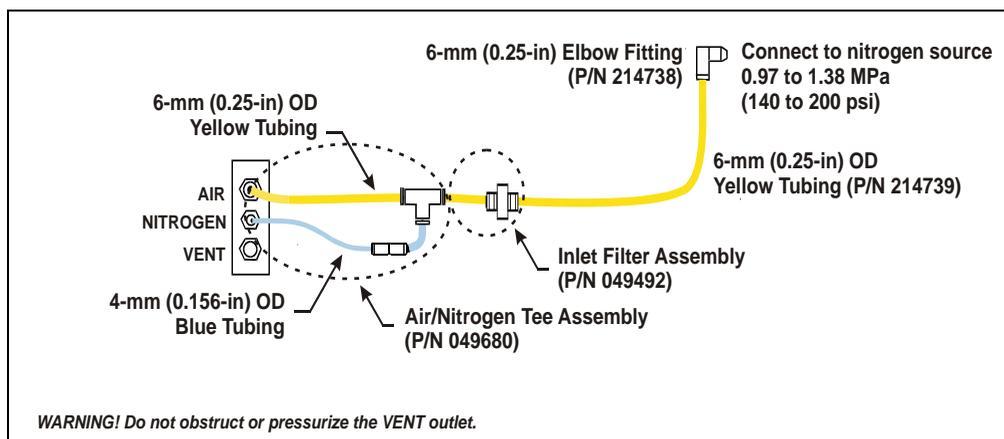


Figure B-2. Connections for Substituting Nitrogen for Air

1. Connect the yellow 6-mm (0.25-in) OD tubing end of the air/nitrogen tee assembly (P/N 049680) to the **AIR** connector on the Dionex ASE 350 rear panel.
2. Connect the blue 4-mm (0.156-in) OD tubing end of the tee assembly to the **NITROGEN** connector on the Dionex ASE 350 rear panel.
3. Connect the 6-mm (0.25-in) elbow fitting (P/N 214738) to the regulator on the nitrogen gas source.

4. Push one end of the yellow 6-mm (0.25-in) OD tubing (P/N 214739) into the 6-mm (0.25-in) elbow fitting; push the other end into the tee assembly.

B.4 Connecting the USB Cable (Optional)

If you plan to use Chromeleon software to control the Dionex ASE 350 remotely, a USB connection is required for communication between the Dionex ASE 350 and the PC on which Chromeleon software is installed. Before connecting the USB cable or turning on the Dionex ASE 350 power, verify that the following tasks have been completed:

- Chromeleon software is installed on the PC.
- The Chromeleon software license is installed.

If necessary, refer to *Chromeleon 7 Installation Guide* (provided on the Chromeleon software DVD) for installation instructions.

NOTE Thermo Fisher Scientific strongly recommends installing Chromeleon software *before* connecting the Dionex ASE 350 to the PC. When you install Chromeleon software first, the USB driver for the Dionex ASE 350 is automatically loaded and the Windows operating system detects the Dionex ASE 350 when the power is turned on.

To connect the USB cable

IMPORTANT

The USB standard limits the USB cable length to 5 meters (16 ft). Each USB device can be separated from the PC by no more than five hubs. Thus, each Dionex ASE 350 can be located no more than 30 meters (32 yds) from the PC. Up to four Dionex ASE 350 systems can be automated by one Chromeleon software workstation.

1. Locate the USB cable (P/N 960777) in the Dionex ASE 350 Ship Kit (P/N 066060).

2. Connect the “B” connector on the USB cable to the USB receptacle on the Dionex ASE 350 rear panel (see [Figure 2-7](#)).
3. Connect the “A” connector on the USB cable to any available USB port on the Chromeleon software PC.

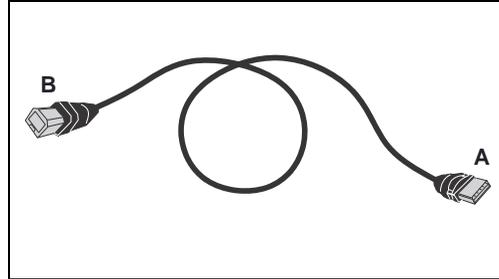


Figure B-3. Standard USB Cable

B.5 Connecting the Power Cord

1. Verify that the power switch on the Dionex ASE 350 right-side panel (see [Figure 2-6](#)) is turned off.
2. Connect a modular power cord (IEC 320 C13) from the main power receptacle on the Dionex ASE 350 right-side panel to a grounded, single-phase power source of 100 to 240 VAC, 50/60 Hz.



SHOCK HAZARD—To avoid electrical shock, a grounded receptacle must be used. Do not operate the Dionex ASE 350 or connect it to AC power mains without an earthed ground connection.



The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the Dionex ASE 350 and is easily accessible.



Operation at AC input levels outside of the specified operating voltage range may damage the Dionex ASE 350.



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



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



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



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

B.6 Setting Up Chromeleon Software (Optional)

When you start the Dionex ASE 350 after connecting to the Chromeleon software PC, the PC detects the presence of the new USB device. You can then create a new Chromeleon software *instrument* and add the Dionex ASE 350 to it. (An instrument consists of all modules that are combined in a single chromatography system to enable chromatographic separations. For extractions, each Dionex ASE 350 must be configured as a different instrument.)

Up to four Dionex ASE 350 systems (each configured as a different instrument) can be automated by one Chromeleon software workstation. Systems are configured in the Instrument Configuration Manager. Refer to *Chromeleon 7 Installation Guide* or the Chromeleon software Help for detailed instructions.

Create a New Instrument in Chromeleon Software

1. Turn on the computer power and log onto Windows as an administrator.
2. Right-click the Chromeleon software tray icon in the notification area of the Windows taskbar, and then click **Show Chromeleon Services Manager**.
3. In the Chromeleon Services Manager, click **Start Instrument Controller**.
4. Right-click the Chromeleon software tray icon, and then click **Configure instruments** to start the Instrument Configuration Manager.
5. Right-click a PC icon in the left pane of the Instrument Configuration Manager, and then click **Add Instrument**. If necessary, edit the default name in the **New Instrument** dialog box.

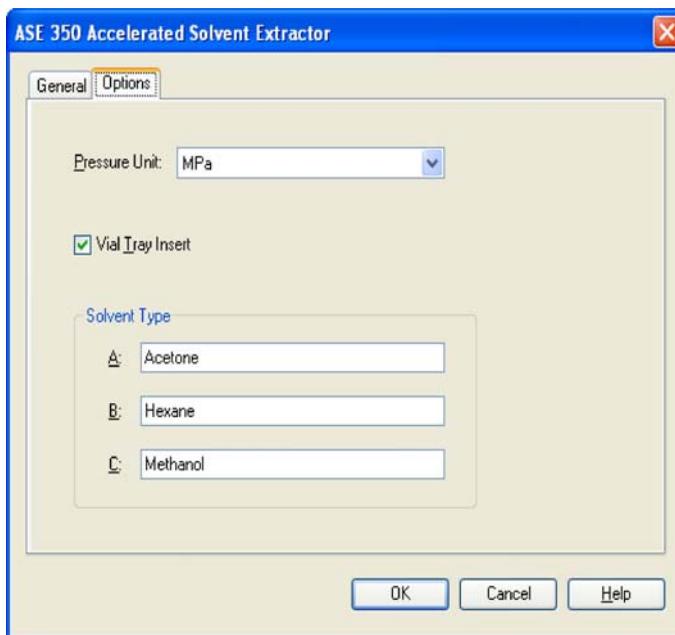
Add the Dionex ASE 350 to a Chromeleon Instrument

1. Right-click the name of the instrument in the Instrument Configuration Manager, and then click **Add Module**. The **Add module to instrument** dialog box appears.

2. In the **Manufacturers** list under **Dionex**, select **Extraction Modules**.
3. In the **Modules** list, select **ASE 350 Accelerated Solvent Extractor**. Click **OK**. The **Properties** dialog box appears.

Configure the Dionex ASE 350 in Chromeleon Software

1. Chromeleon software support requires Dionex ASE 350 firmware version 2.00.00 (or later). If necessary, download a new firmware version from the **General** tab page in the **Properties** dialog box.
 - a. Select the serial number of your Dionex ASE 350 in the **Module Serial No.** list.
 - b. Click **Download**, and then select the appropriate firmware file. The update can take several minutes. The update is complete when the related message appears in the **Messages Instrument Controller** window.
2. On the **Options** tab page (see below), specify the unit of measure for pressure displays and the default solvent names. If the vial tray insert is installed in the collection tray, select the **Vial Tray Insert** check box. When you finish selecting options, click **OK**.



3. Click **File > Save Installation**.
4. Close the Instrument Configuration Manager.

Connect to Chromeleon Software

1. To start the Chromeleon software client, click **Start > All Programs > Chromeleon 7 > Chromeleon 7**.
2. Click the **Instruments** Category Bar in the Console, and then click your instrument in the Navigation Pane. Chromeleon software will connect to the instrument and display the Dionex ASE 350 ePanel (see [Figure B-4](#)).

ASE 350 Extractor Audit Queue

Extractor Status
System Status: Idle
 Method:
 Temperature Set: 40 °C
 Temperature Actual: 40 °C
 Volume Delivered: 0.0 mL
 Time Remaining: 0:01
 Step Time: 0:01
 Cell Size: ---
 Cell Pressure: 0 [psi]

Rinse	Extraction	Method
Idle	Idle	Idle

Manual Rinse
 Volume: 1 mL Ratio A: 1
 Cycles: 1 Ratio B: 0
 Ratio C: 0

Rinse

Mode
Standard

Connected

	Date	Time	Retention Time	Device	Message
1	12/21/2011	1:42:02 PM		Extractor	Connection established successfully.
2	12/21/2011	1:42:02 PM		Extractor	Trying to connect...

Figure B-4. Chromeleon Software: Example Dionex ASE 350 ePanel

3. Check the lower-left corner of the system graphic on the Dionex ASE 350 ePanel:
 - When the Dionex ASE 350 is connected to Chromeleon software, a green button labeled **Connected** is displayed.
 - When a red button labeled **Disconnected** is displayed, click the button to connect the system to Chromeleon software.

B.7 Checking Pressure Readings

1. Turn on the Dionex ASE 350 power switch, if it is not already on.
2. On the **MAIN MENU OF SCREENS**, select **DIAGNOSTICS** and press **Enter**. The **DIAGNOSTIC MENU** appears (see [Figure B-5](#)).

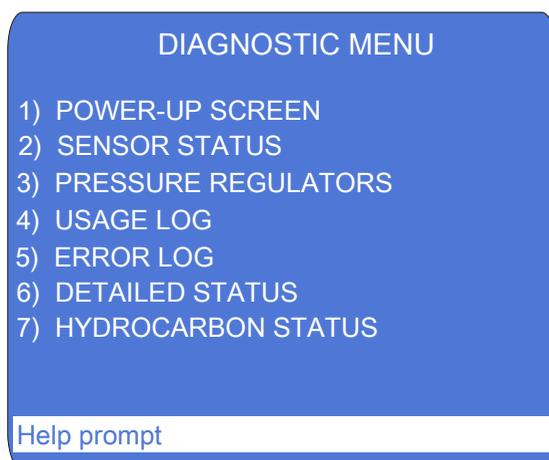


Figure B-5. Diagnostics Menu

3. Select **PRESSURE REGULATORS** and press **Enter**. The **PRESSURE REGULATORS** screen appears (see [Figure B-6](#)).



Figure B-6. Pressure Regulators Screen

NOTE The **COMPRESSION**, **AUTOSEAL**, and **CELL PRESSURE** readings are zero unless a run is in progress and a cell is in the oven.

4. Compare the pressure readings on the **PRESSURE REGULATORS** screen with the values listed in the table below. If a pressure reading does not meet specification, contact Technical Support for Dionex products.

Field	Function	Pressure Spec
SYSTEM AIR	Pressure applied to the system	0.34 ± 0.02 MPa (50 ± 3 psi)
COMPRESSION	Pressure applied when the oven is compressed	0.90 ± 0.03 MPa (130 ± 5 psi)
SOLVENT BOTTLE	Pressure applied to the solvent reservoir	48.26 ± 6.89 kPa (7 ± 1 psi)
AUTOSEAL	Pressure applied when the AutoSeal air cylinder is actuated	172.37 ± 14 kPa (25 ± 2 psi)

B.8 Connecting the Solvent Reservoirs

For information about selecting and preparing solvents, see [Section 3.1.1](#).



Use only Thermo Fisher Scientific solvent reservoirs. These are glass reservoirs with a plastic, shatterproof coating. To prevent personal injury, make sure the pressure applied to the reservoirs does not exceed 0.07 MPa (10 psi).



Utilisez uniquement des réservoirs à solvant Thermo Fisher Scientific. Ce sont des réservoirs en verre à revêtement incassable en plastique. Veillez à ce que la pression exercée sur ces réservoirs ne dépasse pas 0,07 MPa.



Verwenden Sie ausschließlich die Lösemittelbehälter von Thermo Fisher Scientific. Dabei handelt es sich um Glasbehälter mit einer splittersicheren Plastikbeschichtung. Vergewissern Sie sich, daß der Druck, der auf die Behälter ausgeübt wird, 0,07 MPa nicht übersteigt.



Never fill the solvent reservoir or disconnect the tubing connections to the solvent and gas connectors (see [Figure B-7](#)) during a run or rinse cycle. At these times, the solvent reservoir is pressurized. If you remove the reservoir cap when the solvent reservoir is pressurized, the Dionex ASE 350 may not operate to specification.

1. Fill the solvent reservoir (P/N 045901) with prepared solvent (for example, acetone) to the level indicated in [Figure B-7](#).



The solvent level in the reservoir must remain below the gas line. This prevents solvent from coming into contact with the pneumatic valves.

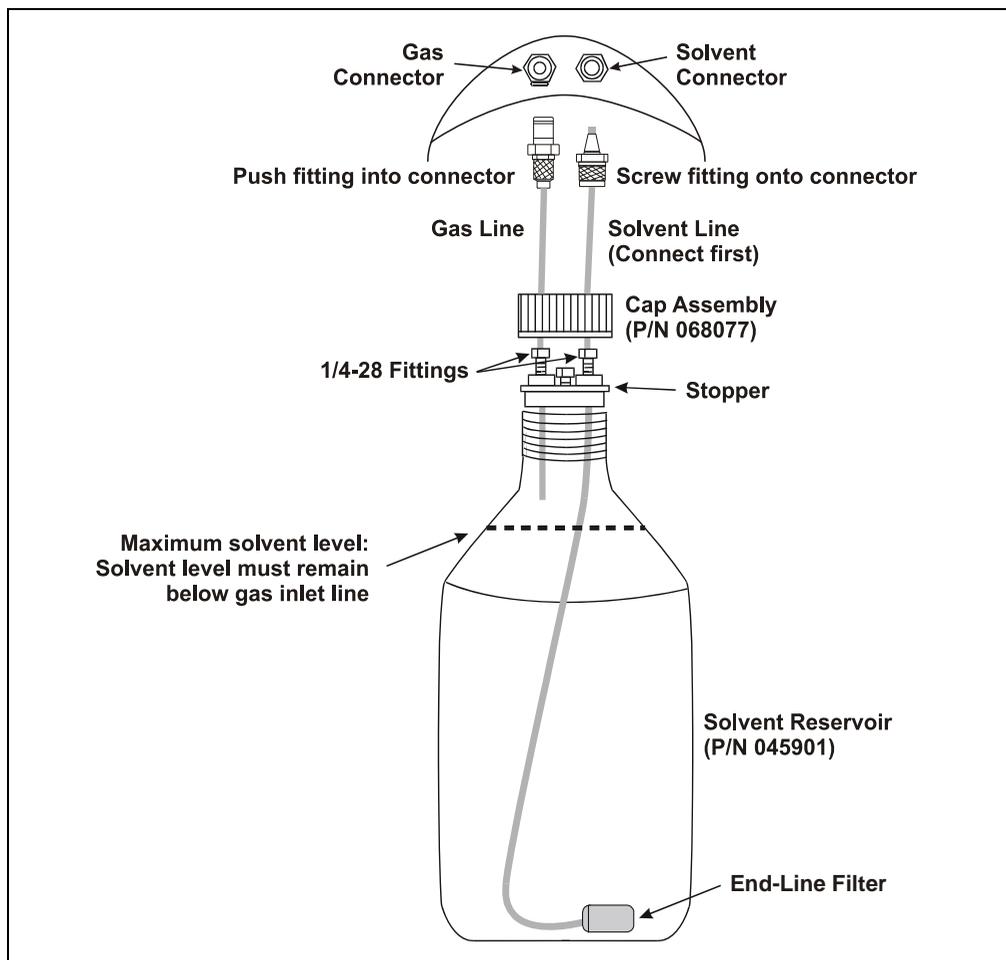


Figure B-7. Solvent Reservoir Connections

2. Insert the solvent line extending from the underside of the reservoir cap assembly (P/N 068077) into the reservoir (see [Figure B-7](#)). Make sure the end-line filter rests on the bottom of the reservoir.

If necessary, loosen the fitting on top of the stopper, gently pull on the solvent line to bring more tubing into the reservoir, and then tighten the fitting.

3. Hand-tighten the gray lock ring cap securely over the stopper.

4. Set the reservoir in the recess reserved for solvent A in the solvent reservoir tray (see [Figure B-8](#)).

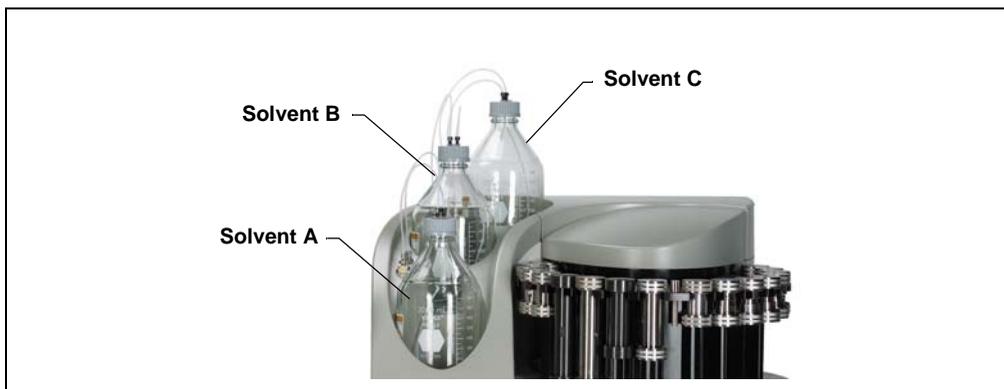


Figure B-8. Location of Solvent Reservoirs

5. Screw the fitting on the solvent line into the **SOLVENT** connector closest to the bottle. Push the fitting on the gas line into the connector closest to the reservoir. (To disconnect the line, push the small latch at the top of the connector toward the center of the connector.)

NOTE Always connect the solvent line to the solvent connector first, and then the gas line to the gas connector. When disconnecting the lines, first disconnect the gas line and then the solvent line. This prevents inadvertent pressurization of the reservoir and solvent flow when it is not connected.

6. Follow the steps above to install additional reservoirs. Be sure to install the reservoirs in the locations indicated in [Figure B-8](#); when proportioning solvents, the method will be programmed according to these positions.

To refill a solvent reservoir:

It is not necessary to disconnect the solvent and gas lines before refilling the reservoir. Loosen the gray lock ring cap, and then remove the stopper and attached tubing from the reservoir. Remove the reservoir from the solvent reservoir tray and place it on a bench for refilling.

B.9 Installing the Waste Bottle

A 250-mL collection bottle (P/N 056284, pkg. of 12), minus the bottle cap, is used to collect condensed, vented solvent during a sample extraction.

The waste bottle is installed in a holder on the inside wall, to the left of the trays (see [Figure B-9](#)).

1. Open the safety shield.
2. With one hand, tilt the black bracket to a 15-degree angle.
3. With your other hand, screw the lip of the bottle into the top opening under the black bracket and then gently release the bottle.
4. Close the safety shield.



Figure B-9. Installing the Waste Bottle

B.10 Installing the Heat Exchanger (Optional)

A heat exchanger (P/N 068247) is required for applications that use volatile solvents at temperatures above 175 °C. The heat exchanger must be installed by qualified Thermo Fisher Scientific personnel.

B.11 Inspecting the Cell and Rinse Tube

Before each run, inspect the O-rings installed in the exterior ends of each sample cell end cap and rinse tube (see [Figure B-10](#)).

PTFE O-rings (P/N 049482, pkg. of 10) are included in the Dionex ASE 350 Startup Kits. For dioxins and other high temperature applications, use Viton O-rings (P/N 056325, pkg. of 50).

- If an O-ring is dislodged, press it into place, using the O-ring insertion tool (P/N 049660) provided in the Dionex ASE 350 Ship Kit (P/N 066060).

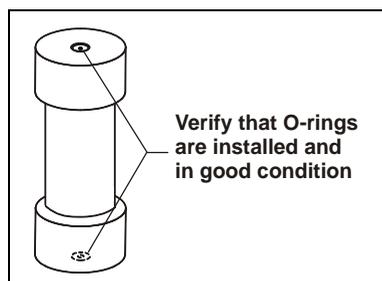


Figure B-10. Inspecting Cell O-Rings

- If an O-ring is missing, place a new one over the opening in the end of the cell end cap. Press the O-ring into place, using the O-ring insertion tool (see [Figure B-11](#)).
- If an O-ring has a hole size of less than 0.5 mm, replace it. If a white PTFE O-ring is discolored, replace it.

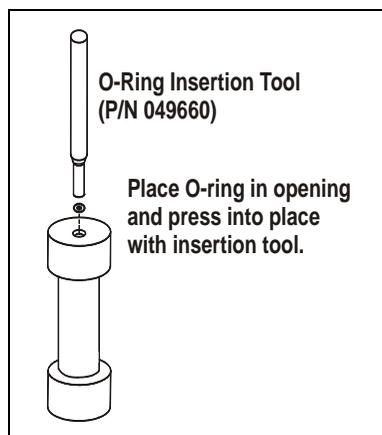


Figure B-11. Installing a Cell End Cap O-Ring

B.12 Rinsing (Priming) the System

Manual rinse cycles are used to rinse the system and to prime the pump and the solvent line.

1. Place the two rinse positions on the cell tray (between positions 6 and 7 and positions 18 and 19).

NOTE Always install rinse tubes in both positions.

2. Place collection vessels in positions R1 and R2 in the collection tray.
3. Close the safety shield.
4. Press the **Menu** button on the control panel keypad to display the **MAIN MENU OF SCREENS** and press **5** to display the **SYSTEM SETUP** screen (see [Figure B-12](#)).

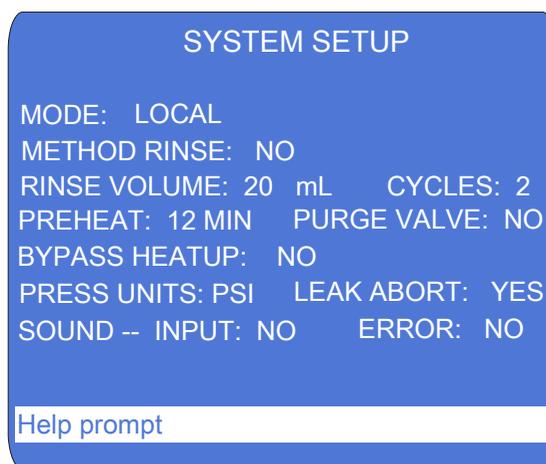


Figure B-12. System Setup Screen

5. Press an arrow button to move the cursor to the **RINSE VOLUME** field. Enter the rinse volume and press **Enter** or a cursor arrow button.
6. Move the cursor to the **RINSE CYCLES** field. Select **2** and press **Enter** or a cursor arrow button. (Up to nine cycles can be programmed.)

7. Press the **RINSE** button to start the manual rinse cycle. During the cycle, the cell tray rotates to the nearest rinse tube, the collection tray rotates to a rinse position, and the selected amount of solvent is pumped through the system. This is followed by a 20-second nitrogen purge step.



To avoid personal injury, exercise caution whenever the tray is in motion.



Pour éviter de vous blesser, faites bien attention lorsque le plateau est en mouvement.



Um Verletzungen vorzubeugen, seien Sie vorsichtig, wenn sich der Probenteller bewegt.

8. After the second rinse, the pump and the solvent line are primed. Remove the rinse bottles from the system, empty them, and place them in positions R1 and R2 in the collection tray.

B.13 System Setup

The **SYSTEM SETUP** screen controls several parameters, including the unit of measure for on-screen pressure displays and whether a beep is emitted each time a button is pressed. [Figure B-13](#) shows the factory default settings; [Table B-4](#) describes each option.

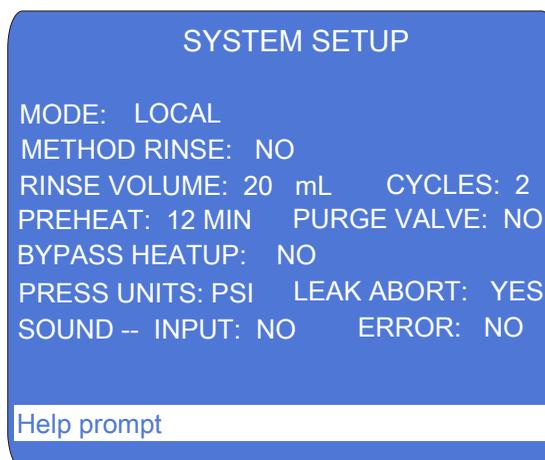


Figure B-13. System Setup Screen

At installation, select the preferred options for the system. These options will remain in effect until you make new selections from the screen; turning the Dionex ASE 350 power off and on does not reset the options to the factory defaults.

To set options:

1. Press the **Menu** button to display the **MAIN MENU OF SCREENS** and press **5** to display the **SYSTEM SETUP** screen.
2. Press an arrow button to move the cursor to an option. For example, press ∇ to move to the **METHOD RINSE** field.
3. In most fields, pressing the **Select** Δ or **Select** ∇ button steps through a list of predetermined options. In the **RINSE VOLUME**, **RINSE CYCLES**, and **PREHEAT** fields, you must press the numeric buttons to enter new values.
4. After selecting a new option, press the **Enter** button or a cursor arrow button to save the change.

Option	Description	Values
MODE	Specifies the Dionex ASE 350 control mode. At power-up, the system is always in Local mode. Remote mode is selected when you connect the Dionex ASE 350 to an ePanel in Chromeleon software. To return the system to Local mode, click the Connected button on the ePanel (to toggle to the Disconnected state) or turn off the Dionex ASE 350 power briefly.	LOCAL (default) REMOTE
METHOD RINSE	Determines whether an automatic rinse (see Section 3.1.10) is performed after each sample run when running under method control.	NO (default) YES
RINSE VOLUME	Specifies the volume of solvent pumped through the system in a single rinse operation. The selected volume will be used during both manual and automatic rinses.	0 to 99 mL (default=5 mL)
RINSE CYCLES	Specifies the number of rinse cycles the system performs during a manual rinse.	1 through 9 (default=1)
PREHEAT	Specifies for how long the cell is preheated before solvent is pumped into the cell.	0 to 99 min (default=0)
PURGE VALVE	Determines whether the purge valve is open or closed during the preheat step. When the valve is open, purge gas flows through the cell.	NO (default) YES

Table B-4. System Setup Parameters

Option	Description	Values
BYPASS HEATUP	<p>Determines whether the initial heat-up step occurs. When NO is selected, the oven block is heated to ensure that the cell contents reach the set point during the heat-up step. All Thermo Scientific Dionex ASE Application Notes are based on this default setting; to obtain equivalent results, do not bypass the heat-up step.</p> <p>When YES is selected, the oven block is not heated; thus, the cell contents probably will not achieve the set point. Do not select this option unless you can achieve complete recovery of analytes without the additional heating.</p> <p>NOTE: The duration of the heat-up step depends on the method's temperature set point (see Table 3-3).</p>	NO (default) YES
PRESS UNITS	Specifies the unit of measure for pressure readings displayed on the Dionex ASE 350 front panel screen.	PSI (default) MPa ATM BAR
LEAK ABORT	Determines whether a run is aborted when a liquid leak is detected. When the default setting (NO) is selected, an error message is displayed but the run continues without interruption.	NO (default) YES
INPUT SOUND	Determines whether a beep is emitted when a keypad button is pushed.	NO (default) YES
ERROR SOUND	Determines whether a beep is emitted when an error occurs (for example, you press the wrong keypad button).	NO (default) YES

Table B-4. System Setup Parameters (Continued)

C • Diagnostic Screens

This appendix describes the Dionex ASE 350 diagnostic screens. To display the **DIAGNOSTIC MENU**, press **6** on the **MAIN MENU OF SCREENS**.

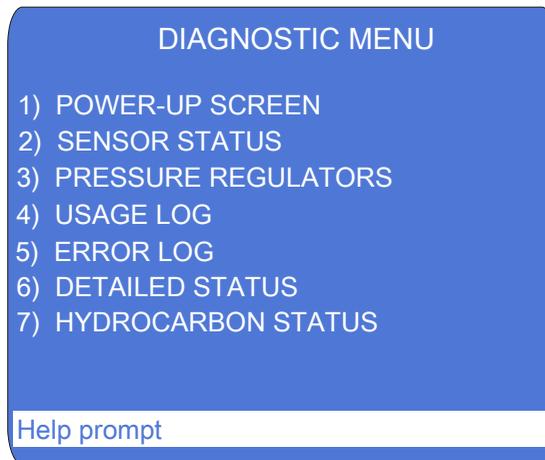


Figure C-1. Diagnostic Menu

There are two ways to select a **DIAGNOSTIC MENU** option:

- Press the numeric button that corresponds to the screen name.
- or–
- Press an up or down arrow button to move the cursor to the screen name, and then press the **Enter** button.

C.1 Power-Up Screen

The **POWER-UP** screen displays the revision numbers of the Dionex ASE 350 internal control programs. The **POWER-UP** screen is also displayed for a few seconds when the Dionex ASE 350 power is turned on.



Figure C-2. Power-Up Screen

C.2 Sensor Status Screen

The **SENSOR STATUS** screen displays the status reported by various internal sensors. Screen entries are updated in real time at 2-second intervals. The information displayed here is read-only and is intended primarily to help Technical Support personnel troubleshoot operating problems with the system.

Not all of the status information can be displayed on the screen at once. To view more information, use the arrow up and down buttons to scroll through the list.

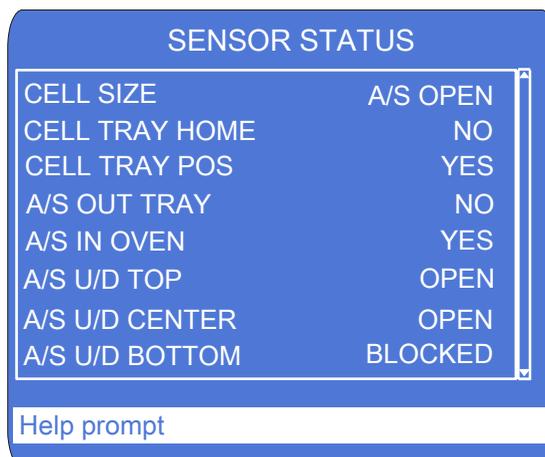


Figure C-3. Sensor Status Screen

Parameter	Description
CELL SIZE	The size of the cell in the oven (1, 5, 10, 22, 34, 66, or 100 mL), NO CELL (when no cell is in the oven), or A/S OPEN (when the AutoSeal arm is open, which prevents detection of the cell size).
CELL TRAY HOME	The cell tray location: YES (the tray is at the home position) or NO (the tray is not at the home position).
CELL TRAY POS	The cell tray location: YES (the tray is at a cell position) or NO (the tray is not at a cell position).
A/S OUT TRAY	The AutoSeal arm location: YES (the arm is at the cell tray) or NO (the arm is not at the cell tray).
A/S IN OVEN	The AutoSeal arm location: YES (the arm is in the oven) or NO (the arm is not in the oven).

A/S U/D TOP	The top AutoSeal “up/down” sensor reading: OPEN or BLOCKED . This sensor works in conjunction with the A/S U/D CENTER and A/S U/D BOTTOM sensors. To identify the AutoSeal arm position, the status of all three sensors must be known.
A/S U/D CENTER	The center AutoSeal “up/down” sensor reading: OPEN or BLOCKED . This sensor works in conjunction with the A/S U/D TOP and A/S U/D BOTTOM sensors. To identify the AutoSeal arm position, the status of all three sensors must be known.
A/S U/D BOTTOM	The bottom AutoSeal arm “up/down” sensor reading: OPEN or BLOCKED . This sensor works in conjunction with the A/S U/D TOP and A/S U/D CENTER sensors. To identify the AutoSeal arm position, the status of all three sensors must be known.
A/S VALVE UP/DN	The AutoSeal “up/down” pneumatic valve state: DN/ON (the valve is open) or UP/OFF (the valve is closed).
A/S VALVE IN	The AutoSeal “in” pneumatic valve state: ON (the valve is open) or OFF (the valve is closed).
A/S VALVE OUT	The AutoSeal “out” pneumatic valve state: ON (the valve is open) or OFF (the valve is closed).
BLT/VIAL PRESENT	PRESENT (a collection vessel is present in this position) or NONE (no collection vessel is present in this position).
BOTTLE OR VIAL	BOTTLE (no vial tray insert is detected) or VIAL (a vial tray insert is detected).
BOTTLE FULL	The collection bottle in this tray position is FULL or NOT FULL .
VIAL FULL	The collection vial in this tray position is FULL or NOT FULL .
VIAL TRAY HOME	The collection tray location: YES (the tray is at the home position) or NO (the tray is not at the home position).
VIAL TRAY POS	The collection tray location: YES (the tray is at a collection vessel position) or NO (the tray is not at a collection vessel position).

NEEDLE UP	The “needle up” sensor reading: YES (the needle is up) or NO (the needle is not up).
NEEDLE DOWN	The “needle down” sensor reading: YES (the needle is down) or NO (the needle is not down).
NEEDLE IN	The “needle in” sensor reading: YES (the needle is at the inside row) or NO (the needle is not at the inside row).
NEEDLE OUT	The “needle out” sensor reading: YES (the needle is at the outside row) or NO (the needle is not at the outside row).
NEEDLE VLV UP/DN	The needle “up/down” pneumatic valve state: DN/ON (the valve is open) or UP/OFF (the valve is closed).
NEEDLE VLV IN	The needle “in” pneumatic valve state: ON (the valve is open) or OFF (the valve is closed).
NEEDLE VLV OUT	The needle “out” pneumatic valve state: ON (the valve is open) or OFF (the valve is closed).
SAFETY SHIELD	The safety shield sensor reading: OPEN (the shield is open) or CLOSED (the shield is either closed or is not installed).
LIQUID LEAK	The drip tray sensor reading: LEAK or NO LEAK .
HYDROCARBON	The hydrocarbon concentration reading (0 to 4700).

C.3 Pressure Regulators Screen

Use the **PRESSURE REGULATORS** screen to monitor various pressure readings for the Dionex ASE 350. The information displayed here is read-only.

Select the unit of measure for these pressure readings on the **SYSTEM SETUP** screen (see [Section B.13](#)).



SOURCE	PRESSURE
SYSTEM AIR	+50.00 PSI
COMPRESSION	+130.00 PSI
SOLVENT BOTTLE	+7.00 PSI
AUTOSEAL	+25.00 PSI
CELL PRESSURE	+1500 PSI

Help prompt

Figure C-4. Pressure Regulators Screen

NOTE The **COMPRESSION**, **AUTOSEAL**, and **CELL PRESSURE** readings are zero unless a run is in progress and a cell is in the oven.

Parameter	Description
SYSTEM AIR	Indicates the pressure from the system air regulator. The specification is 0.34 ± 0.02 MPa (50 ± 3 psi).
COMPRESSION	Indicates the nitrogen gas pressure applied to the oven compression system. When the oven is compressed during an extraction, the reading is 0.90 ± 0.03 MPa (130 ± 5 psi). When the oven is not compressed, this reading is zero.
SOLVENT BOTTLE	Indicates the pressure applied to the solvent reservoirs. The specification is 48.26 ± 6.89 kPa (7 ± 1 psi).

AUTOSEAL	Indicates the pressure applied to the AutoSeal air cylinder. When the air cylinder is actuated, the reading is 172.37 ± 14 kPa (25 ± 2 psi). When the air cylinder is not actuated, this reading is zero.
CELL PRESSURE	Indicates the pressure applied to the cell.

C.4 Usage Log Screen

Use the **USAGE LOG** screen to check the number of extractions the system has run, the number of cycles the static valve has performed, and other performance history. This information can be helpful in scheduling the routine replacement of certain parts.

Not all of the reported information can be displayed on the screen at once; use the arrow up and down buttons to scroll through the list.

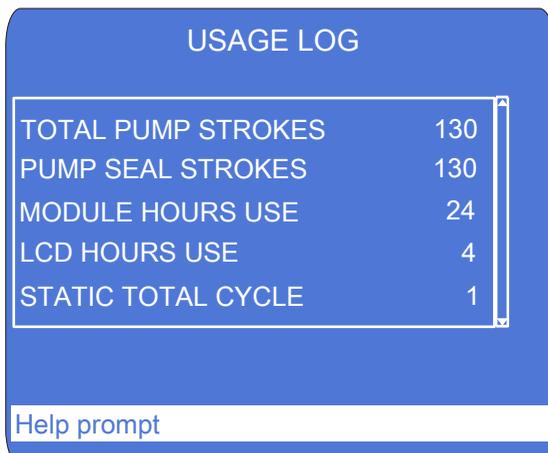


Figure C-5. Usage Log Screen

NOTE You can also view performance history on the Wellness panel in Chromeleon software (see [Section 2.5.3](#)). To reset the usage counters on the Wellness panel, contact Technical Support for Dionex products.

Parameter	Description
TOTAL PUMP STROKES	Indicates the number of pump strokes performed since the Dionex ASE 350 was installed or the pump strokes counter was last reset (read-only).
PUMP SEAL STROKES	Indicates the number of pump strokes performed since the Dionex ASE 350 was installed or the pump piston seals were last replaced. To reset the pump seal counter, press the button with the decimal point on the numeric keypad.
MODULE HOURS USE	Indicates the number of hours the Dionex ASE 350 has been in operation (read-only).
LCD HOURS USE	Indicates the number of hours the front panel display has been lighted (read-only).
STATIC TOTAL CYCLE	Indicates the number of cycles the static valve has performed (read-only).
STATIC SEAL CYCLES	Indicates the number of cycles the static valve seal has performed since the Dionex ASE 350 was installed or the static seals were last replaced. To reset the static seal counter, press the button with the decimal point on the numeric keypad.
EXTRACTION COUNT	Indicates the number of runs performed since the Dionex ASE 350 was installed (read-only).

C.5 Error Log Screen

Use the **ERROR LOG** screen to review errors that occurred during operation.



Figure C-6. Error Log Screen

When an error occurs during a run, the Dionex ASE 350 displays an error message and records the error (including the cell and the collection vessel affected) in an error log. Unless the error causes a shutdown, the run continues. Any additional errors that occur during a series of runs will also be recorded in the error log. A maximum of 20 errors can be logged.

To view the list of errors, display the **ERROR LOG** screen (press **Menu, 6**, and then **5**). If there are too many errors to be shown on one screen, use the arrow up and down buttons to scroll through the list.

Errors remain on the screen until deleted; to delete them, press the button with the decimal point on the numeric keypad.

For an explanation of the error messages, as well as troubleshooting guidance, see [Section 4.1](#).

C.6 Detailed Status Screen

The **DETAILED STATUS** screen displays detailed information about the current Dionex ASE 350 status. All of the information shown on the **SYSTEM STATUS** screen (see [Figure 2-10](#)) is displayed here, as well as additional details about the status of certain valves and sensors. Only fields that do not appear on the **SYSTEM STATUS** screen are described below.



Figure C-7. Detailed Status Screen

Parameter	Description
PLATE T SET	Indicates the set point (programmed) temperature for the top plate heater.
PLATE T ACTUAL	Indicates the current temperature of the top plate heater.
SOLVENT	Indicates the selected solvent (A, B, or C).
STATIC	Indicates the position of the static valve.
PRIME	Indicates the position of the prime valve.
PURGE	Indicates the position of the purge valve.
RELIEF	Indicates the position of the pressure relief valve.

C.7 Hydrocarbon Status Screen

Use the **HYDROCARBON STATUS** screen to monitor the current hydrocarbon level at the Dionex ASE 350 installation site and to adjust the solvent vapor threshold, if necessary.

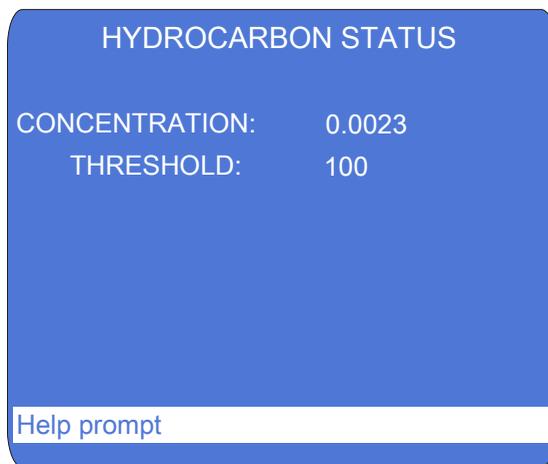


Figure C-8. Hydrocarbon Status Screen

Parameter	Description
CONCENTRATION	Indicates the hydrocarbon level detected by the hydrocarbon sensor (read-only).
THRESHOLD	Selects the solvent vapor threshold (1 to 500). If the threshold is often tripped, increase the selected value; if the threshold is never tripped, decrease the selected value.

D • Reordering Information

Part Number	Item	Quantity
<i>Sample Cells and Accessories</i>		
068095	Stainless steel sample cells, 1 mL (assembled)	Pkg. 6
068096	Stainless steel sample cells, 5 mL (assembled)	Pkg. 6
068097	Stainless steel sample cells, 10 mL (assembled)	Pkg. 6
068098	Stainless steel sample cells, 22 mL (assembled)	Pkg. 6
068099	Stainless steel sample cells, 34 mL (assembled)	Pkg. 6
068100	Stainless steel sample cells, 66 mL (assembled)	Pkg. 6
068101	Stainless steel sample cells, 100 mL (assembled)	Pkg. 6
068085	Stainless steel sample cell, 1 mL (assembled)	1
068086	Stainless steel sample cell, 5 mL (assembled)	1
068087	Stainless steel sample cell, 10 mL (assembled)	1
068088	Stainless steel sample cell, 22 mL (assembled)	1
068089	Stainless steel sample cell, 34 mL (assembled)	1
068090	Stainless steel sample cell, 66 mL (assembled)	1
068091	Stainless steel sample cell, 100 mL (assembled)	1
068261	Stainless steel sample cell body, 1 mL	1
068262	Stainless steel sample cell body, 5 mL	1
068263	Stainless steel sample cell body, 10 mL	1
068264	Stainless steel sample cell body, 22 mL	1
056646	Stainless steel sample cell body, 34 mL	1
056696	Stainless steel sample cell body, 66 mL	1
056693	Stainless steel sample cell body, 100 mL	1
056775	Stainless steel frits for sample cell end caps	Pkg. 50
068106	Stainless steel end caps for sample cells (includes frits and seals)	Pkg. 2
068104	Zirconium sample cells, 66 mL (assembled)	Pkg. 3
068105	Zirconium sample cells, 100 mL (assembled)	Pkg. 3
068102	Zirconium sample cell, 66 mL (assembled)	1
068103	Zirconium sample cell, 100 mL (assembled)	1
068265	Zirconium sample cell body, 66 mL	1

Dionex ASE 350 Solvent Extraction System

Part Number	Item	Quantity
068266	Zirconium sample cell body, 100 mL	1
068260	Zirconium frits for sample cell end caps	Pkg. 10
068107	Zirconium end caps for sample cells (includes frits and seals)	Pkg. 2
061687	PEEK seals for sample cell end cap	Pkg. 50
056778	Snap rings for sample cell end caps	Pkg. 10
056684	Snap ring tool	1
049457	External O-rings for sample cell end caps, PTFE	Pkg. 50
056325	External O-rings for sample cell end caps, Viton	Pkg. 50
049660	O-ring insertion tool	1
068093	Cellulose filters, 27 mm	Pkg. 100
056780	Cellulose filters, 30 mm	Pkg. 100
068092	Glass-fiber filters, 27 mm	Pkg. 100
056781	Glass-fiber filters, 30 mm	Pkg. 100
056929	Filter insertion tool (for 30-mm filters)	1
068076	Sample funnel (for 1-mL sample cells)	1
068075	Sample funnel (for 5-mL, 10-mL, and 22-mL sample cells)	1
056699	Sample funnel (for 34-mL, 66-mL, and 100-mL sample cells)	1
<i>Startup Kits</i>		
068254	Small Volume Startup Kit (for 1-mL, 5-mL, 10-mL, and 22-mL sample cells)	1
068255	34-mL Volume Startup Kit (for 34- mL sample cells only)	1
068256	Large Volume Startup Kit (for 66-mL and 100-mL sample cells)	1
<i>Solvent Reservoirs</i>		
045901	Bottle, plastic-coated glass, 2 liters	1
068077	Bottle cap assembly	1

Part Number	Item	Quantity
<i>Collection Vessels</i>		
056284	Collection bottles (clear), 250 mL (includes lids and septa)	Pkg. 12
048784	Collection vials (clear), 60 mL (includes lids and septa)	Pkg. 72
048781	Collection vials (amber), 60 mL (includes lids and septa)	Pkg. 72
055395	PTFE/silicone low-bleed septa for collection bottles Note: These are high-purity septa for trace analysis applications.	Pkg. 72
066176	Vial tray insert	1
<i>Waste Bottle</i>		
060078	Waste bottle septum assembly (punched)	1
<i>Tubing</i>		
049296	Nitrogen line: 4-mm (0.16-in) OD tubing	Per inch
053514	Vent line: 8-mm (0.31-in) OD tubing	Per inch
214739	Air line: 6-mm (0.25-in) OD tubing	Per foot
049492	Inlet filter assembly (Use only when air source may contain VOCs or oil)	1
055075	Drain hose	1
067402	Clamp (for drain hose)	1
<i>Pump</i>		
047755	Check valve cartridge, inlet	1
047664	Check valve assembly, inlet	1
057346	Check valve cartridge, outlet	1
059791	Check valve assembly, outlet	1
066109	Piston guide	1
066110	Pump piston	1
066162	Piston high-pressure seal	1
066163	Piston air seal	1

Dionex ASE 350 Solvent Extraction System

Part Number	Item	Quantity
<i>Static Valve</i>		
068115	Static Valve Repair Kit	1
068116	Static Valve Tool Kit	1
066165	Static valve seal	1
067381	Static valve piston	1
<i>Miscellaneous</i>		
954746	Fast-blow IEC127 fuse, 10 amps	1
960777	USB cable, 6 ft	1
046985	Flathead screwdriver	1
068955	Source needle assembly	1
068247	Heat exchanger	1
938914	Hydrocarbon sensor	1
062819	Dionex ASE Prep DE (pelletized diatomaceous earth)	1 kg
080024	Dionex ASE Prep CR Resin	500 g
068954	Dionex ASE 150/ASE 350 Preventive Maintenance Kit	1

E.1 Introduction

Scientists are continually striving to develop a means to accomplish desired results faster, better, cheaper, and more efficiently. However, the application of new technologies to sample preparation has often lagged behind advances in the determinative steps of the overall analytical scheme. Consider the major technological changes in chromatography that have occurred in the last few years. What a contrast exists between the first commercial gas chromatographs (GC) introduced in the late 1950s and the modern GCs we have today! On the other hand, analytical chemists can still be seen preparing samples for chromatographic analysis using the same type of Soxhlet apparatus that was used to prepare samples for the first GC instruments. In fact, Soxhlet apparatus has been in use, with few modifications, for over 100 years. Traditional extraction techniques suffer from several shortcomings. They often take long periods of time (several hours), use large quantities of solvent (hundreds of milliliters per sample), and require a great deal of labor and user intervention.

In addition to the costs associated with sample preparation, studies have shown that sample preparation is the primary source of errors in any analytical process. If we want to have the biggest impact on improving the performance of any analytical method, we would be well-advised to attack the sample preparation portion of the method. That is one reason that Thermo Fisher Scientific has been involved in research dealing with sample preparation for many years. Accelerated solvent extraction (ASE) was developed to address the problems inherent with traditional extraction techniques such as Soxhlet and sonication.

ASE is an automated extraction technique that uses elevated temperatures and pressures to achieve extractions in very short periods of time (for example, 10 g of sample can be extracted in less than 15 minutes, using less than 15 mL of solvent). Why would we do extractions at elevated temperatures and pressures? There are several physicochemical reasons for this. First, let's consider temperature.

It is a well-known fact that the use of higher temperatures increases the capacity of solvents to solubilize analytes. For example, the solubility of anthracene increases nearly 13-fold as we increase temperature from 50 to 150 °C. Increasing the temperature also leads to faster diffusion rates. This means that analytes move more quickly from the boundary layer near the surface of the matrix from which

they are extracted to the bulk solvent at higher temperatures. Higher temperatures also mean lower solvent viscosities, meaning that the solvent can penetrate the pores of the matrix more easily. Finally, increased temperature makes it easier to disrupt solute-matrix interactions (dipole attractions, van der Waals forces, hydrogen bonding, and so on) and remove analytes from the matrix. Taken all together, the net effect is that doing extractions at elevated temperatures means that the extractions happen much faster and use less solvent. However, temperature alone is not enough because many of the organic solvents used in extractions boil at relatively low temperatures. This is one limitation of techniques such as Soxhlet or automated Soxhlet; the highest temperature at which extractions take place in these techniques is the boiling point of the solvent.

If sufficient pressure is exerted on the solvent during the extractions, temperatures above the boiling point can be used. This means that all of the advantages of working at elevated temperature can be realized even with solvents with relatively low boiling points. Operating at elevated pressures also helps the overall extraction process to happen more quickly. Pumping solvent through a packed bed is easier at elevated pressures. Pressurized solvent is forced into the pores of the sample matrix, coming into more intimate contact with analytes in those areas. Hence, the combination of elevated temperatures and pressures allows extractions to occur rapidly and completely. For a more detailed discussion of the theoretical consideration of ASE, consult Richter et al., *Anal. Chem.* **1996**, *68*, 1033-1039.

E.2 Operation

ASE was first described in 1995 and 1996 (1–9). As a technique, it has grown steadily in use since that time. [Figure E-1](#) shows a schematic of the ASE process.

To perform an extraction, the solid sample is loaded into a sample cell (1 to 100 mL) and the end caps (each containing a frit) are tightened by hand onto the cells. The filled sample cells are loaded onto a cell tray and collection vessels (bottles or vials) are loaded onto a collection tray. A robotic arm transfers each cell separately into the oven for extraction. The oven is maintained at the selected operating temperature throughout the extractions (room temperature to 200 °C). The cell design and associated fluid apparatus allow operation of the extractions at elevated pressures (500 to 1500 psi) to maintain the solvents as liquids at temperatures above their boiling points. The temperature and pressure are controlled independently for each cell regardless of the solvent used, the moisture or mineral content of the sample, or any characteristic of the matrix that might affect the actual extraction temperature. This is an advantage when compared to

microwave extraction, in which the actual pressure and temperature of the extraction are influenced strongly by the above mentioned sample parameters.

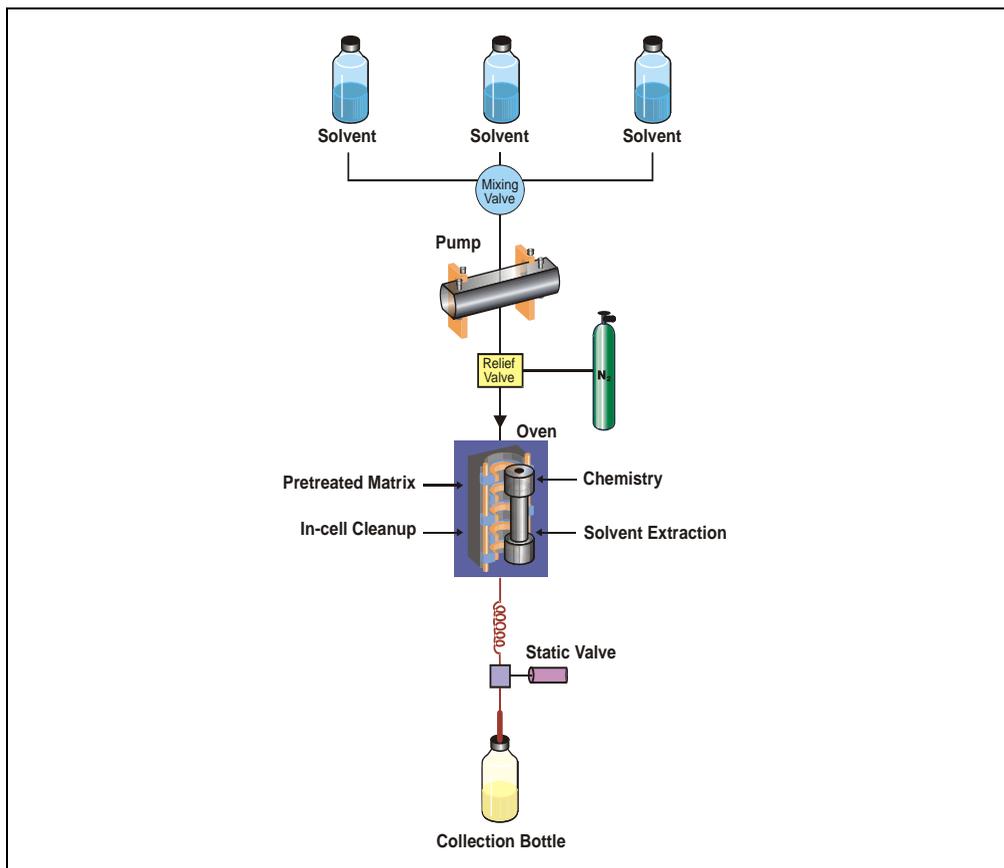


Figure E-1. Schematic of the ASE Process

When the cell is in place in the oven, the pump immediately begins to deliver the solvent of choice to the sample cell. Single solvents or premixed solvents can be used from a single collection vessel; alternatively, any combination of up to three different solvents can be programmed. Once solvent has made its way through the sample cell and reaches the collection vessel, the static valve closes to allow pressurization of the cell. Since the solvent expands as it heats, the pressure in the cell will increase when the static valve closes. When the pressure reaches 200 psi above the set point, the static valve opens rapidly to relieve the pressure and then closes again. The pump also delivers fresh solvent to the cell in an effort to return

the pressure to the set point value. This addition of fresh solvent during ASE is analogous to fresh solvent dripping down from the condenser onto the extraction thimble during Soxhlet extraction.

During the first phase of a run (called the heat-up time), the cell contents are heated by the oven to the selected operating temperature. Heat-up times vary between 5 minutes for 100 °C and 9 minutes for 200 °C. After the heat-up time, the extraction enters a static period with a duration selected by the user. Typical static times are 5 minutes, but can vary from 0 to 2000 minutes. After the static time, fresh solvent is pumped through the cell to remove extracted analytes while the sample and solvent are still hot. The amount used for the rinse can vary from 0% to 150% of the volume of the cell used for the extraction (40% to 60% is most common).

The user can select the number of times the sample will be in the static mode, and enter this as the number of static cycles. The rinse volume is divided by the number of static cycles, so that fresh solvent is present at the beginning of each static cycle. In other words, if one static cycle is selected, the entire rinse volume will be pumped through the cell at the end of the static time. If three static cycles are selected, one-third of the total rinse volume will be pumped through the cell at the end of each static cycle. From one to nine static cycles can be programmed; one cycle is the most common.

Following the final solvent rinse, solvent is purged out of the cell (using nitrogen at 150 psi) for a predetermined period of time. The total time for the extraction is usually less than 15 minutes and the amount of solvent used is approximately 1.5 times the volume of the sample cell (for example, about 15 mL for a 10-mL cell).

The extracts are delivered to the collection vessels through a filter, and in many cases do not need any additional preparation prior to analysis. Since the extract is diluted by the total volume of extraction solvent plus the rinse solvent, a further concentration step (for example, evaporation or solid-phase extraction) may be required when performing trace analysis.

Upon completion of the purge step, the cell is returned to the tray and the next sample is taken to the oven to begin the extraction process again. Current ASE instrumentation allows for unattended overnight operation of the extraction system without user intervention.

Under method control, each sample is extracted using the same conditions. When using sequence control, each cell can be extracted using different conditions, including different solvents (if multiple solvent reservoirs are installed).

ASE instrumentation includes many features designed to minimize safety issues with the use of solvents at elevated temperatures and pressures. Among the safety measures in place are: flammable vapor sensors, liquid leak detectors, checks for collection vessel overfill conditions, three levels of overpressurization prevention (electronic and mechanical), solvent flow monitoring, and pneumatic source pressure monitoring.

E.3 Method Optimization

Method optimization consists of two main elements: sample preparation and extraction parameters.

E.3.1 Sample Preparation

Sample preparation is an essential part of every solvent-based extraction procedure. While many sample types can be efficiently extracted without any pretreatment, other samples require some manipulation for an efficient extraction to occur. In general, the same sample preparation that is done prior to Soxhlet or sonication extraction should be done prior to extraction by ASE. This section discusses three sample preparation techniques: grinding, dispersing, and drying.

Grinding

For an efficient extraction to occur, the solvent must make contact with the target analytes. The more surface area that can be exposed in a sample, the faster an extraction will occur. Samples with large particle sizes should be ground prior to extraction. Efficient extraction requires a minimum particle size, generally smaller than 1 mm. Grinding can be accomplished with a conventional mortar and pestle or with electric grinders and mills. Often a large, representative sample can be ground and weighed portions of the ground sample can be used for extraction. Soil and sediment samples generally do not need to be ground, although it may be necessary to remove stones or sticks from the samples prior to extraction. Polymer samples must be in a ground state for an efficient extraction of additive compounds. Materials such as polymers and rubbers are best ground at reduced temperatures (for example, while chilling with liquid nitrogen). Animal or plant tissue samples can be homogenized using any procedure, including a blender or tissue homogenizer.

The effects of sample surface area on extraction efficiency have been studied. Three samples of mozzarella cheese were prepared differently and extracted for fat content using ASE. The chopped samples were cut into pieces ranging in size between 2 and 5 mm. The ground samples were ground with diatomaceous earth (1:2). Each sample was extracted with a mixture of hexane:2-propanol (3:2) using these ASE conditions: 1000 psi, 125 °C, 6-minute heat-up, three 10-minute static cycles, 100% rinse, 60-second purge, and one 11-mL cell containing one cellulose filter. The extracts were analyzed gravimetrically. The ASE results were compared to results obtained from Mojonnier extraction (see [Figure E-2](#)). These results demonstrate that increasing the surface area by grinding the sample results in higher extraction efficiencies.

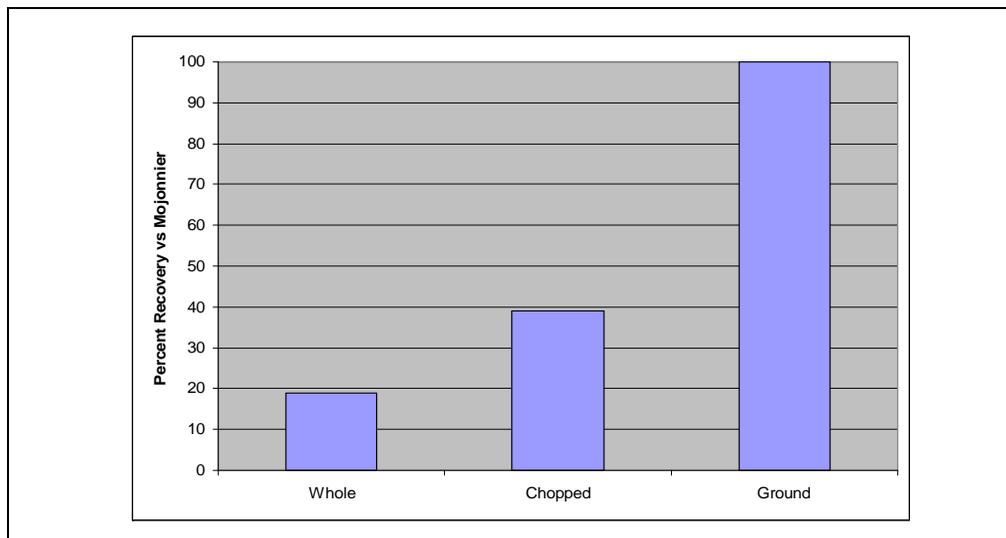


Figure E-2. Effect of Sample Particle Size for ASE vs. Mojonnier Extraction. Whole cheese (0.5 to 1.5 cm size); chopped (2 to 5 mm); ground (mixed thoroughly with diatomaceous earth)

Dispersing

The aggregation of sample particles may prevent efficient extraction. In these cases, dispersing the sample with an inert material such as sand or diatomaceous earth will assist in the extraction process. Dispersing is also recommended with samples that tend to compact in the sample cell outlet. These samples normally contain very small particles that can adhere

tightly to each other when under pressure. Some samples may compact in the sample cell due to the high pressures used in ASE. Mixing the sample with sand or diatomaceous earth will prevent this compacting of the sample that can lead to occlusion of the sample cell.

When extracting soil or sediment samples, it is important to mix the samples with a dispersing agent unless the samples are completely dry. If the soil or sediment samples are wet or high in clay, the samples can be mixed with either sand or diatomaceous earth, but the latter is preferred because it will adsorb some of the water and make the sample easier to handle.

It is important to periodically run blank extractions of the dispersing agent to verify its cleanliness.

Drying

Samples do not have to be completely dry to achieve efficient extractions with ASE. This depends on the analytes and the solvent used for extraction. High levels of water can prevent nonpolar organic solvents from reaching the target analytes. The use of more polar solvents (for example, acetone and methanol) or solvent mixtures (for example, hexane/acetone and methylene chloride/acetone) can assist in the extraction of wet samples. Elevated temperatures can also improve the extraction efficiency of wet samples (10). In addition, the solvent being used will impact the type of drying that needs to be done to a sample and whether it needs to be done at all. With nonpolar solvents, the need for drying is more important. One can also use a bridge solvent such as acetone (for example, hexane/acetone, 1:1) mixed with a nonpolar solvent to improve the extraction efficiency of wet samples.

Sample drying prior to extraction is an efficient way to handle wet samples. Drying can be accomplished by direct addition of a drying agent such as diatomaceous earth. The use of magnesium sulfate is not recommended with ASE due to the extremely hard, concrete-like material that can be produced. Sodium sulfate should be used **only** with nonpolar solvents (hexane, heptane, toluene, and so on). Sodium sulfate can become soluble in the extraction process and then be deposited in the exit lines. Oven drying and freeze drying are other viable alternatives for sample drying prior to extraction; however, these procedures may compromise the recovery of volatile compounds.

The sample drying or dispersing agent should be mixed thoroughly with the sample in a small vial, beaker, or mortar, and then added to the sample cell. For quantitative transfer, the mixing vessel can be rinsed with 1 to 2 mL of the extraction solvent using a Pasteur pipette, and this volume added directly to the sample cell. The main purpose of mixing with a drying agent like diatomaceous earth is not to remove all water from the sample but to make it easy to transfer the sample into the sample cell. If elevated temperatures are used, then some water will be removed from the sample during the extraction process. In this case, sodium sulfate can be added to the extract after collection to remove water.

E.3.2 Extraction Parameters

Solvent

For an efficient extraction, the solvent must be able to solubilize the target analytes while leaving the sample matrix intact. The polarity of the extraction solvent should closely match that of the target compounds. The common adage of “like dissolves like” is very applicable in ASE. Mixing solvents of differing polarities can be used to extract a broad range of compound classes. Generally, if a particular solvent has been shown to work well in a conventional procedure, it will also work well in ASE. Compatibility with the post-extraction analytical technique, the need for extract concentration (solvent volatility), and the cost of the solvent should all be considered. While many ASE methods recommend solvents or solvent mixtures for specific analyte classes, there may be alternatives that better fit the needs of a particular laboratory. For example, Schantz (11) showed that dichloromethane/acetone or acetonitrile can be used to get complete extraction of polycyclic aromatic compounds. If GC or GC-MS is the analytical method, then dichloromethane/acetone would be the solvent of choice. If HPLC is the determinative step, then acetonitrile would be the solvent of choice. Solvent choice will also determine the level of coextractables along with the analytes. Generally, the more polar the solvent or solvent mixture, the less selective it will be.

Solvents that exhibit marginal results at ambient conditions may perform adequately under ASE conditions. Most liquid solvents, including water and buffered aqueous mixtures, can be used in ASE. Strong acids (HCl, HNO₃, H₂SO₄) are not recommended for use with stainless steel cells because they may damage the cells. However, small amounts of these

acids can be used with zirconium cells. When required, weak acids such as acetic or phosphoric can be used. These should be added to aqueous or polar solvents in the 1-10% (v/v) range.

For extraction of polymer samples, select a solvent that will extract the additives but not the matrix itself. For example, isopropanol with small levels of cyclohexane has been shown to work well for the extraction of phenolic antioxidant additives in polymers.

Temperature

Temperature is the most important parameter used in ASE extraction. As the temperature is increased, the viscosity of the solvent is reduced, thereby increasing its ability to wet the matrix and solubilize the target analytes. The added thermal energy also assists in breaking analyte-matrix bonds and encourages analyte diffusion from the matrix surface.

When developing a new method, start at 100 °C or, if the target analytes have a known thermal degradation point, start at 20 °C below this level. Most ASE applications operate in the 75 to 125 °C range, with 100 °C the most common temperature for environmental applications. If the sample has a tendency to melt in the sample cell, a cellulose thimble can be used to facilitate extraction and sample removal.

[Table E-1](#) shows an example of the effect of temperature for the extraction of petroleum hydrocarbons (TPH) from soil. Note that not only does the analyte recovery increase, but the reproducibility improves as a function of temperature.

Temperature	Recovery	RSD (%)
27 °C	81.2	6.0
50 °C	93.2	5.0
75 °C	99.2	2.0
100 °C	102.7	1.0

Samples were analyzed by IR, with n = 5.

Table E-1. Effect of Temperature on TPH; Extraction from Soil

There appears to be confusion about the purpose of the preheat function available on ASE systems. When the preheat function is used, a cell is placed in the oven and heated without any solvent being pumped into the

cell. This function was originally developed to allow pre-extraction derivatization of analytes or the drying of samples by blowing nitrogen through the cell while it is heated. The preheat function should **not** be used in normal operation of ASE. In general, this should be set to zero minutes. If used, recoveries of more volatile compounds (for example, organochlorine pesticides) will be greatly reduced.

Pressure

The effect of pressure is to maintain the solvents as liquids while above their atmospheric boiling points, and to rapidly move the fluids through the system. Because the pressures used in ASE are well above the thresholds required to maintain the solvents in their liquid states, pressure adjustments for changing solvents are not required. Changing the pressure will have very little impact on analyte recovery, and it is not considered a critical experimental parameter. Dionex ASE 350 extractions are performed at 1500 psi.

Static Cycles

The use of static cycles was developed to introduce fresh solvent during the extraction process, which helps to maintain a favorable extraction equilibrium. This effectively approximates dynamic extraction conditions without the need for troublesome flow restrictors to maintain pressure. When more than one cycle is used in a method, the rinse volume is divided by that number. When the first static time is complete, the divided portion of the rinse volume is delivered to the cell, with the “used” solvent directed to the collection vessel. The system then holds the sample and solvent for a second static period. The nitrogen purge step is initiated only after the final static cycle. Because the original rinse volume has only been divided, no additional solvent is used for the extraction.

Static cycles have proven to be useful for sample types with a very high concentration of analyte, as well as samples with difficult to penetrate matrices. The static time can be adjusted to minimize the total extraction time. For example, three 3-minute static cycles can be used in place of one 10-minute static step. When low temperature extractions are desired (< 75 °C), multiple static cycles should be used to compensate for the smaller amount of solvent introduced during the heat-up step, as the static valve pulses to regulate the pressure.

Time

Certain sample matrices can retain analytes within pores or other structures. Increasing the static time at elevated temperatures can allow these compounds to diffuse into the extraction solvent. The effect of static time should always be explored in conjunction with static cycles, in order to produce a complete extraction in the most efficient way possible. Generally, ASE extractions are completed in less than 20 minutes; if times longer than this are necessary to get complete extraction, then a higher temperature, different solvent, or multiple static cycles should be explored to reduce the overall extraction time.

E.4 Method Development

A representative sample should be prepared as outlined in the discussion on sample preparation (see [Section E.3.1](#)). Select a sample cell size that most closely matches the sample size. The sample cells do not need to be filled completely; however, a full cell will use less solvent in the extraction process than a partially filled one. A recommended set of conditions to begin method development are as follows: 1500 psi, 100 °C (or 20 °C below analyte decomposition temperature), 5-minute static time, 40% rinse volume, 60-second purge (up to 300 seconds for a 100-mL cell), and one static cycle. If desired, a sequence can be set up to extract each of a series of samples under a different set of conditions and collect them in separate collection vessels. Samples can also be extracted multiple times to determine extraction completeness. If analyte is present in the second or third vessel, then the initial conditions need to be changed.

The order of change would be (1) change the solvent, (2) increase the temperature, (3) add a second or third cycle, and (4) increase the time. In this way, one can quickly determine the optimized extraction conditions for a particular analyte and matrix.

E.5 Selectivity in ASE

Selectivity in extraction is defined here as being able to extract compounds of interest with little or no interfering coextracted compounds. ASE is generally considered to be an exhaustive extraction technique, and often the extracts obtained from complex samples contain compounds that can interfere with the determination of the desired analytes. Selectivity in ASE could come from the manipulation of the extraction conditions to minimize coextractables while maximizing analyte recovery. There are three basic procedures for obtaining selective extraction in ASE or, in other words, generating extracts that contain the compounds of interest and few, if any, interfering compounds. The three techniques are choice of temperature, choice of solvent, and use of adsorbents in the sample cell. Of course, the most powerful method is to use variations of all three to fine-tune the selectivity during the ASE process.

The choice of temperature alone can affect selectivity. The higher the temperature for the extraction, the less selective the results. Lowering the temperature will make ASE more selective, but the recovery of analytes can diminish unless the time is increased. Similarly, selectivity is decreased when using more polar solvents. However, one can use a series of solvents of increasing polarity to obtain selective extractions or “fractionation.”

Dark-colored fruits such as blueberries are being studied for their antioxidant content. These compounds are polyphenolic in nature and require polar solvents for extraction. However, if the fruit sample is extracted with a polar solvent like methanol, acetonitrile, ethanol, or water, the resulting extract contains many compounds that make the analysis for antioxidant compounds more challenging. We have found that the samples can first be extracted with nonpolar solvents such as hexane or DCM to remove unwanted wax compounds. Then, by extracting the same sample with solvents of increasing polarity and collecting the fractions in separate vessels, one can obtain extracts that are easier to analyze.

[Figure E-3](#) is a photo of extracts obtained from a single sample of wild blueberries extracted with hexane, followed by DCM, ethyl acetate, acetonitrile, and then ethanol. Clearly, using a fractionation procedure like this can offer advantages when analyzing extracts of plant materials that can contain several hundred compounds of interest.

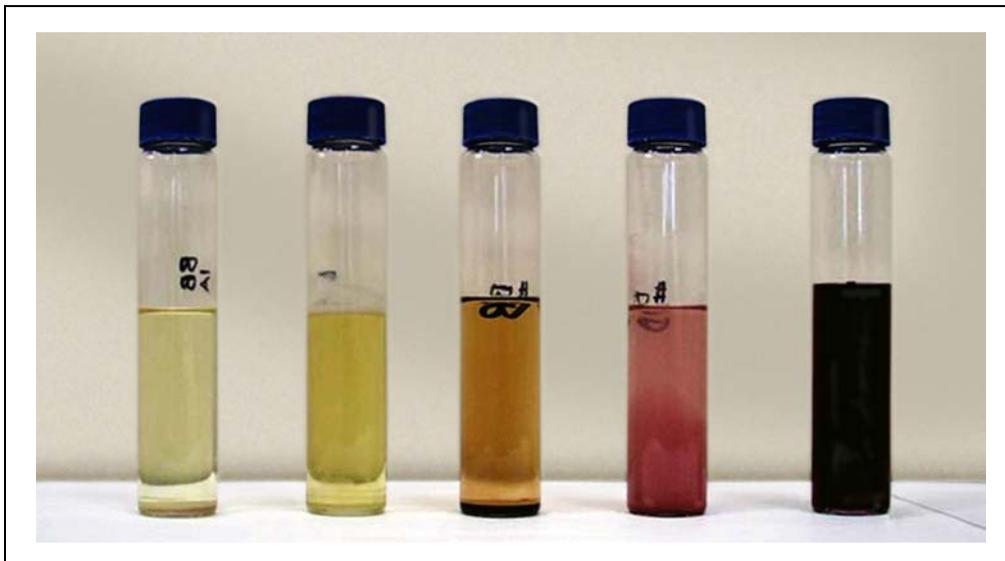


Figure E-3. Selectivity in ASE. The same sample of blueberries was extracted with (from left to right) hexane, DCM, ethyl acetate, acetonitrile, and ethanol

Another unique use of solvent selectivity was reported by Draisci et al. (12). The authors reported the use of ASE for the extraction of corticosteroids from beef liver prior to analytical determination by LC-MS. First, the sample was extracted with hexane to remove the majority of lipids that would interfere in the determinative step. Next, the samples were extracted with hexane/ethyl acetate to remove the steroid compounds. Solvent fractionation using ASE is an area that needs to be explored to more fully understand its potential.

The use of sorbents in the sample cell along with the sample has offered some of the highest level of selectivity in ASE. Typically, the adsorbent is loaded into the sample cell first (outlet end) and the sample is loaded on top of the adsorbent. This way, the flow of solvent during the extraction is such that unwanted compounds are retained in the cell by the adsorbent. Azalea et al. (13) first reported the use of alumina in sample cells to retain lipids when extracting PCBs from fish tissue using ASE. They reported that, as the temperature of the extraction goes up or as the polarity of the solvent increases, the capacity of the alumina to retain lipids decreases. For example, if hexane or heptane is the extraction solvent, heat-activated alumina will retain about 70 mg of lipid per gram of alumina. If DCM is used, then the capacity is only about 35 mg lipid/g of alumina. When the correct

amount of sorbent is used, extracts can be produced using ASE that require no additional post-extraction treatment other than volume adjustment.

The most common use of adsorbents in ASE has been for the extraction and determination of nonpolar compounds such as PCBs, OCPs, PCDDs, and PCDFs in high lipid content matrices such as food or animal tissue. Other adsorbents that have been used include copper, C18 resin, Florisil, silica gel, acid-impregnated silica gel, and ion-exchange resins. [Table E-2](#) shows a summary, with related references.

Adsorbent	Uses	References
Silica	Removes nonpolar lipids	4
Florisil	Removes nonpolar lipids	15, 16
Alumina	Removes nonpolar lipids and colored compounds	13, 17, 18
C18 resin	Removes nonpolar lipids	19, 20
Ion-exchange resins	Removes ionic interferences	22
Copper powder	Removes sulfur	11, 23
Carbon	Assists with purification of PCDDs, PCDFs, and coplanar PCBs	24, 25

Table E-2. List of Adsorbents Used in ASE

There are a few interesting things to note from this table. Acid-impregnated silica gel has the highest capacity for lipid retention of all the adsorbents that were reported: It has roughly double the capacity of alumina. The sulfuric acid is typically present on the silica at 40% (wt/wt). Copper powder (cleaned with HCl first) can be used to retain sulfur when extracting sediment samples.

Gentili and colleagues (21) reported an interesting use of a C18 adsorbent. In this case, they were extracting polar antibiotics (sulfonamides) from animal tissues. They put C18 material in the outlet of the cell to retain some of the lipids that extracted with the analytes. Then, the samples were placed in a freezer to allow them to separate and harden. The samples were centrifuged and aliquots were removed and analyzed. This is a case in which the analytes were polar, the solvent was polar (water), and the matrices (baby food and meat) were polar. This is in contrast to several publications in which the analytes were relatively nonpolar (PCBs, PCDDs, OCPs, and so on) and the solvents were nonpolar (such as

hexane). In this case, the adsorbents have higher capacity than when the extraction solvent is polar.

Ion-exchange resins can be used to remove unwanted ionic species (22). For example, Thermo Fisher Scientific has been involved in projects to determine perchlorate in soils and vegetation samples. Water at 80 °C was used as the extraction solvent. Alumina was used to remove colored compounds such as chlorophyll. Ion-exchange resins were placed in the ASE cells to remove chloride and sulfate. This allowed the determination of perchlorate at the sub-ppb levels.

Björklund and his coworkers have published many articles on the use of adsorbents in-line in ASE cells to improve selectivity (25–31), especially for PCB and PCDD analysis. One article discusses the results of the development of a scheme that allows the separation of non-ortho PCBs and PCDD/Fs from the bulk of the PCBs, gravimetric fat determinations, and minimum post-extraction clean-up prior to analysis (25). This offers an obvious savings in time, labor, and solvent costs over traditional extraction procedures followed by clean-up methods such as GPC.

ASE has been shown to work well for the extraction of acrylamide from many food matrices (32). The use of adsorbents in the ASE cell has now been extended to this application (33). Florisil was added to the cell when extracting coffee or chocolate samples. The authors report that 6 g of Florisil in the cell, along with 2 g of sample, produced clear extracts without any interferences. [Figure E-4](#) shows the effect of varying the level of the Florisil.

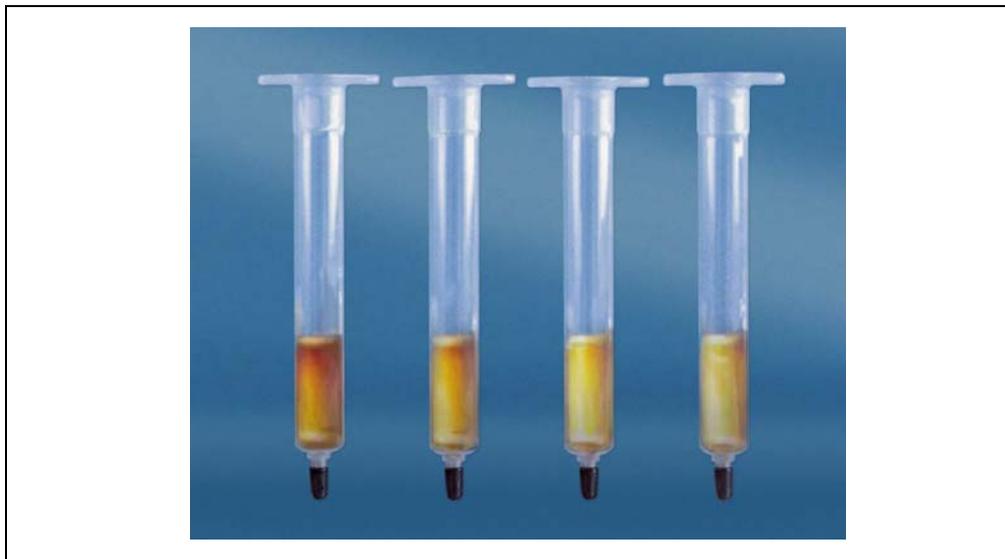


Figure E-4. Residual co-extractives after the ASE extraction of a coffee sample trapped on an SPE cartridge, the ASE cells containing varying from 0 to 6 g Florisil. From left to right: 1) No Florisil in the ASE cell, 2) 2 g Florisil in the ASE cell, 3) 4 g Florisil in the ASE cell, and 4) 6 g Florisil in the ASE cell

One of the most fascinating uses of selective extraction with ASE was reported by Poerschmann et al. (34, 35). In this work, a combination of adsorbents in the cells, varying temperature, and solvent allowed fractionation to be achieved that was superior to conventional SPE after exhaustive extraction. This work demonstrated the fractionation of lipid classes using sequential extractions.

Unmodified silica and cyanopropyl silica were used as the adsorbents in the outlet of the cells. The biological samples were extracted with hexane/acetone (9:1, v/v) at 50 °C to remove the neutral lipids. Then, the same samples were extracted with chloroform/methanol (1:4, v/v) at 110 °C to remove polar lipids such as phospholipids and hydroxy-containing fatty acids.

What is intriguing about this work is that the authors demonstrated that this fractionation scheme could be used to screen for diagnostic central nervous system (CNS) lipid markers in meat products. This is of particular interest for risk assessment studies for bovine spongiform encephalopathy (BSE) and food labeling legislation. The ASE fractionation scheme worked better than the widely used exhaustive lipid extraction procedure followed by SPE with regard to lipid

recoveries and clean fractionation of the lipid classes. Clearly, the combination of temperature, solvent, and adsorbent materials in the ASE cell can provide unique selectivity and capability and provide additional advantages over other sample preparation techniques.

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