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ED40 ELECTROCHEMICAL DETECTOR OPERATOR'S MANUAL

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1 • Introduction

1.1 Overview

The ED40 Electrochemical Detector measures current resulting from the application of potential (voltage) across electrodes in flow-through cells. Depending on the method by which the potential is applied and the current measured, several different properties of the flowing solution can be determined. These measurements help answer the major questions analytical chemists ask: What's in it, and how much is there?

Of course, other detectors are used in high performance liquid chromatography (HPLC). The UV-visible absorbance detector dominates all other forms of detection. Sensitivity is excellent for many analytes, especially aromatic species, and transparent mobile phases are readily available. However, there are numerous analytes that have very poor absorbance and are not detected with sufficient sensitivity by UV absorbance. Most of these nonchromophoric molecules are aliphatic organic molecules and inorganic ions. Low wavelength UV detection can be used, but at a loss in selectivity. Refractive index detection can also be used. However, maintaining a stable baseline can be difficult, and RI detection is less sensitive and substantially less selective than UV detection.

Fortunately, a wide variety of nonchromophoric molecules can be detected with good or excellent sensitivity by one of several forms of electrochemical detection. These molecules include carboxylic, sulfonic and phosphonic acids; alcohols, glycols, aldehydes, and carbohydrates; primary, secondary, tertiary, and quaternary amines; sulfates, sulfoxides, thiols, sulfides, and mercaptans; and inorganic anions and cations. In addition, when compared to UV absorbance detection, electrochemical detection provides substantial improvements in sensitivity and selectivity for amine and hydroxy-substituted aromatics such as catecholamines.

Several forms of electrochemical detection have become popular for certain HPLC applications. Conductivity is the workhorse detection method in ion chromatography, just as UV detection is for HPLC. DC amperometry is the preferred method for neurochemical analyses. Pulsed amperometry is now established as the superior detection method for carbohydrates. For most of the numerous analytes listed in the previous paragraph, detection by UV-visible absorbance is poor, while one of the three main techniques of electrochemical detection provided by the ED40 provides superior sensitivity and selectivity.

Electrochemical detection is not a substitute for UV-visible absorbance detection, but is an important complement. A liquid chromatograph equipped with both a Dionex AD20 Absorbance Detector and an ED40 Electrochemical Detector is a versatile and powerful analytical instrument.

1.2 Modes of Detection

The ED40 provides the three major forms of electrochemical detection: Conductivity, DC Amperometry, and Integrated Amperometry. Pulsed Amperometry is a form of Integrated Amperometry.

- *Conductivity detection* is based on the measurement of the magnitude of electrical current carried by dissolved ions in an electric field.
- *DC Amperometric detection* is based on the measurement of current resulting from oxidation or reduction (electrolysis) of analyte molecules at the surface of an electrode.
- *Integrated and Pulsed Amperometric detection* are similar to DC Amperometry in that molecules are oxidized or reduced at the surface of an electrode. However, current is measured by integration during a portion of a repeating potential vs. time waveform.

In addition, the *Voltammetry* mode is used to determine potentials used in DC and Integrated Amperometry.

1.3 About This Manual

Chapter 1, **Introduction**, provides a brief overview of the ED40 Electrochemical Detector. It also explains the product safety messages that appear throughout the manual.

Chapter 2, **Description**, describes the physical aspects of the ED40, including front panel controls, electronics, and cells. This is followed by a description of the ED40 operating modes.

Chapter 3, **Operation and Maintenance**, discusses operating features and methods (how to create, edit, and run them). Routine preventive maintenance requirements appear at the end of this chapter.

Chapter 4, **Troubleshooting**, lists possible causes of problems, along with step-by-step instructions to isolate and eliminate their sources.

Chapter 5, **Service**, presents step-by-step instructions for routine service and parts replacement procedures.

Chapter 6, **Electrochemical Detection**, describes electrochemical detection and its applications.

Appendix A, **Specifications**, lists the ED40 specifications and facility requirements.

Appendix B, **Installation**, describes the installation and interface necessary to place the ED40 into operation. Also included are setup procedures for operating the ED40 as a stand-alone module or connected to other Dionex products.

Appendix C, **User Interface**, describes the front panel display and controls in detail and includes illustrations of all menus and screens used for ED40 operation and diagnostics.

Appendix D, **Signal Processor Functions**, lists the functions of the Signal Processor (SP) card.

Appendix E, **Connector Pinouts**, describes the pinouts for all ED40 connectors.

Appendix F, **Further Reading**, is a bibliography for electrochemical detection.

1.3.1 Typefaces

Typefaces are used in this manual as follows.

- Capitalized bold Helvetica indicates a front panel button:

  Press **Enter** to begin running the method.

- Upper-case bold Helvetica indicates the name of a screen, the name of a menu, or an on-screen entry:

  Go to the **METHOD** screen.

  Move the cursor to the **EDIT** field.

1.3.2 Safety Messages and Notes

This instrument has been designed to comply with the requirements for safety set forth in IEC 1010 *Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use.*

This manual contains warnings and precautionary statements that can prevent personal injury and/or damage to the instrument when properly followed. Safety messages appear in bold type and are accompanied by icons.



Indicates a potential hazard which could result in serious injury or loss of life. Any hazard of this type will be located behind a barrier and will be accessible only by use of a tool. Access may be required during installation, maintenance, or service.



Indicates a potential hazard to the operator, or damage to the instrument or other property.



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

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 you to an unexpected result of an action, suggest how to optimize the performance of the instrument, etc.

1.3.3 Symbols

The symbols below appear on the detector, or on detector labels.

 Alternating current

 Protective conductor terminal

 Power supply is on

 Power supply is off

1.4 Related Manuals

During installation and operation of the ED40, you may need to refer to one or more of the following manuals (depending on your system) for information about other modules and components included in a DX 500 system.

The following manuals are provided with their respective products:

LC10 Chromatography Organizer Operator's Manual (Document No. 034858)

LC20 Chromatography Enclosure Operator's Manual (Document No. 034859)

LC30 Chromatography Oven Operator's Manual (Document No. 034860)

GP40 Gradient Pump Operator's Manual (Document No. 034856)

IP20 Isocratic Pump Operator's Manual (Document No. 034857)

The following manual is provided in the ED40 Ship Kit:

Installation of Dionex Ferrule Fittings (Document No. 034213)

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2 • Description

The ED40 Electrochemical Detector is housed in a single-unit DX 500 enclosure (see Figure 2-1). The detector is designed to be stacked on top of other DX 500 units, up to a maximum height of four units. Figure B-1 in Appendix B illustrates the recommended stacking configuration for DX 500 systems.

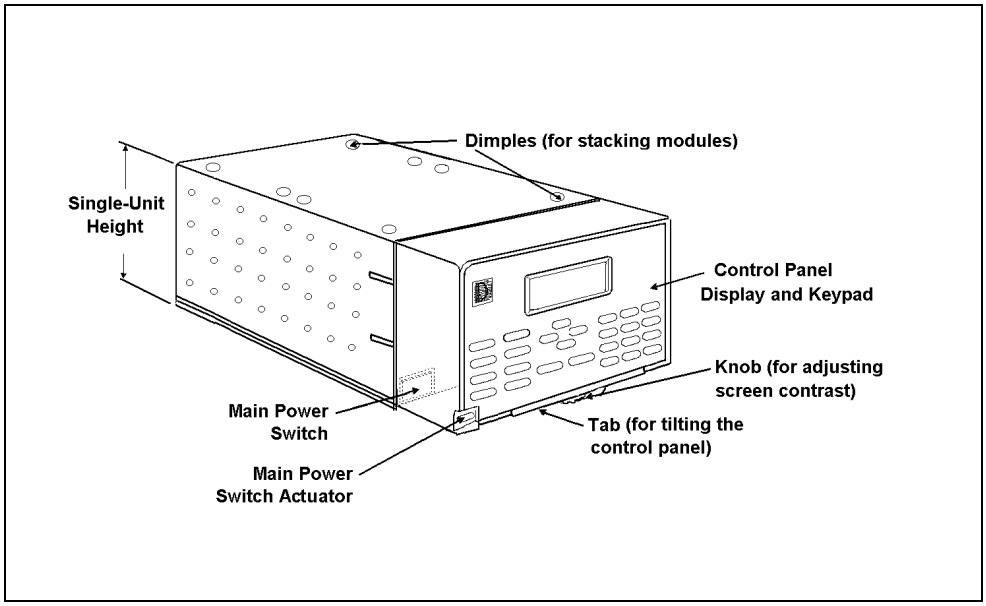


Figure 2-1. ED40 Enclosure

2.1 Front Control Panel

The control panel on the front door of the ED40 contains the liquid crystal display (LCD), the membrane keypad, and the actuator for the main power switch. The door opens to provide access to the detector electronics, which are described in Section 2.3.

Screen Contrast

Information is displayed on the LCD, also called the *screen*, in white letters on a blue background. To adjust the screen contrast, use the knurled knob in the recess below the **Help** and **Menu** buttons (see Figure 2-2).

Tilt Panel

To maximize visibility, the front control panel can be tilted to four different positions.

- To tilt the panel, support the door at the left side (to prevent it from opening) and lift firmly on the tab in the recess below the **Help** and **Menu** buttons (see Figure 2-2).
- Push on the tab to return the panel to its vertical position.

Power Switches

The main power switch is on the bulkhead behind the front door. An actuator for the main power switch is on the outside of the door, at the lower left corner (see Figure 2-2). The actuator functions only when the door is fully closed. When the door is open, press the main power switch on the bulkhead to turn the ED40 on and off.

2.1.1 Control Panel Keypad

The front panel keypad (Figure 2-2) is used to control detector operation directly, as well as to create and modify programmed series of timed events, called *methods*.

- Pressing **Menu** displays a list of screens available for display.
- Only the fields shown in reverse video on a screen can be edited—the other fields are informational displays.
- Use the four directional arrow buttons to move the cursor to an editing field. Use the numerical buttons to enter a variable value. Use the **Select** Δ and **Select** ∇ buttons to choose from predetermined options. Pressing a **Select** button increases (or decreases) a numerical value by one, while holding down a **Select** button increases (or decreases) the numerical value continuously.
- Pressing **Enter** executes the selected value.

A beep sounds each time you press a front panel button. When an error occurs, this beep is lower in frequency. The beeps can be disabled from the **MODULE SETUP** screen.

Offset

Returns the analog (recorder) output to a predetermined baseline and zeros the display. The resultant value of the offset required is displayed on the **DETAIL** screen. This function can be programmed in a method.

Mark

Sends a 10% positive event mark to the analog (recorder) output. (A mark is typically used to indicate a sample injection.) This function can be programmed in a method.

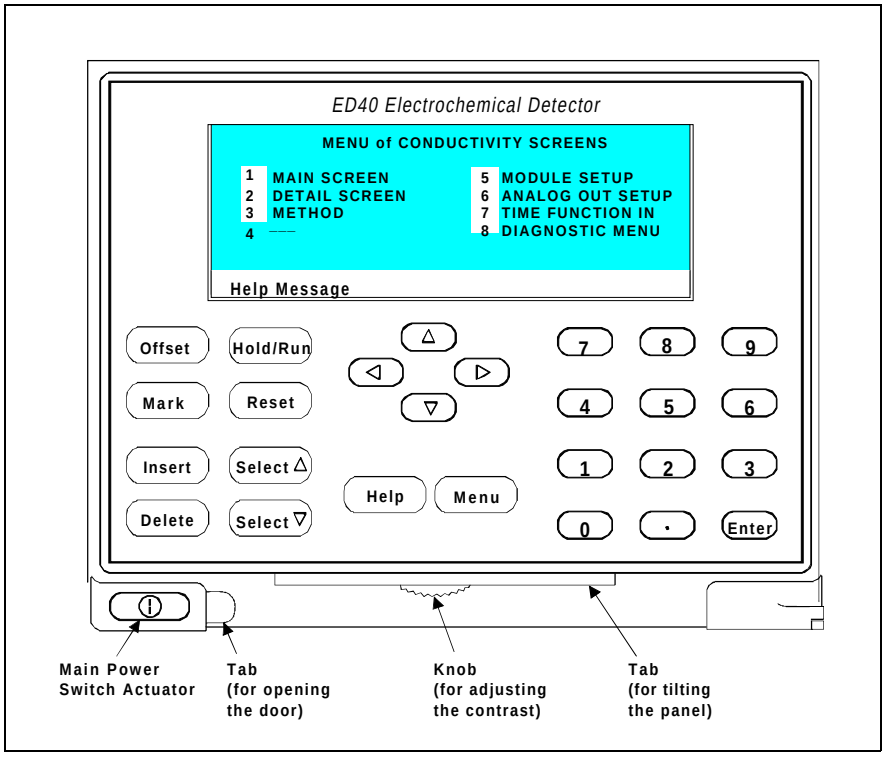


Figure 2-2. ED40 Front Panel

Insert

Inserts a new step into a method or waveform. Move the cursor to the **TIME** field and press **Insert** to add a new step after the cursor position. The parameter fields in the new step will be blank. Enter a time value for the new step and press **Enter**. If you move the cursor to a different field before entering the time, the new step is incomplete and will disappear.

Insert steps in any order; after you press **Enter**, they will be automatically organized in the correct chronological order.

Delete

Removes the value from the current entry field, allowing entry of a new value. To restore the previous value, move the cursor from the field without entering a new value.

On the **METHOD** and **WAVEFORM** screens, pressing **Delete** when the cursor is in a step entry field "blanks" the step parameter value. When the cursor is moved from the field, the field remains blank (that is, the previous value is not restored as it would have been on other screens). Blank step fields indicate that there is no change from the previous step.

To use **Delete** to delete an entire method step:

1. Position the cursor in the method's time field and press **Delete**. The time is removed and the help line displays this message:

```
TO DELETE THIS STEP, PRESS DELETE AGAIN
```

2. Press **Delete** again to delete the step. If you do not want to delete the step, press any button except **Delete** or the original time and step parameters will be restored.

Hold/Run

Turns the method clock off (**Hold**) and on **(Run)**. This button functions only when the detector is under Method control (see Section 2.6.2).

When the method clock is in Hold, pressing **Hold/Run** starts the clock. The clock starts either at the initial step of a new method or, if resuming an interrupted method, at the time at which the clock was put in Hold.

When the method clock is in Run, pressing **Hold/Run** stops the method clock, thereby "holding" the method and freezing the current conditions.

Reset

Changes the method clock time to **INIT**, causing the initial conditions specified by the method to occur. This button functions only when the detector is under Method control (see Section 2.6.2).

If the method is running, it continues running. If the method is in Hold, the method clock executes the initial conditions and holds.

Select Δ and **Select** ∇

When the cursor is positioned at a field with predetermined parameters, these buttons cycle through the options. For instance, to enter a negative potential when operating in the Amperometry or Voltammetry mode, press **Select** Δ or **Select** ∇ to toggle from the plus sign to the minus sign.

In fields which have predetermined numerical values, pressing **Select** Δ increases the value by one unit, while pressing **Select** ∇ decreases the value by one unit. Holding down a **Select** button increases (or decreases) the value continuously. Press **Enter** to place the new value into effect.

←, ↑, →, and ↓

The four cursor directional buttons move the cursor, in the direction of the arrow, to the next entry field. If there is no changeable field in that direction, the cursor moves diagonally or remains where it is.

Help

Displays a help screen with information pertaining to the current entry field.

Menu

From any operational screen, pressing **Menu** displays the **MENU of SCREENS**. From any diagnostic screen, pressing **Menu** once returns you to the **DIAGNOSTIC MENU**. Pressing **Menu** again returns you to the **MENU of SCREENS**.

Numeric Buttons

Enters numeric values into the current entry field. The numeric buttons are 0 through 9 and the decimal.

Enter

Saves and/or executes changes made in entry fields. After pressing **Enter**, the cursor moves back to the left margin of the same field. It does not automatically move to the next field.

From menu screens, pressing **Enter** displays the highlighted screen.

On the **METHOD** and **WAVEFORM** screens, pressing **Enter** after editing a method parameter does not save the change. To save your editing changes, move the cursor to the **SAVE TO** field, enter the method number, and press **Enter**.

2.2 Rear Panel

The ED40 rear panel (Figure B-2) contains fuses, connectors for line power, and a connection for the optional DX LAN interface.

Power Entry

The power entry, fusing, and EMI filter are mounted on the rear of the 45 W power supply module. The power entry is socketed for a modular power cord (IEC 320 C13). The detector requires a grounded, single-phase power source. The detector may be operated from 85 to 270 Vac, 47 to 63 Hz power. The input power is 50 W maximum. The line voltage is automatically selected and requires no adjustments.



SHOCK HAZARD—If a grounded receptacle is not used, a shock hazard may result. Do not operate or connect to AC power mains without earthed ground connections.

Fuses

The ED40 uses two 3.15 A slow-blow fuses (IEC 127 type 1, P/N 954745). See Section 5.5 for instructions on replacing the fuses.



For continued protection against risk of fire or shock, replace with fuses of the same type and rating.

DX LAN Connection (Optional)

When you order the DX LAN network, a standard BNC connector is factory-installed in the upper left corner of the ED40 rear panel (see Figure B-2).

NOTE

In order to use PeakNet Software to control ED40 operation via the DX LAN, a detector interface card (P/N 044196) must be installed in the ED40. See Section B.3.2 for card installation instructions.

External Connection Access

Connections to the front of the electronics chassis, such as TTL and relay cables, are routed to the back of the detector through the cable chase in the bottom of the electronics chassis; they may also be passed through slots at the front of the detector. Cables exit the ED40 through an opening in the rear panel.

2.3 Electronics Chassis

The electronics chassis contains the ED40 electronics and electrical connections. The chassis is located immediately behind the front door of the ED40. A label on the inside of the door illustrates the position of the electronics cards (printed circuit boards) and connectors, as does Figure 2-3 in this manual.



Do not remove any of the electronics cards from the detector. There are no user-serviceable components on the cards. If servicing is required, it must be performed by qualified personnel following appropriate electrostatic discharge (ESD) handling procedures.

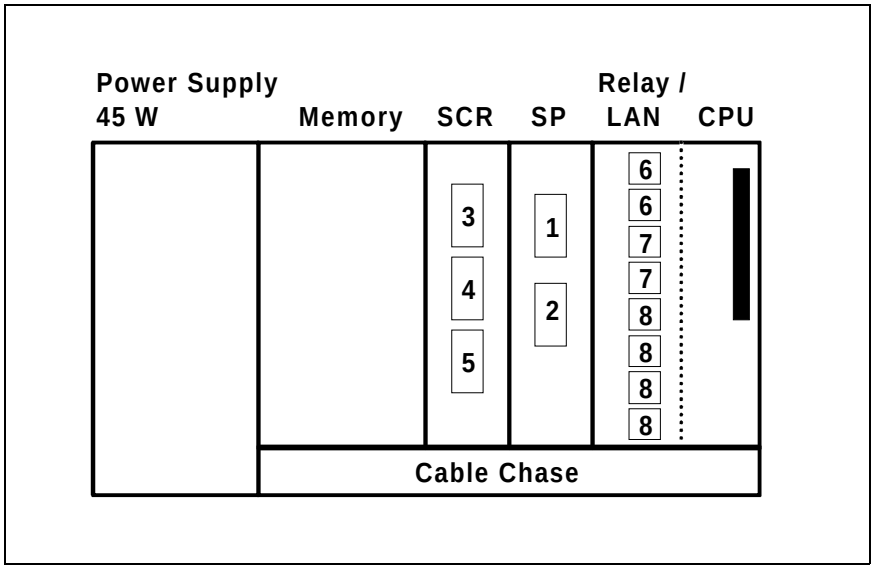


Figure 2-3. ED40 Electronics Chassis Components

2.3.1 Connectors

There are electrical connections to three cards in the ED40 electronics chassis. The numbers in the list below correspond to the numbered connectors identified in Figure 2-3.

- 1—**Amperometry Cell**—Provides all connections to the amperometry cell.
- 2—**Conductivity Cell**—Provides all connections to the conductivity cell, including temperature compensation.
- 3—**Analog Output**—Provides for easy attachment of hook-up wire leads, using a small screwdriver. This connector is typically used with a recorder/integrator or diagnostic instruments. For the connector pinout descriptions, see Table E-1 in Appendix E.
- 4—**SRS**—Provides for all connections, including power, to the Self-Regenerating Suppressor (SRS™).

- 5—**DS3**—Provides for all connections, including power, to the DS3 Detection Stabilizer.
- 6—**Relay**—Provides 2 relay output connector plugs for easy attachment of hook-up wire leads, using a small screwdriver.
- 7— **TTL Output**—Provides 2 TTL output connector plugs for connecting the ED40 to another TTL-compatible instrument.
- 8—**TTL Input**—Provides 4 TTL input connector plugs for control of the detector from another TTL-compatible instrument. These connectors are identified as TTL1 through TTL4 on the **TIME FUNCTION IN** screen.
- **60-pin ribbon connector**—Provides the connector plug for the 60-pin ribbon cable to the ED40 front panel (display and keypad).

2.3.2 Cards

Power Supply Card

Provides 45 watts of power for the detector electronics.

Memory Card

Contains memory chips used by the CPU card.

SCR (Supply Control/Relay) Card

Interfaces to the CPU. The SCR card contains three functions:

- *16-bit Recorder Output Digital-to-Analog Converter*—Includes an electronic switch for selection of full-scale outputs of 0.01, 0.1, and 1.0 V.

- *SRS Power Supply*—Supplies a regulated current (set by the user) of 50, 100, 300, or 500 mA to the Self-Regenerating Suppressor. An over-voltage detector shuts off the power if the voltage exceeds 8.5 V. An over-temperature detector shuts off the power if the SRS temperature exceeds 40 °C. If either of these events occurs, the SCR card sends an "SRS Alarm" error message to the CPU.
- *DS3 Power Supply*—Supplies heating power to the DS3 Detection Stabilizer. While warming or cooling to a lower set point, a "BELOW TEMP" or "ABOVE TEMP" message is displayed. When a set point in the range of 25 to 45 °C is reached, proportional heat control maintains a constant temperature.

SP (Signal Processor) Card

Contains the digital circuitry to interface to the CPU, as well as all the analog circuitry required for the four detection modes. Table D-1 in Appendix D lists the SP card functions.

Relay/DX LAN and CPU Cards

The ED40 control Moduleware and BIOS reside on the CPU logic and Relay I/O cards.

- The CPU card provides control and monitoring of the other modules. A 60-pin ribbon cable assembly links the logic to the ED40 front panel display and keypad.
- The Relay I/O card provides two isolated low voltage relay outputs, two TTL outputs, and four TTL inputs.

The cards are in slot 5 of the card cage. The Relay card is a half-card which rides piggyback on the CPU card and extends over the front of slot 4.

The Relay I/O card is short enough to allow a detector interface card (P/N 044196) to be mounted behind it in slot 4. The interface card is required for communication between the ED40 and PeakNet Software.

Below the I/O connections is a multicolor LED that indicates the state of the power supply.

- A green LED indicates normal operation.
- A red or yellow LED indicates a fault. If a fault occurs, the ED40 enters its diagnostic state and no other control is permitted until the fault is corrected.

2.4 Conductivity Cell

NOTE

Two versions of the conductivity cell are available. One is in a DS3 Detection Stabilizer (P/N 044130), while the other is inside a shield (P/N 044132). Because the DS3 maintains a constant temperature, thus reducing the effects of temperature variations, the cell with a DS3 is recommended for conductivity detection.

The flow-through cell has an active volume of about 1.0 µL. Two 316 stainless steel electrodes are permanently sealed into the PEEK cell body. The cell constant has a nominal value of 200 cm^{-1} and is calibrated electronically. A sensor located slightly downstream from the electrodes senses the temperature of liquid exiting the cell. The measured value is used to calculate temperature compensation.

The advanced geometry of the cell provides several benefits:

- Excellent accuracy and linearity within the working range.
- Efficient sweepout and low volume for low dispersion.
- Low drift due to stable electrode surface conditions.
- Low electrode mass.
- Effective temperature compensation.

Temperature Control and Compensation

Temperature directly affects the conductivity of a solution. As conductivity increases, the effect of temperature changes becomes more pronounced. For example, building temperature control systems can cause a regular oscillation in the baseline. This, in turn, can affect the reproducibility of an analysis.

In ion chromatography, suppressing the eluent conductivity reduces the effect of minor temperature variations. At high sensitivities, however, conductivity changes resulting from even minor temperature variations represent a significant percentage of the total conductivity. Installing the conductivity cell in a DS3 Detection Stabilizer reduces these effects to below the detection limit. Temperature compensation in the ED40 further reduces the effects of temperature by normalizing all measured conductivities to 25 °C.

2.4.1 DS3 Detection Stabilizer

The DS3 Detection Stabilizer is a temperature-controlled chamber consisting of a cast aluminum base and cover enclosed in insulating foam. The chamber houses both the conductivity cell and the eluent heat exchanger. Figure 2-4 identifies the major components of the DS3.

The DS3 provides the following benefits:

- Conductivity measurements that are nearly impervious to temperature variation.
- Very low dispersion in the eluent heat exchanger.
- Excellent peak height reproducibility.
- Remote mounting at either the column or suppressor.
- User-settable temperature control.

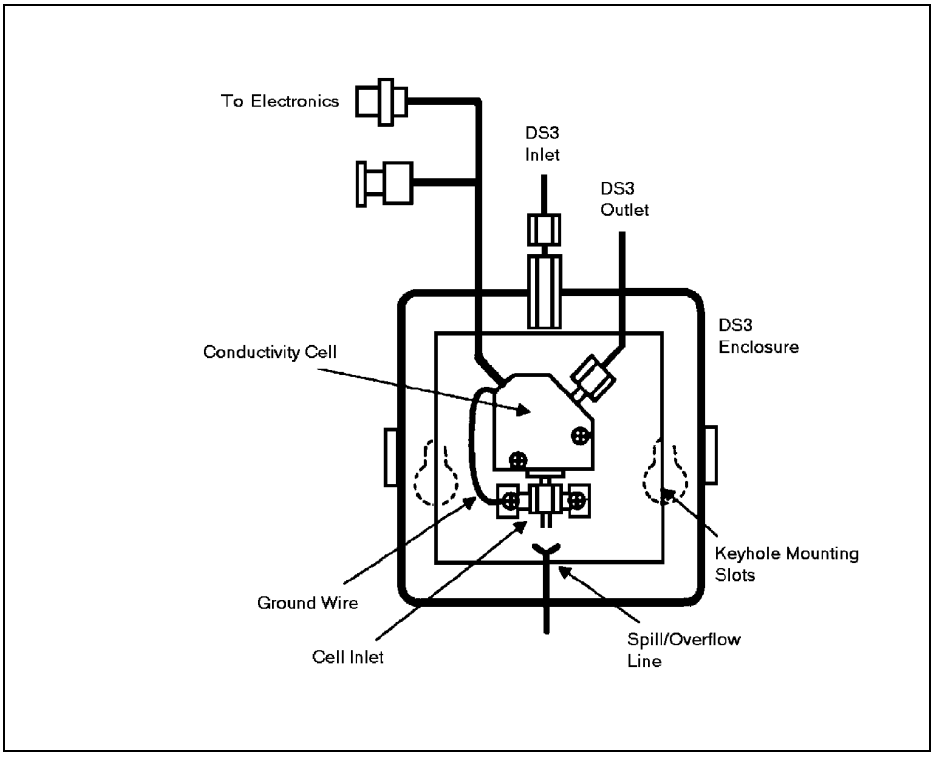


Figure 2-4. DS3 Detection Stabilizer

Power input to a pair of transistors on opposite sides of the DS3 heats it to a set temperature from 25 °C to 45 °C. A sensor near the heat exchanger outlet senses the eluent temperature. The ED40 circuitry compares this temperature with the user-selected temperature.

The bottom edge of the DS3 housing is sealed, so as to trap spilled liquid. Two leak sensors become wet after 5 mL of liquid is collected. As filling continues, liquid exits via the spill overflow line. The second leak thermistor stays dry and acts as a reference. When the first thermistor gets wet, its temperature drops below the reference, which triggers an alarm to the detector.

2.4.2 Shielded Cell

The shield is an aluminum housing consisting of a base and a cover. The aluminum housing provides protection from electromagnetic interference (EMI).

2.5 Amperometry Cell

The ED40 amperometry cell is a miniature flow-through cell with a titanium cell body (the counterelectrode) and a combination pH–Ag/AgCl reference electrode. The working electrode is available in four materials. Order the appropriate cell for your application:

- Amperometry cell, gold W.E. (P/N 044108)
- Amperometry cell, platinum W.E. (P/N 044109)
- Amperometry cell, silver W.E. (P/N 044110)
- Amperometry cell, glassy carbon W.E. (P/N 044111)

Oxidation or reduction of analyte molecules is accomplished by applying a potential between the working and reference electrodes. The reference electrode is chosen so that the potential difference between it and the solution is fixed by an electrochemical redox couple. Either half-cell of the pH–Ag/AgCl reference electrode may serve as the cell reference electrode. Any changes in the potential applied between the working and reference electrodes will be developed between the working electrode (where analyte reduction or oxidation takes place) and the solution. To maintain a constant potential difference between the reference electrode and the solution, the cell current must be prevented from flowing through the reference electrode. A section of the ED40 electronic circuit (the *potentiostat*) diverts the cell current through the counterelectrode. The potentiostat automatically compensates for the solution resistance between the reference electrode and the counterelectrode.

The ED40 amperometry cell is a thin-layer design. Mobile phase flows in a thin channel parallel to the surface of a flat disk electrode. The resulting smooth flow minimizes noise. The low volume (0.2 µL) of the channel also allows operation with high efficiency, narrow bore columns. The cell design minimizes the electrical resistance between the working electrode and the counterelectrode by locating the counterelectrode (the titanium cell body) directly across the thin-layer channel from the working electrode. This results in a wide linear dynamic range.

The counterelectrode is connected to ground by a length of titanium inlet tubing. This shunts minute electric currents that might conduct from the pump through the flow stream into the working electrode. The working electrode current is processed using low noise analog amplifiers and filters. Additional digital filtering of the analog output is available.

The ED40 amperometry cell is installed directly after the column (a suppressor is generally not used). A second detector, such as the AD20 Absorbance Detector, may be installed in-line with the amperometry cell as long as the pressure at the amperometry cell inlet remains less than 700 kPa (100 psi). Because of the volume within the reference electrode section of the cell (67 µL total cell volume), there may be some band broadening at the second detector. However, this is reduced by the precision flat-bottomed reference electrode.

2.5.1 Combination pH–Ag/AgCl Reference Electrode

The reference electrode is a standard combination pH electrode containing a glass membrane pH half-cell and a Ag/AgCl half-cell. The combination electrode monitors mobile phase pH, which is displayed on the **DETAIL** screen and is also available as an analog output. To obtain an accurate pH readout, calibrate the electrode before use from the **pH CALIBRATION** screen.

The Ag/AgCl half-cell is normally used as the cell reference electrode. The pH half-cell can be used as the reference electrode during a pH gradient, to minimize changes in the baseline. The potentials at which many redox reactions take place on metallic electrodes are pH-dependent, with the potential shifting -0.059 V per pH unit. This is especially true for metal oxide formation and reduction reactions. Since the reference potential of the pH half-cell also shifts by -0.059 V per pH unit, pH-dependent potential shifts at the working electrode are canceled.

At a mobile phase pH of 7, the reference potential of the pH half-cell is the same as that of the Ag/AgCl half-cell. As the mobile phase pH is increased, the pH half-cell potential decreases approximately 0.059 V per pH unit. For example, at a mobile phase pH of 12, the reference potential of the pH half-cell would be -0.295 V relative to the Ag/AgCl half-cell. Therefore, at pH 12, the potentials applied to the working electrode must be raised approximately 0.3 V when switching from the Ag/AgCl reference to the pH reference.

In acidic mobile phases, the reference potential of the pH half-cell is positive with respect to the Ag/AgCl half-cell, and all applied potentials must be decreased by 0.059 V per pH unit when switching from the Ag/AgCl half-cell reference to the pH reference.

2.6 Functional Description

This section discusses the ED40 Electrochemical Detector functional operation, including operating modes and types of control.

2.6.1 Modes of Operation

The ED40 has two operating modes: ***Local*** and ***Remote***. When the detector is powered up, it is always in Local. To change the operating mode, go to either the **MAIN** or **DETAIL** screen. There, move the cursor to the **LOCAL** field and use the **Select** Δ or **Select** ∇ button to toggle the mode. Press **Enter** when the mode is correctly selected. When you select **REMOTE**, you will be prompted to confirm the selection.

Local Mode

Local mode allows two types of commands:

- Direct entry onto a screen, using the front panel buttons
- TTL and Relay inputs

TTL logic levels can control any four of the detector functions listed below. The functions are defined from the **TIME FUNCTION IN** screen.

- OFFSET
- HOLD/RUN
- SRS OFF/ON
- METHOD NUMBER INCRement
- METHOD NUMBER DECRement
- MARK Recorder
- Increase RANGEX10

This allows control of the ED40 via a remote controller or the timed event function of an integrator. The remaining functions are controlled from the ED40 front panel buttons.

No special setup or switch is required for TTL control, which is always available in Local mode. Front panel control remains active.

Remote Mode

NOTE
For more information about Remote control, refer to the *PeakNet Software User's Guide.*

Remote mode allows you to monitor the detector status and control all detector functions from PeakNet Software, via the DX LAN interface. Select the Remote mode from the **MAIN** or **DETAIL** screen, or from within PeakNet.

There are two forms of Remote control: Remote and Locked Remote. While operating in normal Remote mode, all buttons on the ED40 front panel function except **Hold/Run**. **Enter** can be used to select display functions that do not interfere with DX LAN remote control of a method while it is running.

The Locked Remote mode locks out all parameter changes from the ED40 front panel. Locked Remote can be selected only from the computer; it can be cleared only from the computer or by turning off the ED40 power. When the ED40 is powered up again, it will be in Local mode.

If you select the Remote mode while the ED40 is running a method, the computer will not interrupt the method unless you select the Abort command from within PeakNet.

After PeakNet downloads a method to the ED40, the computer activates the method number with a DX LAN command and the **INIT**ial conditions step is activated. If a method is already running, it is interrupted and the ED40 method clock is reset to **INIT**ial conditions. A subsequent Run command will cause the method clock to run, activating the timed event starting with the time 0.00 step of the method.

2.6.2 Control

There are two types of control in either Local or Remote mode: ***Direct*** control and ***Method*** control.

Direct Control

In Direct control, commands are executed as soon as they are entered. Since there is no time-based program, the method clock is not used. The **Hold/Run** and **Reset** buttons are not operable in Direct control.

To select Direct control, first go to either the **MAIN** or **DETAIL** screen. If **DIRECT CNTRL** is displayed, the mode is already selected and no further action is necessary. If **METHOD** is displayed, move the cursor to **METHOD** and press the **Select** Δ or **Select** ∇ button to toggle between **DIRECT CNTRL** and **METHOD** in the display. Press **Enter** to activate the selection.

If a method is running when you select Direct control, that method is aborted and the method clock is reset.

Method Control

In Method control, commands are executed as programmed in a method containing time-based program steps. The parameters below are method-controlled and cannot be changed from the ED40 front panel:

- Detection mode (Conductivity, DC Amperometry, Integrated Amperometry)
- Analog range
- Offset
- Mark
- Relays and TTLs
- SRS current (Conductivity)
- DS3 temperature (Conductivity)

- Temperature Compensation factor (Conductivity)
- Potential (DC Amperometry)
- Waveform number (Integrated Amperometry)

There are three ways to change a method-controlled parameter:

- Edit the currently running method, then save the changes. The changes will be implemented when the method is saved.
- Switch to a different method.
- Abort the method, go to Direct control, and enter the new parameters directly.

Methods are programmed and controlled from the **METHOD** screen. To select Method control while in Direct control, go to either the **MAIN** or **DETAIL** screen. Move the cursor to the **DIRECT CNTRL** field and press the **Select** Δ or **Select** ∇ button, then press **Enter**. **METHOD** will replace **DIRECT CNTRL** in the display.

Each method can contain up to 32 separate time-based steps, starting at time zero (**TIME = 0**). A method is created by first selecting a method number from 0 through 99. The steps are then created, one-by-one, by entering first the time and then the operating parameters you want to be in effect at that time.

Methods are retained in memory even after the detector power is turned off. Up to 100 methods (00 through 99) can be stored in ED40 memory. The actual total depends on the size of each method and the amount of available memory and is usually less than 100.

Besides storing and running methods, the ED40 allows you to run the detector under Method control while you are entering or editing any method, even one that is currently running. When saving changes to the currently running method or switching to a different method, the method clock continues

running unaffected. Only those parameter changes which affect the method *after* the current time will be implemented in the current run. Of course, you may intentionally press **Reset** to implement the initial conditions.

You may enter non-method programmed parameters from the front panel, as well as display screens to monitor detector operation.

Another instrument, such as the GP40 Gradient Pump, can control the ED40 by a method in the pump through the ED40 TTL input ports.

The chromatography system is typically set up so that one module (such as the pump) contains the Method control and drives other modules (such as the detectors).

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3 • Operation and Maintenance

3.1 Getting Ready to Run

After installing the ED40 Electrochemical Detector, or after the detector power has been off for some time, use the following check lists to ready the detector for operation.

All Detection Modes

- Verify that all cables are correctly connected.
- Verify that the ED40 power cord is plugged into the main power.
- Press the power switch actuator on the ED40 front panel to turn on the power (see Figure 2-2).
- Verify that the ED40 passed all of its power-up tests (see Section 3.2).

Conductivity Mode

- If the conductivity cell is in a DS3 Detection Stabilizer, set the DS3 temperature from the **DETAIL** screen:

  If the DS3 is installed in an LC30 Chromatography Oven, select a temperature at least 5 °C above the oven temperature.

  If the DS3 is not installed in an LC30, select a temperature at least 5 °C above the highest expected ambient temperature surrounding the DS3.

- Turn on the DS3 power. The DS3 warms at about 1 °C/minute. After it reaches the set temperature, the baseline conductivity should stabilize.
- Turn on the SRS as soon as the proper current is determined and eluent is flowing through the suppressor at the correct rate. The suppressor may take longer to stabilize than the DS3. The drift usually decreases as SRS efficiency improves.

- While waiting for acceptable drift, you may want to select a lower sensitivity. Set the offset to 50%.
- When starting a run, if you have not already done so, select the desired sensitivity and offset. Press **Offset** before injection and during a run, also, if necessary.
- The SRS is programmed and monitored from the **DETAIL** screen. For operational requirements, review the manual shipped with the SRS.

Integrated Amperometry Mode

- Create a potential vs. time waveform or edit an existing waveform. In Local mode, do this from the **WAVEFORM** screen. Verify that the correct waveform is selected on the **MAIN** or **DETAIL** screen or, if using a method, on the **METHOD** screen.
- If necessary, calibrate the reference electrode from the **pH CALIBRATION** screen.
- Polish the working electrode (Section 5.5).
- Verify that the cell is installed and that all tubing is properly connected.
- Turn on the pump.
- Turn on the cell and allow the baseline to stabilize. The detector output normally drifts downward for about 1 hour as the baseline stabilizes.

DC Amperometry Mode

- Enter the applied potential on the **MAIN** or **DETAIL** screen or, if using a method, on the **METHOD** screen.
- If necessary, calibrate the reference electrode from the **pH CALIBRATION** screen.
- Polish the working electrode (Section 5.5).
- Verify that the cell is installed and that all tubing has been properly connected.

- Turn on the pump.
- Turn on the cell and allow the baseline to stabilize.
- When the working electrode is glassy carbon, the detector output typically drifts downward for up to one day. Setting up the ED40 the day before beginning an analysis allows enough time for the baseline noise to diminish considerably. To conserve mobile phase during this time, set the flow rate to 25% of the value required for the analysis. The ED40 will stabilize quickly once the flow rate is increased to the proper value.

3.2 Initial Screens

Each time the ED40 power is turned on, the detector begins running a series of internal diagnostic and calibration procedures. As each test is executed, the test name and result appear on-screen.

If the detector passes all the diagnostic tests, the **POWER-UP** screen is displayed momentarily.

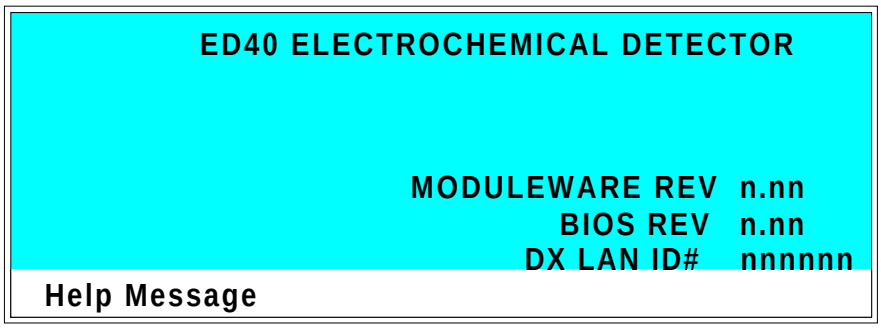


Figure 3-1. Power-Up Screen

The revision codes on the **POWER-UP** screen identify the Moduleware and BIOS, in the event that service is ever needed. If the ED40 is connected to a Dionex PeakNet Workstation, via the DX LAN interface, the DX LAN identification number also appears here.

If a system component fails a power-up diagnostic test, the tests will continue to run; however, subsequent tests may have to be omitted. If a critical feature fails, the ED40 displays the **DIAGNOSTIC TESTS** screen (one of the options on the **DIAGNOSTIC MENU**). You can run the diagnostics without turning off the detector power and repeating the power-up sequence. Refer to Appendix C for more information about diagnostic tests.

After 6 seconds, the display changes from the **POWER-UP** screen (or the **DIAGNOSTIC TESTS** screen) to the **MAIN** screen. This screen displays active data in large characters, to enable easy viewing from a distance. Because each of the ED40 detection modes requires different parameters, each mode has a unique **MAIN** screen. Figure 3-2 shows the **MAIN** screen for the Conductivity mode.

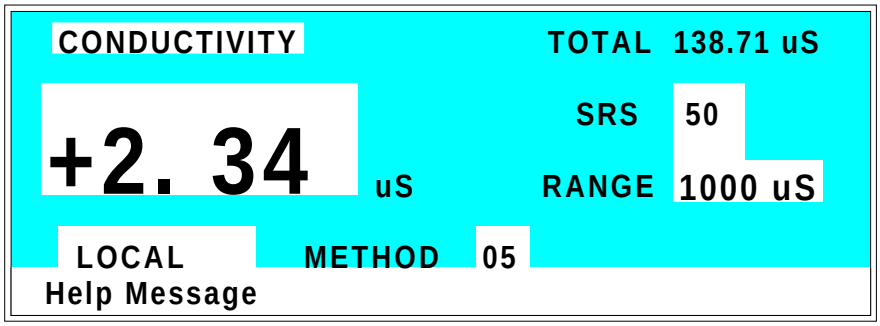


Figure 3-2. Main Screen—Conductivity

Press the **Menu** button to go to the **MENU of SCREENS**. There, begin selecting parameters for the Direct control or Method control operating mode. The operating modes are described in Sections 3.3 and 3.4.

3.3 Running Under Direct Control

When the Direct control operating mode is selected, real-time commands are carried out instantly and all detector settings are in effect until you change them. Changes to parameters are executed when entered. Because there are no time-based steps, the method clock is not used. The **Hold/Run** and **Reset** buttons are not operable in Direct control.

To select Direct control, first go to either the **MAIN** or **DETAIL** screen.

- If **DIRECT CNTRL** is displayed, the mode is already selected and no further action is necessary.
- If **METHOD** is displayed, move the cursor to **METHOD** and press the **Select** Δ or **Select** ∇ button to toggle between **METHOD** and **DIRECT CNTRL** in the display. Press **Enter** to activate the selection.

3.4 Running Under Method Control

In the Method control mode, a series of programmed timed events, known as a ***method***, controls the ED40. Methods are retained in memory even when the detector power is turned off. The detection mode determines which parameters can be controlled by a method. For detailed information about method parameters, refer to the appropriate section:

- Conductivity—Section C.1.4
- Integrated Amperometry—Section C.1.8
- DC Amperometry—Section C.1.13

There are two ways to select Method control:

- Go to the **MAIN** or **DETAIL** screen. If **METHOD** is displayed, the mode is already selected and no further action is necessary. If **DIRECT CNTRL** is displayed, move the cursor to **DIRECT CNTRL** and press the **Select** Δ or **Select** ∇ button to toggle between **DIRECT CNTRL** and **METHOD** in the display. Type in a method number and press **Enter** to activate the selection.
- Go to the **METHOD** screen. Move the cursor to the **RUN** field, enter a method number, and press **Enter**.

If the clock on the **MAIN** or **DETAIL** screen is **INIT**ial when you press **Enter**, the ED40 uses the method **INIT**ial condition parameters to control the module. If the method clock is greater than zero when you press **Enter**, the ED40 uses the method parameters in effect at that time.

Pressing **Run** starts the method clock. From the **INIT**ial conditions, the time 0.00 step is executed as soon as **Run** is pressed. The remaining steps will be executed according to their programmed times.

3.4.1 Creating a New Method

1. Go to the **METHOD** screen for the detection mode. Enter an unused method number from 0 through 99 in the **EDIT** field and press **Enter**. A blank method will be displayed on-screen.

   The first step of every method is an initial conditions step with **INIT** in the **TIME** field. The second step is always a time step with 0.00 in the **TIME** field. You cannot delete these steps, although you may change their parameters.

2. Enter the parameters for the initial conditions and time 0.00 steps. Use the arrow buttons to move the cursor from one field to another.

   NOTE

   The TIME field is the only field in each method step that *must* have an entered value. Leaving any other field blank indicates that there is no change from the value selected for that parameter in the preceding step.

3. To create a new method step, move the cursor to a blank **TIME** field, enter the time (in minutes) at which you wish an action to be performed, and press **Enter**. Enter the values for each step parameter, or leave a field blank to have the previously selected value remain in effect.

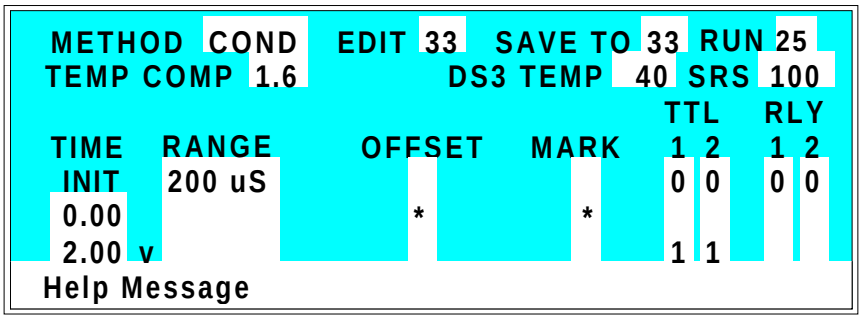


Figure 3-3. Method Screen—Conductivity

4. Repeat Step 3 to add additional steps. Up to 30 steps (32 steps, if PeakNet Software is used) can be added after the time 0.00 step. If the method contains more steps than can be displayed on the screen at one time, you can view the additional steps by moving the cursor to the top or bottom screen entry and then stepping one more line, as described here:

   When there is a small **v** next to the time entry at the bottom of the screen, move the cursor *down* to view the additional steps.

   When there is a caret (^) next to the top time entry, move the cursor *up* to view the additional steps.

5. To save the new method, move the cursor to the **SAVE TO** field, enter the number that appears in the **EDIT** field, and press **Enter**.

3.4.2 Editing an Existing Method

You can modify an existing method by changing, adding, or deleting steps and/or parameters. If you edit a method while it is running, the changes are stored in memory when you **SAVE TO** the method number. Changes take effect as soon as they are saved.

To edit an existing method, go to the **METHOD** screen, enter the method number in the **EDIT** field, and press **Enter**. Follow the instructions in the sections below. Remember to save your changes before exiting the **METHOD** screen; if you do not, the changes will be lost.

NOTE

Once you save editing changes to a method, there is no way to recall the original method. To make experimental changes to a method while retaining the original method in its unmodified form, save the new method, or a copy of the original method, under a different method number.

Changing Method Parameters

Move the cursor to the field(s) you wish to edit and enter a new value, using the ED40 front panel buttons. Press **Enter** after each editing change.

Adding a Method Step

There are two ways to add a step to an existing method:

- Move the cursor on the **METHOD** screen to any **TIME** field. Enter the time and parameters for the new step, and then press **Enter**. If the new step is not at the correct chronological point in the method, it will be moved to the correct location.
- Move the cursor on the **METHOD** screen to the line immediately preceding the intended location of the new step. Press **Insert** to insert a new, blank line below the cursor location. Enter the time and parameters for the new step, and then press **Enter**.

Deleting a Method Step

Move the cursor on the **METHOD** screen to the time of the step to be deleted and press **Delete** twice.

Deleting an Entire Method

Move the cursor on the **METHOD** screen to the **EDIT** field and press **Delete** twice.

Saving a Modified Method

To ***replace*** the original method with a modified version, enter the number of the original method in the **SAVE TO** field and press **Enter**.

To ***retain*** the original method and save the modified version elsewhere in memory, enter an unused method number in the **SAVE TO** field and press **Enter**.

3.4.3 Running a Method

1. Go to the **MAIN** or **DETAIL** screen. If necessary, toggle from **DIRECT CNTRL** to **METHOD** and from **REMOTE** to **LOCAL**.
2. In the **METHOD** field, enter the desired method number and press **Enter**. (You can also select the method number from the **METHOD** screen. To do so, move the cursor to the **RUN** field, enter a method number, and press **Enter**.)
3. If the method clock is already running when you enter the method number, the method starts immediately. If the clock is in Hold, press **Hold/Run** to start the method.
4. The elapsed time on the method clock when the method begins determines where (i.e., at what step and parameters) the method begins running:

   If the method clock is at **INIT** or time zero, the method begins running using the **INIT** condition parameters.

   If the method clock is greater than zero, the method begins running using the parameters specified in the step for that elapsed time. Press **Reset** to start the method at the **INIT** conditions.

3.4.4 Changing the Running Method

To change from the method currently running and begin running a different method, enter the new method number in the **RUN** field on the **METHOD** screen and press **Enter**. The new method will begin running, using the parameters specified in the step for the current elapsed time. Press **Reset** to start the method at the **INIT** conditions.

3.5 Optimizing Temperature Compensation

The ED40 built-in temperature compensation reduces the effects of temperature variations, thereby improving conductivity detection. Temperature compensation corrects for long-term and medium-term changes in ambient temperature; it is not intended to compensate for a difference between the set and calibrated temperatures. For more information about temperature control and compensation, see Section 2.4.

3.5.1 With a DS3 and Conductivity Cell



If the ambient temperature exceeds 40 °C, damage to the DS3 may result. If the DS3 is installed in an LC30 Chromatography Oven, do not set the oven temperature above 40 °C (104 °F). Higher temperatures may permanently damage the DS3.

When the conductivity cell is inside a DS3 Detection Stabilizer, the temperature variations of the liquid reaching the cell should be negligible. Once you set **TEMP COMP** on the **DETAIL** screen to 1.7% per ºC, it should not have to be reset.

The DS3 normally operates at a single temperature. For optimal accuracy, calibrate the conductivity cell at this temperature, using the proper temperature coefficient setting. If you select a different temperature, the temperature compensation operates on the difference and normalizes conductivity measurements to 25 °C (77 °F).

If temperature-induced baseline cycling occurs, it is probably caused by another component of the chromatography system. If the variation increases as the eluent reservoir empties, relocate the reservoir to a more temperature-stable environment and/or wrap the reservoir in insulation.

3.5.2 With a Shielded Conductivity Cell

If the conductivity cell is not inside a DS3, the baseline will drift up and down with fluctuations in laboratory temperature. This is especially noticeable when the thermostat cycles on and off in laboratories with thermostatically-controlled temperature. Selecting the proper temperature compensation factor can help to minimize the effect of temperature fluctuations.

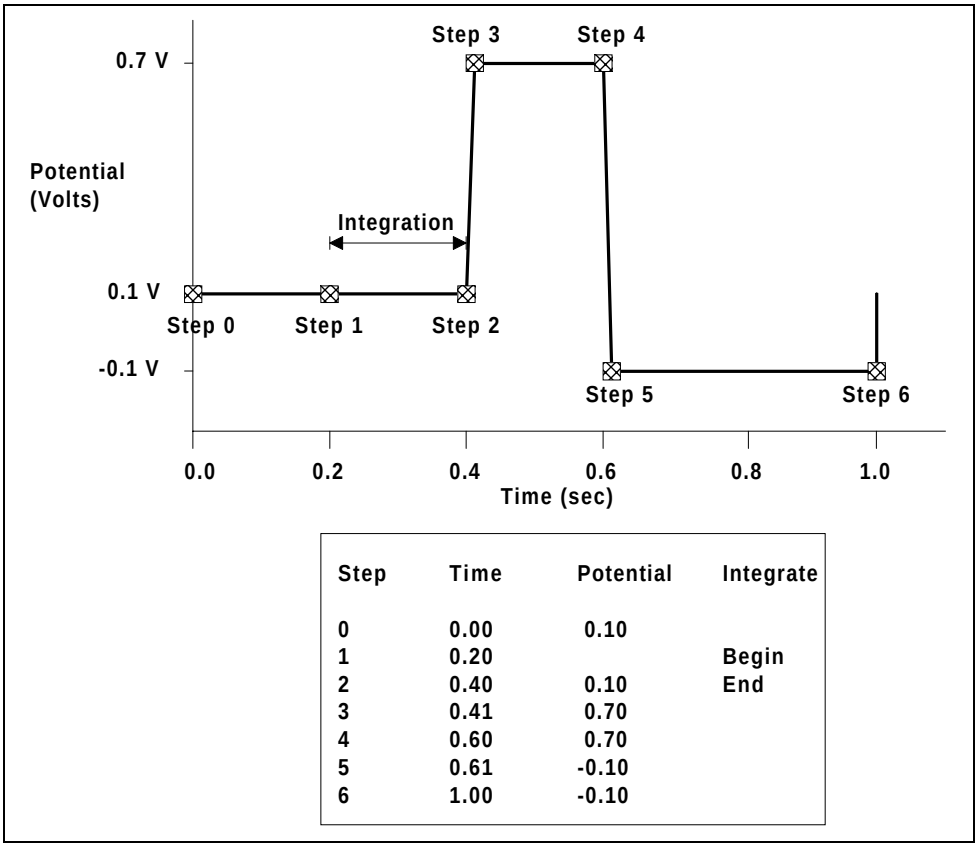
Start by setting **TEMP COMP** on the **DETAIL** screen to 1.7%. If a sinusoidal baseline variation of the same period as the laboratory cooling or heating occurs, increase or decrease the temperature compensation setting. Continue adjusting it until you find the optimal setting; this is typically between 1.5% to 2% per ºC for most systems, suppressed or not.

If you notice a slowly increasing or decreasing temperature pattern in a laboratory without a temperature control system, look for a corresponding baseline drift. If this drift occurs, adjust the temperature compensation setting until you find the optimal setting.

3.6 Waveforms

A waveform is a series of steps, defined as points on a plot of potential vs. time. Waveforms must be defined for the Integrated Amperometry and Voltammetry modes. Entering a waveform is similar to entering a method.

Figure 3-4 shows an example waveform and the waveform program that created it. In this program, the potential does not need to be entered in Step 1 because it remains the same as in Step 0. After Step 6, the waveform automatically reverts to the Step 0 potential.



Step	Time	Potential	Integrate
0	0.00	0.10	
1	0.20		Begin
2	0.40	0.10	End
3	0.41	0.70	
4	0.60	0.70	
5	0.61	-0.10	
6	1.00	-0.10	

Figure 3-4. Sample Waveform

3.7 Voltammetry

The Voltammetry mode is used to develop waveforms for Integrated Amperometry and to determine appropriate potentials for DC Amperometry. It is similar to Integrated Amperometry in that a repeating potential vs. time waveform is applied to the cell. It differs in that the ED40 output is the cell current, which is continuously monitored and reported as in DC Amperometry. The information gained by studying instantaneous cell current can be useful for developing waveforms for Integrated Amperometry.

With the pump on, and mobile phase and analyte flowing through the amperometry cell, results are similar to those obtained by rotated disk voltammetry in a standard beaker cell. With the flow off, rapid depletion of analyte next to the working electrode is typical of thin-layer voltammetry.

To perform cyclic voltammetry with the ED40, program a triangle wave as the waveform (see Section 3.9.3).

3.7.1 Non-Dionex Voltammetry Cells

You may use cells other than the ED40 flow-through cell, so long as you install them correctly. For instance, Cypress Systems, Inc.[1] provides a low volume (several mL) voltammetry cell. Because the working electrodes of the Cypress cell have a diameter of 1 mm, equal to that of the ED40 cell working electrodes, current magnitudes obtained from the two cells are comparable.

The following procedure explains how to connect the Cypress Systems cell electrodes to the ED40 pre-amp board. This setup requires a special cable (P/N 049161), available from Dionex.

[1]Cypress Systems, Inc.
P.O. Box 3931
Lawrence, KS 66046 USA

Phone: 913-842-2511
Toll-Free: 800-235-2436
FAX: 913- 832-0406

1. Disconnect the ED40 cell from the chromatograph. Remove the reference electrode and store it in a solution of saturated KCl. (The soaker bottle originally supplied with the reference electrode is convenient.)
2. Remove, rinse, and store the working electrode.
3. Rinse the cell flow path to prevent mobile phase from precipitating in the cell. Dry the cell.
4. Attach the special cell cable (P/N 049161) to the pre-amp board as follows:
   a. Plug the two-pin socket of the cable into the working electrode connector (J1) on the board. The connector can be attached in either direction.
   b. Plug the three-pin socket of the cable into the reference electrode connector (J2). The actual connection is to the center pin, so the connector can be attached in either direction. Since there is no connection to the pH input, disregard the pH readings reported by the detector.
   c. Attach the spade lug (counterelectrode) to the cell body, using the cell cover thumbscrew.
5. Attach the three pins on the other end of the special cell cable to the cell electrodes, as follows:

   Black: Working Electrode
   White: Reference Electrode
   Red: Counterelectrode

3.7.2 Recorder Connections

Cell current is monitored in the Voltammetry mode by connecting the ED40 analog output to a recording device. Use the recorder negative and positive connections on the SCR (Supply Control/Relay) card (pins 1 and 2). See Section E.1 of Appendix E for the Recorder/Diagnostic Signal pinouts.

Data output from cyclic voltammetry is traditionally plotted as current vs. potential, rather than time. This is accomplished by connecting the Amperometry Cell Drive output on the SCR card (pin 7) to the X axis of an X-Y recorder, oscilloscope, or a computer equipped with A/D conversion and X-Y plotting software. Note that PeakNet Software does not currently support the Voltammetry mode.

NOTE

The cell drive output polarity is reversed. Connect the negative input from the recording device to pin 7, and the positive input to pin 10.

To place zero current in the middle of the Y axis, set the **ZERO POSITION** on the **ANALOG OUT SETUP** screen to 50%. Select the most sensitive setting for the range that keeps the current on scale; a less sensitive analog output range may produce an output with excessive digital steps.

3.7.3 Programming the Voltammetry Waveform

Voltammetry waveforms are defined by points on an X-Y graph of potential vs. time, as in Integrated Amperometry. The ED40 includes one Voltammetry waveform program. To create a different waveform, edit this program from the **WAVEFORM** screen.

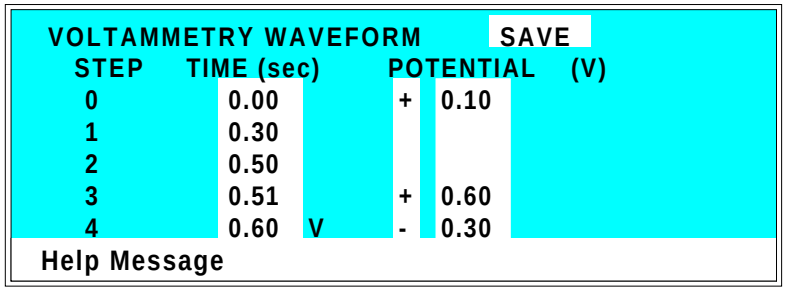


Figure 3-5. Waveform Screen

Cyclic voltammetry programs consist of three steps, each with a time and a potential. The first step is always at time zero. Since this cannot be changed, the first value you enter will be the first potential. The first and third steps have the same potential. To create a new waveform step, enter the time first, and then the potential.

In the Voltammetry mode, the ED40 measures and reports the current every 10 ms. Since the change in potential during each measurement period must be equal, the available potentials are limited to ensure that the potential change is an integer number of mV per 10 ms. (The slowest scan rate is therefore 1 mV per 10 ms, or 0.1 V/s.) If the potential you enter results in a noninteger change, the ED40 will substitute the closest acceptable potential. You may want to use the **Select** Δ and **Select** ∇ buttons to find available potentials.

To calculate the time for the second step of the waveform, divide the total voltage scanned by the desired sweep rate. Enter the remaining times and potentials, in sequence, until the waveform is complete. Be sure to save these editing changes before exiting the **WAVEFORM** screen.

Figure 3-6 shows an example of a triangle wave used in cyclic voltammetry. In this example, the potential is cycled between -0.80 and +0.60 V at a sweep rate of 0.1 V/s.

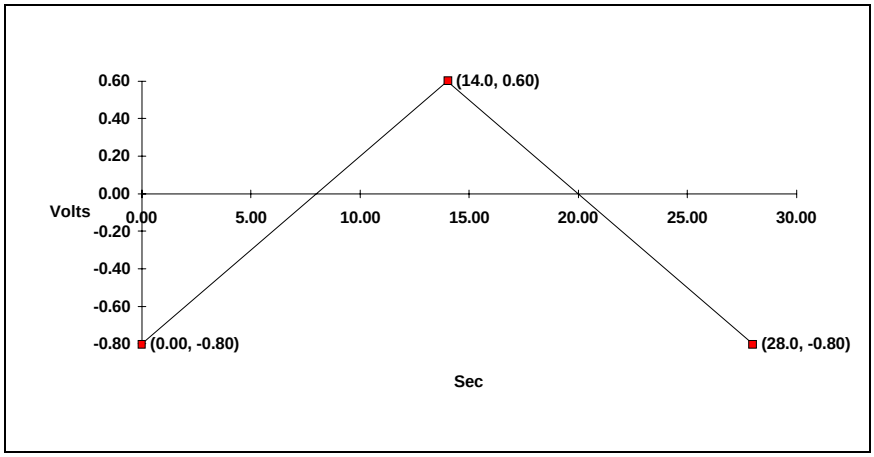


Figure 3-6. Cyclic Voltammetry Example

The first waveform step at 0.00 s is the initial potential of -0.80 V. To calculate the time for the second step, divide the total voltage scanned by the desired sweep rate:

$$\frac{Total\ Voltage\ Scanned}{Sweep\ Rate} = \frac{1.4\,V}{0.1\,{}^{V}\!/_{s}} = 14\,s$$

The time for the second step is 14 s. The third step is an equal interval later, or 28 s.

3.7.4 Running the Waveform

When the cell is turned on, the ED40 applies the initial potential programmed at time zero. At that point, you may do any of the following:

- To begin the waveform, press **Run**.
- To freeze the scan at the current potential, press **Hold**. Press **Run** to continue from that point.
- To return to the initial potential without stopping the scan, press **Reset**.
- To return to the initial potential and hold at that potential, press **Hold** and **Reset**, in that order.

3.8 Routine Maintenance

- The ED40 electronics are not user-serviceable. Do not remove any of the electronics cards (printed circuit boards) from the detector. If servicing is required, it must be performed by qualified personnel.
- Periodically check liquid line connections to the cells (inside the chromatography module) for leaks and clean up any spills.

The ED40 amperometry cells are designed to require minimal maintenance. If you observe the following precautions, the working electrode should rarely require polishing.

- To prevent electrode contamination:
  a. Run only clean, filtered samples.
  b. Prepare all eluents with high purity deionized water.
  c. Avoid contamination of the cell with incompatible eluents.
  d. Never apply potential to the electrode unless a stream of eluent or water is flowing through the cell.

- Be careful to keep the polished surface of the amperometry cell body clean and dry. The gold, spring-loaded working electrode contact must also remain clean and dry. If a salt bridge forms, it can cause an electrical short between the working electrode contact and the cell body.
- Over the lifetime of the working electrode, it is normal for the surface to gradually become pitted. However, if the electrode becomes discolored or if you notice a degradation in performance (baseline noise, tailing peaks, etc.), polish the electrode as instructed in Section 5.5.

3.9 Shutdown

- Whenever the amperometry cell is not being used, remove the pH reference electrode and store it in a solution of saturated KCl, as instructed in the procedure below. If the reference electrode is left in the cell and mobile phase is not being pumped through the cell, the reference electrode frit may partially dry out. If this occurs, regenerate the electrode by soaking it in a solution of 1 M KCl plus 1 M HCl.

  1. Prepare a saturated solution of KCl in deionized water.
  2. Remove the cap of the soaker bottle in which the electrode was shipped.
  3. Fill the soaker bottle at least three-fourths full with the prepared KCl solution.
  4. Remove the pH reference electrode from the cell.
  5. Slip the electrode through the hole in the soaker bottle lid until the electrode cap bottoms out on the top of the lid.
  6. Screw the soaker bottle lid, with the electrode attached, onto the soaker bottle.
  7. Store the assembly in the original shipping box.

4 • Troubleshooting

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4 • Troubleshooting

This chapter is a guide to troubleshooting routine problems that may occur while operating the ED40 Electrochemical Detector. Turn to the section of this chapter that best describes the operating problem. There, the possible causes of the problem are listed in order of probability, along with the recommended courses of action. For additional help, refer to the description of the ED40 diagnostic screens in Appendix C. If unable to eliminate a problem on your own, notify your Dionex office.

4.1 No Detector Response

- **Cell is off**

  Turn on the cell (from the **MAIN** or **DETAIL** screen).

- **Analog output range set too high; although the display indicates a response, no recorder response observed**

  Select a more sensitive analog output range.

- **Wrong full-scale output (or no full-scale output) selected**

  Select 0.01, 0.10, or 1 volt full-scale.

- **No flow from pump**

  Check the pressure reading on the pump to verify that the pump is on.

- **Detector offset out of range**

  Press **Offset** on the ED40 front panel.

4.2 Low Detector Output

- **Analog output range set too high; although the display indicates a response, no recorder response observed**

  Select a more sensitive analog output range.

- **Insufficient sample injected**

  Increase the injection size or concentration.

- **(Conductivity mode)—Cell out of calibration**

  Recalibrate the conductivity cell (see Section 5.4).

- **(DC Amperometry and Integrated Amperometry modes)—Working electrode fouled**

  Polish the working electrode (see Section 5.5).

4.3 High Detector Output

- **Auto offset not activated recently**

  Press **Offset** on the ED40 front panel before making an injection.

- **(Conductivity mode)—Regenerant fails to suppress background**

  Use a higher regenerant flow rate.

- **(Integrated Amperometry mode)—Excessive number of integration periods**

  Integration periods were not properly deleted during editing; delete any unnecessary integration periods now.

- **(DC Amperometry and Integrated Amperometry modes)—Amperometry cell working electrode shorted to counterelectrode**

  Polish the working electrode (see Section 5.5). Remove any precipitate on the counterelectrode by cleaning the spot directly opposite the working electrode with a wet paper towel and

coarse polishing compound (P/N 036319).

4.4 Noisy or Drifting Baseline

- **Flow system leak ahead of cell; erratic baseline**

  Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace all liquid line connections. If the connections are made with ferrule fittings, first refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

- **Pump not properly primed**

  Prime the pump as instructed in the pump manual.

- **Rapid changes in ambient temperature**

  Direct heating and air conditioning vents away from the cell.

  Install the cell in an LC30 Chromatography Oven.

- **Insufficient system equilibration following any changes to operating parameters; especially apparent when operating at high sensitivities**

  Allow longer system equilibration before beginning operation.

- **(Conductivity mode)—Air trapped in cell; excessive regular pulses in baseline**

  Remove the trapped air (Section 5.2). To prevent air from becoming trapped in the cell in the future, increase backpressure on the cell by lengthening the waste line.

- **(Conductivity mode)—Inappropriate suppressor operating conditions**

  See the suppressor manual for the correct operating conditions.

- **(Conductivity mode)–Temperature compensation setting not optimized**

  Optimize the selected setting (see Section 3.7).

  Purchase a cell with a DS3 Detection Stabilizer (P/N 044130).

- **(DC Amperometry and Integrated Amperometry modes)—Frequent, random spikes in the baseline**

  The reference electrode is plugged. First, try regenerating the reference electrode frit by soaking the electrode in a solution of 1 M KCl plus 1 M HCl. If spiking continues, replace the electrode (P/N 046333).

- **(DC Amperometry and Integrated Amperometry modes)—Regular baseline oscillation on high-sensitivity ranges**

  Reconnect the short length of titanium tubing to the cell inlet.

- **(DC Amperometry and Integrated Amperometry modes)—Dirty or pitted working electrode**

  Polish the working electrode (see Section 5.5).

- **(Integrated Amperometry mode)—Regular baseline oscillations**

  The water used to prepare the eluent contains trace sugar contaminants. Remake the eluent, using high purity DI water.

4.5 Tailing Peaks

- **(DC Amperometry and Integrated Amperometry modes)—Dirty or pitted working electrode**

  Polish the working electrode (see Section 5.5).

4.6 Amperometry Cell pH Readout Always 7.0

- **Short circuit in pH reference electrode**

  Check connections to the cell pre-amp board.

  Look for salt on the cell pre-amp board.

  Replace the pH reference electrode (P/N 046333).

4.7 Cannot Set Amperometry Cell pH Readout to 7.0

- **Inaccurate calibration buffer**

  Use a pH meter to check the pH of the buffer.

- **Contaminated pH reference electrode**

  Soak the reference electrode in a solution containing 1 M KCl and 1 M HCl.

  Replace the electrode (P/N 046333).

4.8 No Amperometry Cell pH Readout

- **Disconnected or broken pH electrode wire(s)**

  Fix the connections or replace the electrode (P/N 046333).

- **Uncalibrated pH electrode**

  Calibrate the pH reference electrode from the **pH CALIBRATION** screen.

4.9 Discolored pH Reference Electrode

The electrode normally becomes discolored from prolonged use at high pH; this does not impair its correct functioning. The reference electrode is a consumable item which may last from 1 month to 1 year (see Section 4.11).

4.10 Leaking pH Reference Electrode

- **Defective pH reference electrode**

  Replace the pH reference electrode O-ring (see Section 5.6).

4.11 Shift in Ag/AgCl Reference Potential

- **Faulty reference electrode**

  A shift in reference potential causes a shift in the effective potential applied to the working electrode. For example, an applied potential of 0.1 V, using an electrode with a shift of 50 mV, is equivalent to an applied potential of 0.15 V for a new reference electrode with no shift.

  Following the steps below, measure the Ag/AgCl reference electrode potential shift by comparing it to the potential shift of a new electrode. It is convenient to keep a spare reference electrode (P/N 046333) on hand for this purpose.

  1. Turn off the cell.
  2. Remove the reference electrode from the cell body, unplug the electrode, and rinse it in deionized water.
  3. Locate the reference electrode connector (J2) on the cell pre-amp board. Short the two pins of the connector together, using a short piece of wire or a metal clip. **Do not allow the wire or metal clip to contact the cell body or any other conductor.**
  4. Select **CAL** on the **pH CALIBRATION** screen and press **Enter**. When the "pH 7 Calibration Complete" message appears, remove the wire or clip from J2.
  5. Connect the reference electrode being tested to J2 by turning the electrode at right angles and plugging the center connector of the three-connector socket into the pin labeled "pH."
  6. Connect the new reference electrode to J2 by turning the electrode at right angles and plugging the center connector of the three-connector socket into the pin labeled "Ag."
  7. Immerse both reference electrodes in a saturated solution of KCl.

8. When the pH reading has stabilized, calculate the difference in potential between the two reference electrodes, using the following equation:

$$Difference\ (Volts) = (7 - pH\ reading) \times 0.06$$

For most applications, a difference of less than 50 mV is insignificant. Therefore, the electrode can still be considered good if the voltage difference is less than 50 mV.

If the difference is greater than 50 mV, try regenerating the electrode by soaking it in a solution of 1 M KCl plus 1 M HCl. If this does not reduce the potential shift, replace the electrode.

4.12 Liquid Leaks from the DS3

Leaks are detected first by the DS3 leak sensors and eventually by liquid draining from the overflow line (see Figure 2-4).

- **Loose or defective fittings, or overpressurization caused by a restriction**

  Check the waste line for blockage. A particle in the waste line may plug it, causing restriction and/or leaking. If necessary, clear the waste line by reversing the direction of flow.

  Check that the plumbing downstream from the DS3 is clear. A blockage in the plumbing may overpressurize the DS3, causing it to leak.

  Check that the downstream backpressure coils are appropriate for the operating flow rate (see Section B.3.3).

  Following the procedure below, carefully disassemble the DS3 and inspect it to determine the source of the leak. Test and dry before reassembling the DS3.

  1. Turn off the detector power.
  2. Disconnect the cables from the detector.

3. Disconnect the two 10-32 fitting bolts. Be careful not to misplace the ferrule fittings at the end of the tubing.
4. Remove the DS3 from the chromatography module by lifting it upward and then pulling it away from its mounting location. Place the DS3 on the workbench.
5. Open both latches on the DS3 enclosure and carefully lift the DS3 cover, along with its foam insert, exposing the aluminum housing cover.
6. Remove the four Phillips screws holding the housing and the housing cover together. Separate the two pieces. Unplug the interconnecting cable to prevent damage to the small wires.
7. Disconnect the grounding strap. Remove the two cell mounting screws.



Do not attempt to remove the cell fitting bracket; removing the bracket may damage the capillary inlet tubing.

8. Rotate the cell inlet tube fitting counterclockwise, allowing the cell body to back away from the fitting until the threads of the fitting are fully disengaged. **Do not misplace the ferrule.** Check that the end of the inner titanium tubing is flush with the end of the plastic sleeve. If necessary, trim the sleeve slightly to prevent dispersion. Push the tubing into the cell until it bottoms out in the hole; then, hold the tubing in place while tightening the nut.
9. After testing for liquid leaks, dry the DS3 and reassemble it by reversing the procedure described in Steps 1 through 8.

- **Liquid seeping from around cell cables**

  The cell is inoperable and should be returned to Dionex for repair or exchange. Call your Dionex office for assistance.

4.13 DS3 Temperature Inaccurate

- **"DS3 SET POINT XX C READY" message displays continuously**

  The control sensor may be faulty. Remove the cover of the DS3 and measure the temperature of the aluminum housing, using any surface thermometer. The cell sensor can be read on the detector and should be within 1 or 2 degrees of the DS3 set point temperature.

- **"DS3 SET POINT XX C UNDER TMP" or "DS3 SET POINT XX C OVER TMP" message displays continuously**

  Verify that the set temperature is at least 5 °C above or below the external DS3 temperature. Allow 30 to 60 minutes for the DS3 to heat or cool.

  At high flow rates and temperature settings much over ambient, the DS3 requires more time to heat. In extreme cases, such as a cold room and a high DS3 set point, the DS3 may not be able to reach the set point temperature.

- **DS3 does not heat**

  Remove the cover and inspect the DS3 for broken or shorted wires or moisture bridging the control thermistor. If a wire is broken or shorted, replace the wire or call Dionex for assistance. If a leak has caused a short, fix the leak and dry the control sensor.

- **"DS3 SET POINT XX C READY" message displays intermittently**

  Check that the heater transistors are snug and the control sensor has not been pulled out of the heat exchanger plate.

4.14 Conductivity Inaccurate

- **Cell constant reported on CONDUCTIVITY CALIBRATION screen after cell calibration is not 130 to 190 μS**

  The DS3 has not reached its set point temperature. Set the intended operating temperature and wait until 5 minutes after the "NOT READY" message is displayed, or until the reading is stable.

  Make sure the selected temperature compensation setting is 1.6%/°C. Except at 25 °C, an incorrect temperature compensation setting can cause an incorrect reading.

  The 1 mM KCl calibration solution is spoiled, or there was a mixing error. Make fresh solution.

  The electrodes are fouled by grease, precipitate, etc. Flush with a suitable solvent, such as acid, and then flush with 1 mM KCl until the reading is stable.

  There are internal leaks, broken or shorted cell or sensor wires. Check the sensor by comparing the cell temperature readout with the actual DS3 temperature. If necessary, return the cell to Dionex for repair. **Do not remove the cover plate; this will break the hermetic seal and void your warranty.**

- **Cell temperature readout deviates by more than 2 °C from DS3 set temperature**

  This indicates a serious cell or DS3 sensor problem. Call your Dionex office for assistance.

4.15 Faulty DX LAN Communication

- **DX LAN interface incorrectly installed**

  Make sure a BNC tee connector (P/N 921914) is attached to the DX LAN connector on the ED40 rear panel. Connect the DX LAN cable (P/N 960404) to one side of the BNC tee connector. The DX LAN cable **must be RG58U or RG58AU.** Do not use recorder cables; they have the wrong impedance and will interfere with signals on the DX LAN.

  Make sure that the tee connectors at both ends of the network are capped with the terminator plugs (P/N 921034) that are shipped with PeakNet Software (see Section B.3.2).

4.16 Diagnostics

NOTE

Before running the ED40 diagnostics, use the troubleshooting information in this chapter to isolate non-electronic problems.

The ED40 Moduleware includes several diagnostic tests of the electronics. To access these, select the **DIAGNOSTIC MENU** from the **MENU of SCREENS**. All of the diagnostic screens are described in Appendix C.

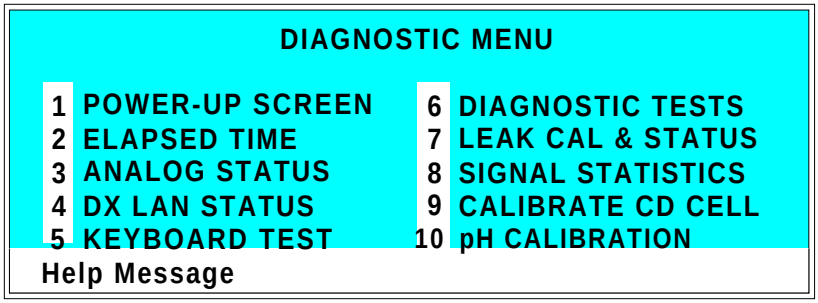


Figure 4-1. Diagnostic Menu Screen

5 • Service

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5.2 Removing Trapped Air from the Conductivity Cell 5-3

5.3 Replacing the Main Power Fuses 5-4

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5.5.1 Polishing with a Pencil Eraser (Gold Electrodes Only) 5-7

5.5.2 Polishing with Polishing Compounds . . 5-8

5.6 Replacing the pH Reference Electrode O-Ring . . 5-9

5 • Service

This chapter describes routine service procedures for the ED40 Electrochemical Detector. Before replacing any parts, refer to the troubleshooting information in Chapter 4 to isolate the cause of the problem. When ordering replacement parts, please include the model number and serial number of the detector.

Substituting non-Dionex parts may impair detector performance, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

NOTE

The ED40 electronic components are not customer-serviceable. Any repairs involving the electronics must be performed by Dionex.

5.1 Eliminating Liquid Leaks

The PEEK version of the ED40 is plumbed with 1.60-mm (1/16-in) PEEK tubing, Dionex ferrule fittings (P/N 043276), and 10-32 fitting bolts (P/N 043275). For tightening requirements, see *Installation of Dionex Ferrule Fittings*.

5.2 Removing Trapped Air from the Conductivity Cell

Air bubbles in the cell can cause regular pulsations of the baseline, random noise, and low readings. Air may have been introduced in the columns during installation, or may result from outgassing of the mobile phase. Connecting a backpressure line to the cell applies enough backpressure to shrink bubbles, thereby allowing them to pass more easily through the cell.

1. Connect a piece of 0.25-mm (0.010-in) ID tubing (P/N 042690), with fittings on both ends, to the cell outlet. Make the line 1 m long for a flow rate of 1.0 mL/min, 2 m for 0.5 mL/min, etc.

2. Use tubing with a minimum ID of 1 mm (0.04 in) the rest of the way to the waste container. Use a union (P/N 042627) to connect the backpressure line to the waste line.

IMPORTANT

Make sure the backpressure does not exceed 0.20 mPa (30 psi). Avoid large flow rate increases that would result in more than 1.4 mPa (200 psi).

5.3 Replacing the Main Power Fuses

1. Turn off the ED40 main power switch.

WARNING

WARNING—HIGH VOLTAGE. Disconnect the main power cord from the rear panel of the ED40, and its source.

2. The fuse holder is in the main power receptacle on the ED40 rear panel (see Figure 5-1). A recessed lock is located on each side of the fuse holder. Using a small screwdriver (or your fingernails), push each lock toward the center. When the locks are released, the fuse holder pops out about 0.16 cm (1/16 in). Pull the fuse holder straight out of its compartment.

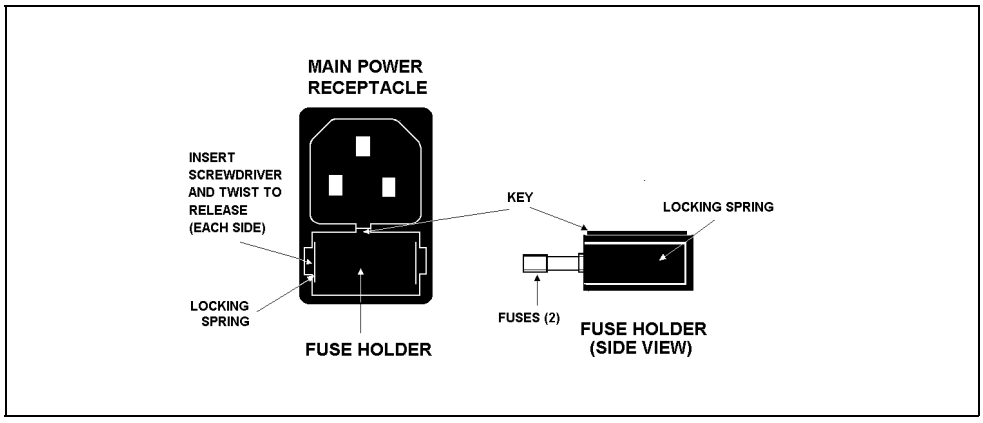


Figure 5-1. Main Power Receptacle

3. The holder contains two fuses. Replace these with new 3.15 amp fast-blow IEC127 fuses (P/N 954745). Replace both fuses, even though only one is open; because the other fuse has been stressed, it may fail even under normal operation.
4. Reinsert the fuse holder into its compartment. The fuse holder is keyed to fit only in its proper orientation. Apply enough pressure evenly against the holder to engage the two locks. When both locks are engaged, the holder is flush against the panel.
5. Reconnect the main power cord and turn on the power.

5.4 Calibrating the Conductivity Cell

Before shipment from the factory, each conductivity cell is calibrated and the cell calibration constant is recorded on a tag attached to the cell cable. The calibration constant is stored in permanent detector memory, for use when calculating the measured conductivity. The calibration constant normally remains unchanged unless the cell is damaged.

To check the value entered in memory, select **CALIBRATE CD CELL** from the **DIAGNOSTIC MENU** to display the **CALIBRATE CONDUCTIVITY CELL** screen. If the on-screen cell calibration constant is not the same as the value recorded on the tag, do one of the following:

- Enter a new value, regardless of whatever is in the cell, **or**,
- Calibrate the cell at 147.00 µS/cm. Calibrating the cell automatically enters a new cell calibration constant value into detector memory.

Calibrate the cell as follows:

1. Disconnect the pump output line from the LC10 Chromatography Organizer, LC20 Chromatography Enclosure, or LC30 Chromatography Oven.
2. Connect the pump output directly to the inlet of the DS3 or the shielded cell.

3. Pump 0.001 M KCl calibration solution through the cell. Conductivity is slightly flow-rate sensitive, so select the flow rate used in the majority of your applications.
4. Set **TEMP COMP** on the **DETAIL** screen to 1.7%.
5. If using a DS3, set **DS3 SETPOINT** on the **DETAIL** screen to the intended operating point. Wait until the "DS3 READY" message appears.
6. Wait until the conductivity reading stabilizes (within 0.1 µS), and then calibrate the cell.
7. After calibration, the conductivity reading should be exactly 147.00 µS/cm. A new value for the cell constant will be displayed on-screen and entered in memory.
8. Flush the KCl solution from the system by pumping deionized water through the DS3 or cell lines. When the conductivity reading drops to near zero, stop the pump.
9. Disconnect the pump from the DS3 or cell.
10. Reconnect the pump to the chromatography module.
11. Reconnect the liquid line from the suppressor outlet to the cell inlet.
12. Reset **TEMP COMP** to the optimal value for the eluent.
13. Set the pump to a flow rate that is safe for the system in use.

5.5 Polishing the Working Electrodes

Polish all working electrodes with fine polishing compound before initial installation in the amperometry cell. Later, if you observe high background current, a decrease in sensitivity, or other degradation in detector output, polish the working electrode to clean it and restore performance.

- Section 5.5.1 describes a mild cleaning procedure for ***gold working electrodes only***. If this procedure is not effective, go on to Section 5.5.2.
- Section 5.5.2 describes a more aggressive cleaning procedure, in which the electrode is cleaned with fine and coarse polishing compounds. Since the cell may need at least 12 hours to re-equilibrate after polishing, perform this procedure only when essential. This procedure applies to all ED40 working electrodes.
- To polish electrodes with fine polishing compound before initial installation in the amperometry cell, follow the instructions in Section 5.5.2.

5.5.1 Polishing with a Pencil Eraser (Gold Electrodes Only)

1. Unscrew the wing nuts holding the working electrode to the cell body, and carefully separate the parts. Handle the cell gasket and the inside surfaces of the cell carefully, to prevent scratches which may subsequently cause leaks.
2. Locate the eraser provided in the ED40 Ship Kit. Rub the surface of the electrode with the clean eraser until all traces of discoloration have been removed from the electrode.
3. Rinse the electrode thoroughly with water or methanol. Wipe the surface with tissue to remove remaining traces of eraser.

4. Reassemble the cell. Reapply the potential to the electrode and equilibrate for 1 to 2 hours while eluent or water flows slowly through the cell.

5.5.2 Polishing with Polishing Compounds

1. Unscrew the wing nuts holding the working electrode to the cell body, and carefully separate the parts. Handle the cell gasket and the inside surfaces of the cell carefully, to prevent scratches which may subsequently cause leaks.
2. Locate the polishing kit (P/N 036313) shipped with the amperometry cell. The kit contains two polishing pads (P/N 036321), a bottle of fine polishing compound (P/N 036318), and a bottle of coarse polishing compound (P/N 036319).
3. Using indelible ink, mark the plastic side of each pad to designate whether it is for use with the coarse or fine polishing compound. Also mark the fine pad to designate for which working electrode type it is to be used.

NOTE

Do not use the same fine polishing pad to polish more than one type of working electrode; this can contaminate the electrode surface with microparticles from the other working electrodes. A separate polishing pad is shipped with each type of working electrode. Using indelible ink, mark each pad to indicate the working electrode with which it is used.

4. Moisten the plastic side of the fine polishing pad slightly with water and place it on a smooth, flat surface.
5. Sprinkle about one-half gram of fine polishing compound in the center of the suede side of the polishing pad. Add enough deionized water to make a thick paste.

6. Using the working electrode block, spread the paste evenly over the pad. Then, applying firm pressure in a figure eight motion, polish the surface of the electrode block for about one minute. If the pad dries out while polishing, add water sparingly.

7. Use deionized water to rinse off all traces of polishing compound from the electrode block. An ultrasonic cleaner is effective for thoroughly cleaning the electrode block. Carefully wipe the surface of the block with a soft damp cloth or damp paper towel. Inspect the surface of the working electrode to make sure that it is clean.

NOTE

Do not rinse the polishing compound from the pads after polishing is complete. The polishing pads are reusable. After the initial use, add only enough polishing compound to maintain the coating on the pad.

8. Reassemble the cell. Reapply the electrode potential and resume eluent flow. The baseline will drift for more than 1 hour as the cell re-equilibrates. Peak area values may require up to 12 hours to stabilize.

5.6 Replacing the pH Reference Electrode O-Ring

A defective pH reference electrode O-ring can cause liquid leaks from the pH reference electrode or compression nut area when the cell operating pressure is below 700 kPa (100 psi).

1. Remove the pH reference electrode from the reference electrode cavity.

2. With a sharp instrument (such as a pin), stick the old reference electrode O-ring and pull it out of the reference electrode cavity. **Be careful not to scratch the cell body.** Do not lose the stop ring (P/N 045967) located below the O-ring. The stop ring prevents the pH reference electrode from bottoming out and thereby damaging the electrode.

3. Verify that the stop ring is in place at the bottom of the cavity. Using a blunt instrument, push the new O-ring (P/N 048410) into the groove within the cavity. Verify that the new O-ring is seated properly.

NOTE

As removing the O-ring destroys it, always replace the old O-ring with a new one.

4. Reinstall the pH reference electrode in the reference electrode cavity.

6 • Electrochemical Detection

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6 • Electrochemical Detection

This chapter describes the fundamentals of electrochemical detection methods to help you take full advantage of the powerful detection capability of the ED40 Electrochemical Detector to solve problems in analytical chemistry.

6.1 Electrochemical Detection Modes

The ED40 Electrochemical Detector can operate in three different detection modes:

- In the Conductivity mode, current conducted by ions in solution in an electric field is measured.
- In DC Amperometry, a constant voltage is applied to the working electrode in the amperometry cell and the resulting current is the detector output.
- Integrated Amperometry is similar to DC Amperometry in that the same cell and associated circuitry are used. After the initial stages of signal processing, the cell current is integrated and the resulting charge is the detector output reported for each integration period. A user-programmed waveform, or repetitive series of potentials, is applied to the cell, and the same waveform program sets the start and finish of the integration period. The program is entered from the **WAVEFORM** screen.

An important distinction between conductivity and amperometric detection is that no electron transfer reactions occur during conductivity detection. During DC and integrated amperometric detection, electrons are actually transferred between the electrode and the analyte molecules. This concept is illustrated in Figure 6-1.

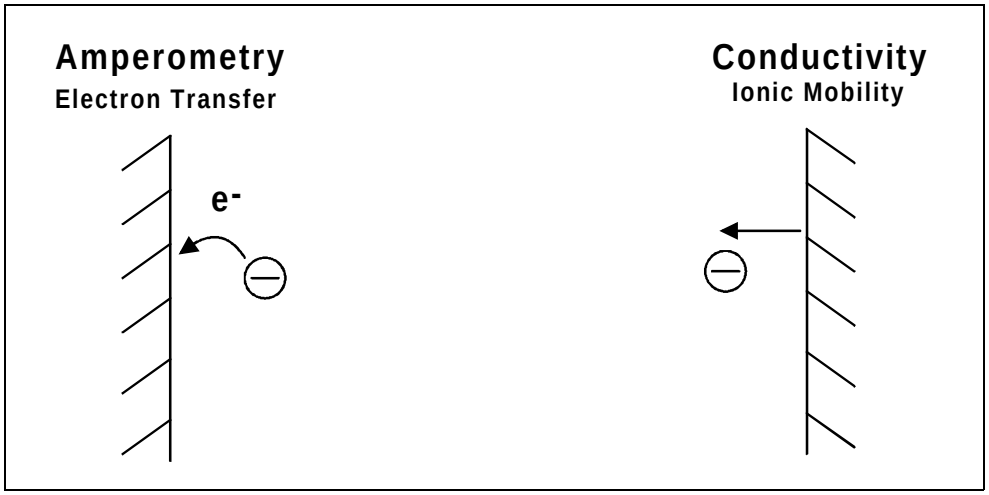


Figure 6-1. Electrochemical Properties Measured

Although, in theory, all three detection modes could employ the same detector cell, a cell optimized for conductivity is not appropriate for amperometry, and vice versa. For this reason, the ED40 uses two detector cells—one for conductivity and another for amperometry.

6.2 Equivalent Circuits

The ED40 accomplishes all three detection modes by measuring current resulting from the application of potential (voltage) across electrodes in a flow-through cell. The three forms of electrochemical detection differ in the manner used to apply the potential and measure the current.

To understand how the ED40 can perform three different detection modes simply by applying voltage and measuring current, it is useful to model electrochemical cells in the equivalent circuit shown in Figure 6-2.

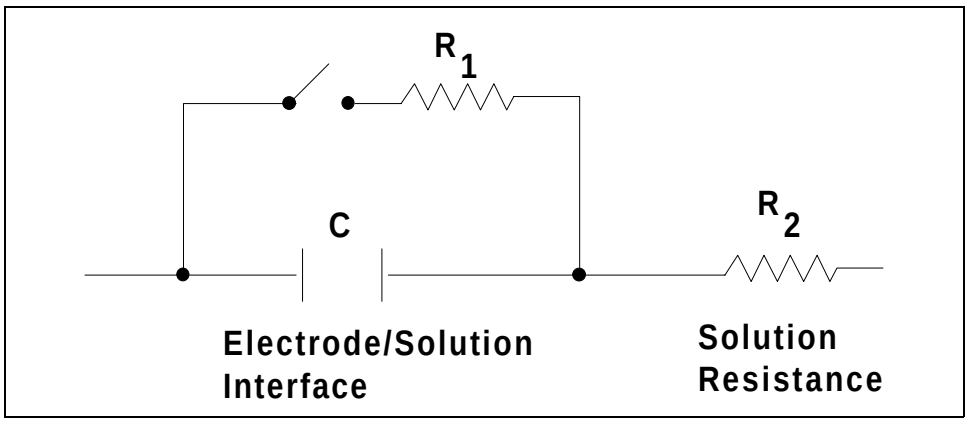


Figure 6-2. Equivalent Circuit (Electrode in Solution)

This circuit is designed to mimic the electrochemical behavior of conductivity and amperometry cells. R_2 represents the resistance to the migration of ions caused by the electrical field applied between the electrodes, measured during conductivity detection. The interface between the solution and the electrode acts as a capacitor, C, in the circuit. (Only one interface is shown in the figure.) When the applied potential is high enough, electrons transfer to or from molecules in solution. This is represented by closing the switch. The resulting current measured during amperometry is controlled mostly by the rate of diffusion of analyte to the electrode surface. A resistance (R_1) may be defined which is inversely proportional to the rate of analyte diffusion to the electrode surface.

6.2.1 Conductivity

The quantity we wish to measure during conductivity detection is the inverse of the solution resistance, $\boldsymbol{R_2}$. It can only be measured when all of the applied potential develops across $\boldsymbol{R_2}$. This is accomplished by applying a high frequency alternating potential across the electrodes. When an alternating potential of several kHz is applied across a capacitor, its resistance to current flow drops to near zero. With zero resistance (or, more accurately, reactance) at the

interface between the electrode and the solution, all the applied potential develops across the solution resistance, $\boldsymbol{R_2}$. Since we know the applied potential and the current is measured, $\boldsymbol{R_2}$ is calculated from Ohm's law:

$$R = \frac{E}{i}$$

The inverse of the solution resistance $\boldsymbol{R_2}$ is the conductance, *G*. The measured conductance is then corrected by the conductivity cell constant *K* to produce the conductance which would be measured in a cell containing electrodes of 1 cm^2 surface area held 1 cm apart. This quantity is the *conductivity*, κ, and the units are siemens per cm, S/cm. Stated as equations:

$$G = \frac{1}{R} = \frac{i}{E}$$

$$\kappa = K \bullet G$$

6.2.2 DC, Integrated, and Pulsed Amperometry

The addition of a supporting electrolyte in high concentration greatly lowers the solution resistance, $\boldsymbol{R_2}$. (An *electrolyte* is a solution containing dissolved ions.) Acids, bases, or salts in concentrations of 1 to 100 mM are generally used. The addition of a supporting electrolyte plus the application of a DC or very low frequency alternating potential waveform causes nearly all the applied potential to develop across the interface between the electrode and the solution. The potential difference between the electrode and the solution can easily be made high enough to cause electron transfer reactions to occur (closing the switch), oxidizing or reducing species in solution. During an oxidation reaction, electrons are transferred from the analyte to the electrode. During a reduction reaction, the reverse occurs: electrons leave the electrode and enter the analyte.

During DC Amperometry, the current is reported directly to the recording device. With the other modes (Conductivity, Integrated Amperometry, and Pulsed Amperometry) the detector converts the measured current into other, proportional, units. Table 6-1 lists the detection modes and the three properties of the solution measured.

Mode	Quantity Measured	Units
Conductivity	Solution conductivity	Siemens (S)
DC Amperometry	Current caused by reduction or oxidation of solution species	Amperes (A)
Integrated and Pulsed Amperometry	Integrated current (charge) from reduction or oxidation of solution species during a portion of a repeating potential vs. time waveform	Coulombs (C)

Table 6-1. Electrochemical Properties Measured

In summary, the detection method is determined by the applied potential waveform, the presence or absence of a supporting electrolyte, and the manner in which the current is measured.

6.3 Conductivity Detection

6.3.1 Conductivity of Solutions

The conductivity of a dilute solution is the sum of the individual contributions to the conductivity of all the ions in the solution multiplied by their concentrations (i.e., conductivity is directly proportional to concentration). This is called Kohlraush's law of independent migration. It states that each ion carries its portion of the total conductivity without being affected by any of the other ions in solution.

Stated as an equation:

$$\kappa = \frac{\sum_i \lambda_i^o c_i}{1000}$$

where:

- κ is the measured conductivity in S/cm.
- c_i is the concentration of the ions in equivalents/L. (Equivalents/L equals moles/L times the charge on the ion.)

The *ionic limiting equivalent conductivity*, λ_i^o, is specific for each ion. It is the conductivity of the ion divided by the concentration and extrapolated to infinite dilution. Table 6-2 lists the limiting equivalent conductivities for a number of organic and inorganic ions. The unit for λ_i^o is $S \cdot cm^2$/ equivalent.

Anions	λ_i^o	Cations	λ_i^o
OH^-	198	H^+	350
F^-	54	Li^+	39
Cl^-	76	Na^+	50
Br^-	78	K^+	74
I^-	77	NH_4^+	73
NO_3^-	71	Mg^{2+}	53
HCO_3^-	45	Ca^{2+}	60
SO_4^{2-}	80	Sr^{2+}	59
$Acetate^-$	41	$CH_3NH_3^+$	58
$Benzoate^-$	32	$N(CH_3CH_2)_4^+$	33

Table 6-2. Limiting Equivalent Conductivities at 25 °C

Values of λ_i^o from this table can be used to calculate conductivities of solutions containing ions. For example, the limiting equivalent conductivity for NaCl at 25 °C is 126.5. This is the sum of the ionic limiting equivalent conductivity for Na^+, which is 50.1, plus that of Cl^-, 76.4. A 0.1 mM solution of NaCl at 25 °C has a conductivity of 0.1 × 126.5, or 12.65 µS/cm. The conductivity of a solution of 0.1 mM NaCl plus 0.1 mM Na_2SO_4 would be calculated as follows:

Ions		Charge		Conc.		λ_i^o		µS/cm	
3	×	1	ö	0.1	×	50.1	=	15.0	(Na^+)
1	×	1	ö	0.1	×	76.0	=	7.6	(Cl^-)
1	×	2	ö	0.1	×	80.0	=	16.0	(SO_4^{2-})
						Total		38.6	

So far, only dilute solutions have been discussed. As concentration increases, the direct proportionality between conductivity and concentration is lost. However, at the analyte concentrations normally encountered in ion chromatography (below 1 mM), conductivity is generally proportional to concentration. For example, the equivalent conductivity at 25 °C of KCl at infinite dilution is 149.9. At 1 mM it is 146.9, a decrease of only 2%. However, the conductivity of an eluting analyte is not necessarily directly proportional to concentration, because ionic components of the mobile phase may be contained in the eluting volume.

If the electrolyte is a weak electrolyte, such as an acid or base with only partial dissociation, then c_i must be replaced by the concentration of the dissociated ions only, since only they contribute to conductivity. For acids and bases, the pK values and the solution pH can be used to calculate the extent of dissociation.

6.3.2 Effect of Hydration Sphere and Solvent on Conductivity

The limiting equivalent conductivity of an ion, λ_i^o , is a measure of the mobility of the ion. Ionic mobility is greatly affected by the properties of the ion in the solvent. Ions with large hydration spheres are less mobile, and therefore less conductive, than ions with small hydration spheres. This explains why λ_i^o for extensively hydrated fluoride (55.4) is lower than λ_i^o for chloride (76.4), which is less hydrated. The viscosity of the solvent also affects ionic mobility—ions are more mobile in solvents of lower viscosity.

It is not necessary to know values such as hydration sphere and viscosity, since quantitative analysis is performed by comparing the conductivity of the analyte in the sample to the conductivity of the same analyte in a standard(s). Even when a solvent gradient is used, the composition of the solvent during the elution of the analyte is the same in both the sample and the standard.

6.3.3 Effect of Temperature on Conductivity

Ionic mobility, and therefore conductivity, are greatly affected by temperature. The conductivity of an aqueous solution has been found experimentally to rise about 2% per °C. (This dependence is described in a complex equation developed by Onsager.) Therefore, it is necessary to hold the mobile phase temperature as constant as possible to maintain a stable baseline. When the conductivity cell is housed in the Dionex DS3 Detection Stabilizer, the DS3 maintains the cell at a set temperature. In addition, the ED40 corrects the measured conductivity to that which would be measured at 25 °C by measuring the cell temperature with a thermistor and multiplying the conductivity by a temperature-dependent constant. This constant, the *temperature compensation factor*, is expressed in units of %/°C. When the DS3 is not used, setting an accurate temperature compensation factor minimizes baseline drift caused by fluctuations in ambient temperature.

6.3.4 Species Detected by Conductivity

Conductivity detection is typically selected for species that are ionic when they enter the detector cell, especially those with weak UV absorbance. This includes both organic and inorganic ions. Conductivity detection is best suited to anions and cations of strong acids and bases, such as chloride, sulfate, trifluoroacetate, sodium, and potassium. Ions of weaker acids and bases are detected, provided that the mobile phase pH is chosen to maximize analyte dissociation. (When a suppressor is used, the mobile phase pH which determines whether an ion will be detected is the pH after suppression.)

Anions

Sensitivity is best for anions with pKa values below 6. As analyte ionization (dissociation) decreases, so does sensitivity. Anions with pKa values above 7 can be detected under certain conditions, but signal-to-noise ratios are generally poorer. Fortunately, all organic acids with either carboxylate, sulfonate, or phosphonate functional groups have pKa's below 4.75, so conductivity is the preferred detection method for these species. Common inorganic strong acid anions include chloride, nitrate, phosphate, and sulfate.

Cations

Inorganic cations detected include the alkali metals and alkaline earths. Most transition metal cations are not detected, since they either hydrolyze water to form anions or precipitate in the suppressor. These metals are usually detected by visible wavelength absorbance following post-column reaction with a chelating indicator. Nearly all organic cations are amines. Aliphatic amines have pKa's around 10 and are easily detected. Aromatic and heterocyclic amines have pKa's between 2 and 7, too low to be detected by suppressed conductivity following cation exchange separation. Although nonsuppressed detection can be used for these species, sensitivity is generally poor. These amines can be detected by UV absorbance or by DC or Integrated Amperometry.

Zwitterions

Amphoteric, or zwitterionic, molecules contain both cationic and anionic functional groups and are difficult or impossible to detect by conductivity. Amino acids, for example, contain both ammonium cationic functional groups and carboxylic acid anionic functional groups. With suppressed conductivity detection, they are generally removed by the suppressor and do not reach the detector. They can be detected by nonsuppressed conductivity detection if the mobile phase pH

is at a value which results in a net charge on the molecule. Since most zwitterions contain primary, secondary, or tertiary amine functional groups, Pulsed or Integrated Amperometry is often the best detection method. UV absorbance can be used for aromatic zwitterions.

6.3.5 Chemical Suppression

Species detected by conductivity are by their nature ionic, so ion exchange and ion-pair chromatography are by far the most commonly used separation methods. These methods require mobile phases containing strong electrolytes. This causes a problem: how to detect the ionic analytes without the detector being overwhelmed by the ions in the mobile phase. The best solution is to neutralize the mobile phase in a suppressor.

Figure 6-3 illustrates the suppression mechanism occurring inside a Dionex Anion Self-Regenerating Suppressor (ASRS) using sodium hydroxide as the mobile phase for anion exchange separation.

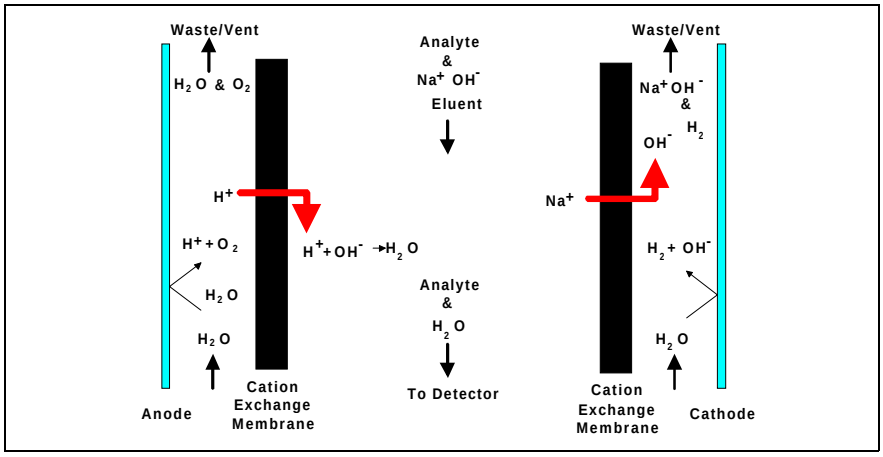


Figure 6-3. Anion Self-Regenerating Suppressor

Analyte anions elute from the column with sodium counterions. Two electrodes, one beside each membrane (on the side opposite the mobile phase), hydrolyze water to hydrogen and hydroxide ions. Hydrogen ions diffuse across the membrane next to the anode, neutralizing the mobile phase hydroxide to water, while sodium ions from the mobile phase diffuse across the other membrane, providing counterions to the hydroxide being generated at the cathode. In effect, sodium hydroxide from the mobile phase is transferred across the membrane and does not reach the detector. The resulting mobile phase background conductivity is near zero, considerably lower than before suppression. Also, the counterion to the anion analytes is now a hydrogen ion, which has a conductivity seven times higher than the original sodium counterion. Since both the anion analyte and the cation counterion produce the detector response, the response is increased. The suppressor lowers the background conductivity (and therefore the baseline noise and drift) and increases the signal. Suppression can also be accomplished without water electrolysis by pumping a dilute sulfuric acid solution (the regenerant) through the suppressor on the side of the membranes opposite the mobile phase.

For ion chromatography of cations, the suppressor membranes are anion exchange polymers. These allow anions to pass freely, but exclude cations. Dilute acids such as methanesulfonic acid are used in the mobile phase. In the Dionex Cation Self-Regenerating Suppressor (CSRS), methanesulfonate counterions are replaced by hydroxide generated by the electrolysis of water. This neutralizes the acidic mobile phase and provides the highly conductive hydroxide counterion to the analyte cations.

Use of a suppressor typically increases signal-to-noise ratios about one order of magnitude for strong acid or base ions. The improvement is somewhat less for ions of weak acids or bases, due to decreased ionization at the neutral pH of the mobile phase following suppression. Nonetheless, the benefits

from reducing the background, and therefore the noise, almost always yield a net improvement in signal-to-noise ratio compared to nonsuppressed conductivity detection.

Suppressors provide several important advantages. The first three listed below are a direct result of the increased signal-to-noise ratio.

- Lower detection limits.
- A possible increase in the dilution of dirty samples, thus extending column life.
- Wider dynamic range.
- More concentrated mobile phases can be used, providing a greater range of elution control and permitting larger sample concentrations or volumes.
- Gradient elution capability.
- Faster equilibration time.
- Elimination of interference from counterions.
- Elimination of system peaks.

6.3.6 Mobile Phases for Conductivity Detection

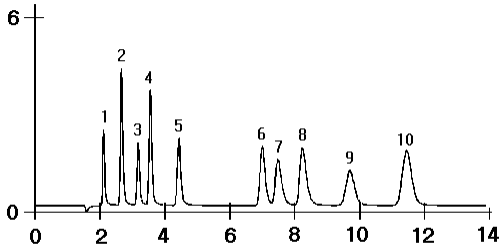
When choosing a mobile phase, the constraints placed by the separation and detection methods must be considered. To obtain optimum separation, the elution strength of the mobile phase, the separation efficiency, and the resolution of the analytes of interest are the major criteria. For conductivity detection, the criteria for a good mobile phase are the relative conductivity response of the analytes and the magnitude of the background.

Mobile phases can be wholly aqueous, containing only water and a strong electrolyte. Or, if an organic solvent-compatible column such as a Dionex OmniPac™ or MPIC column is used, typical reversed-phase solvents such as methanol or acetonitrile can be used. Organic solvents are essential components of ion-pair mobile phases and provide important selectivity control during ion exchange separations. Since these solvents are nonconducting, they do not interfere with conductivity detection.

Anion Exchange

When a suppressor is used, the ionic components of the mobile phase must be such that they are removed or converted to weakly conducting compounds by the suppressor. Sodium salts of weak acids are used because they are converted to the neutral free acid form in the suppressor. The higher the pKa of the acid, the lower the background conductivity following suppression. Weak acids with pKa's above 6 can be used for isocratic separations. For gradient elution, pKa's should be above 8 to minimize baseline shift during the gradient.

Sodium hydroxide solutions make excellent mobile phases for anion exchange because hydroxide is neutralized in the suppressor to water (the free acid form of hydroxide). This is the case regardless of its concentration, making it most useful for gradient elution. Another common mobile phase is a carbonate/bicarbonate buffer. It is suppressed to carbonic acid (pKa = 6.2), which has conductivity low enough for isocratic elution, but not for gradient. Carbonate/bicarbonate buffers are easily prepared and are routinely used for isocratic separations of inorganic anions, as shown in Figure 6-4.



Column: IonPac AS12A and AG12A
Eluent: 2.7 mM Sodium carbonate
0.3 mM Sodium bicarbonate
Flow Rate: 1.5 mL/min
Detection: Suppressed conductivity
Peaks:

1 Fluoride 3 mg/L	6 Bromide 20 mg/L
2 Chlorite 20 mg/L	7 Chlorate 20 mg/L
3 Bromate 20 mg/L	8 Nitrate 20 mg/L
4 Chloride 6 mg/L	9 Orthophosphate 30 mg/L
5 Nitrite 10 mg/L	10 Sulfate 20 mg/L

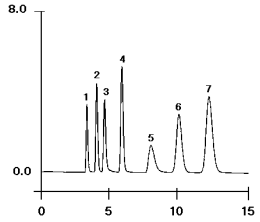
Figure 6-4. Anion Standard

Other mobile phases for suppressed anion chromatography include the sodium salts of boric acid (borax, tetraborate, pKa = 9.2) and p-cyanophenol (pKa = 8.0). Borate forms weak bonds with hydroxy-organic acids, producing changes in selectivity compared to hydroxide. Because of its high pKa, background conductivity following suppression is very low, making it useful for gradient elution. p-Cyanophenol is a powerful monovalent displacer, useful for eluting strongly retained hydrophobic monovalent anions such as iodide and thiocyanate.

In nonsuppressed (sometimes called single-column) ion chromatography, the ionic components in the mobile phase are chosen so that their conductivity is as different as possible from the analyte's. For anion IC, large ions with low equivalent conductivity may be used (e.g., benzoate and phthalate). These produce backgrounds of moderately high conductivity. A buffer composed of a gluconate/borate solution produces somewhat lower backgrounds. Sensitivity and baseline noise are acceptable for analyzing samples containing high concentrations of analytes. However, the advantages provided by a suppressor are not realized.

Cation Exchange

Millimolar concentrations of dilute strong acids, often mixed with organic solvents, are good choices for cation exchange chromatography of both monovalent and divalent cations. Methanesulfonic acid (MSA) is commonly used since it is compatible with the Cation Self-Regenerating Suppressor (CSRS). A large number of amines and inorganic cations can be eluted in a single run using a gradient of MSA and acetonitrile (see Figure 6-5).



Column:	IonPac CS12A (no guard)
Eluent:	20 mN Sulfuric acid
Flow Rate:	1.0 mL/min
Inj. Volume:	25 µL
Detection:	Suppressed conductivity, CSRS AutoSuppression
Peaks:	
1 Lithium	0.5 mg/L (ppm)
2 Sodium	2.0
3 Ammonium	2.5
4 Potassium	5.0
5 Morpholine	25.0
6 Magnesium	2.5
7 Calcium	5.0

Figure 6-5. Amines and Inorganic Cations

Some columns require a stronger displacing ion than hydrogen to elute more strongly retained divalent cations. The zwitterion 2,3-diaminopropionic acid (DAP), mixed with a strong acid to protonate the DAP, is a good choice.

The best mobile phases for nonsuppressed cation IC are dilute strong acids such as 1 mM nitric acid. Backgrounds are very high (around 1 μS), so signal-to-noise ratios are not good enough for low level work. Since the conductivity of the hydrogen ion is greater than that of all other cations, elution of analytes causes decreases in conductivity and eluting analytes produce dips instead of peaks.

Ion-Pair

Mobile phases for ion-pair chromatography contain mixes of aqueous and organic solvent solutions with hydrophobic ion-pair reagents as additives. Commonly used reagents are quaternary ammonium salts for anion separations and long-chain sulfonates for cation separations. For suppressed conductivity detection, these reagents are easily used provided that the counterions are either hydroxide (anion-ion pair) or hydrogen ion (cation-ion pair). These are marketed by Dionex as IonSep reagents. The reagents are purified solutions of quaternary ammonium hydroxide solutions and sulfonic acid solutions.

6.4 Amperometric Detection

The two main forms of amperometric detection (DC and integrated amperometry) measure the current, or charge, resulting from the oxidation or reduction of analyte molecules at the surface of a working electrode. During oxidation reactions, electrons are transferred from molecules of an electroactive analyte to the working electrode in the amperometry cell. Conversely, during reduction reactions, electrons are transferred from the working electrode to the analyte. For analytes which can be oxidized or reduced, detection is usually sensitive and highly selective. Since many potentially interfering species cannot be oxidized or reduced, they are not detected. When a single constant potential is applied to the working electrode, the detection mode is DC Amperometry. Integrated and Pulsed Amperometry employ a repeating sequence of potentials.

6.4.1 Voltammetry

The determination of the optimum potentials to use in amperometry begins with an electrochemical technique called voltammetry. Voltammetry is performed by continuously moving a solution containing the analyte and supporting electrolyte past the surface of the working electrode. This can be accomplished in a standard beaker-type cell employing a rotated disk electrode or, as in the ED40 amperometry cell, with the mobile phase and analyte flowing through the cell. The current that results from oxidation or reduction reactions is measured and plotted vs. applied potential, which is scanned between preset limits.

If the solution is not flowing and the potential is first scanned in one direction and then reversed so that the potential at the end of the scan is the same as at the beginning, the technique is called cyclic voltammetry. You can perform cyclic voltammetry with the ED40 by programming a triangle wave in the Voltammetry mode. The voltammogram is recorded using an X-Y recorder, storage oscilloscope, or computer.

Consider the following generalized electrochemical reaction:

$$A = B + ne^-$$

When the reaction proceeds in the forward direction, species A transfers *n* electrons to the working electrode and is oxidized to species B. The reverse reaction is the reduction of B back to A. The direction of the reaction can be predicted by the Nernst equation:

$$E_{app} = E^o + \frac{0.059}{n} \log\frac{[B]}{[A]}$$

where:

- E_{app} is the applied potential.
- [A] and [B] are the equilibrium concentrations of species A and B *at the surface of the working electrode.*
- E° is the potential at which these two concentrations are equal. (For simplicity, concentrations are used instead of activities.)

Each oxidation/reduction reaction has a characteristic E°. Many chemical reference books contain tables of E°'s for various substances listed as reductions vs. an H_2/H^+ reference electrode. When E_{app} = E°, log [B]/[A] must equal zero; thus [B] = [A]. If E_{app} is set positive of E°, then log [B]/[A] must be greater than zero and therefore [B] > [A]. The reaction proceeds in the forward direction with species A being oxidized to species B. The current will be positive. (Positive currents are called *anodic*.) Conversely, setting E_{app} negative of E° results in the reduction of B to A, generating a negative current (*cathodic*).

Consider a situation in which only species A is present in solution and where E° is +0.4 V vs. an Ag/AgCl reference electrode. Figure 6-6 shows the current as a function of applied potential, the *voltammogram*.

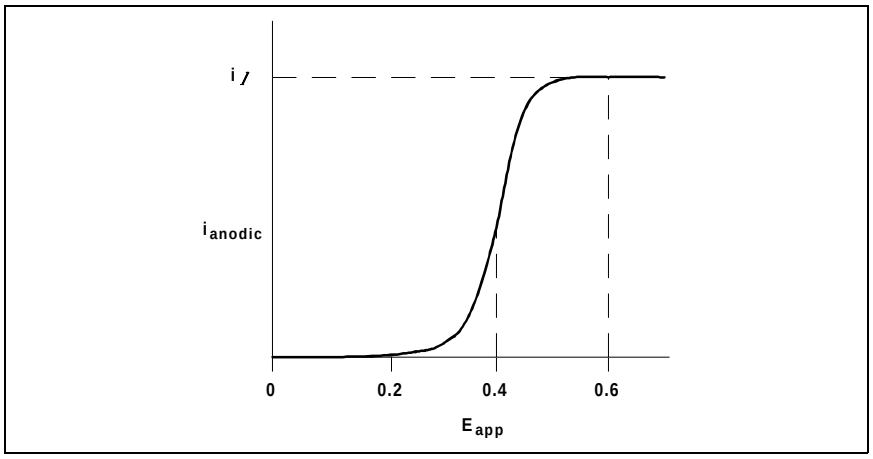


Figure 6-6. Voltammogram

The shape of the voltammogram can be explained by considering the concentration of A at the electrode surface at three different applied potentials. At 0.2 V, E_{app} is negative of E°. The Nernst equation indicates that at 0.2 V, the reduction of B to A is favored. Because the solution contains only substance A, no reaction occurs. As the potential is increased, it approaches a level high enough to cause oxidation of a percentage of analyte molecules, and the current increases. At a higher potential, 100% of the analyte molecules reaching the surface of the working electrode are oxidized and the current is no longer dependent on potential. Since the current is now limited by the rate at which the analyte molecules are transported to the surface of the working electrode, and since this rate is largely dependent on diffusion, this maximum current is called the *diffusion limited current*.

The previous discussion used an example analyte A which could be in equilibrium with its oxidized form B at E°. This is called a *reversible* reaction. More often than not, redox reactions are *irreversible*, and a potential considerably in excess of E° is required to oxidize or reduce the molecule. However, the required potential can still be easily determined using the voltammetric techniques described in Section 6.4.2.

6.4.2 DC Amperometry

Choosing the Applied Potential

The optimum detection potential for DC amperometry is the lowest which will produce a diffusion limited current. In the above example, the optimum potential would be +0.6 V. Increasing the potential beyond this value will increase only the noise and not the signal, and also decrease selectivity by allowing more species to be oxidized. The optimum potential is most easily determined from a plot of peak height vs. applied potential, which is generated using DC amperometry by making multiple injections of analyte and increasing the potential after each injection. A plot created using this method with serotonin as the analyte is shown in Figure 6-7. From this plot, the optimum applied potential should be about 0.7 V.

The current resulting from oxidation or reduction of analyte molecules is dependent on many factors; the most important of these is the analyte concentration. Other factors include temperature, the surface area of the working electrode, and the linear velocity of the flowing stream over the surface of the working electrode. The ED40 cell maximizes linear velocity, thus maximizing signal-to-noise ratio.

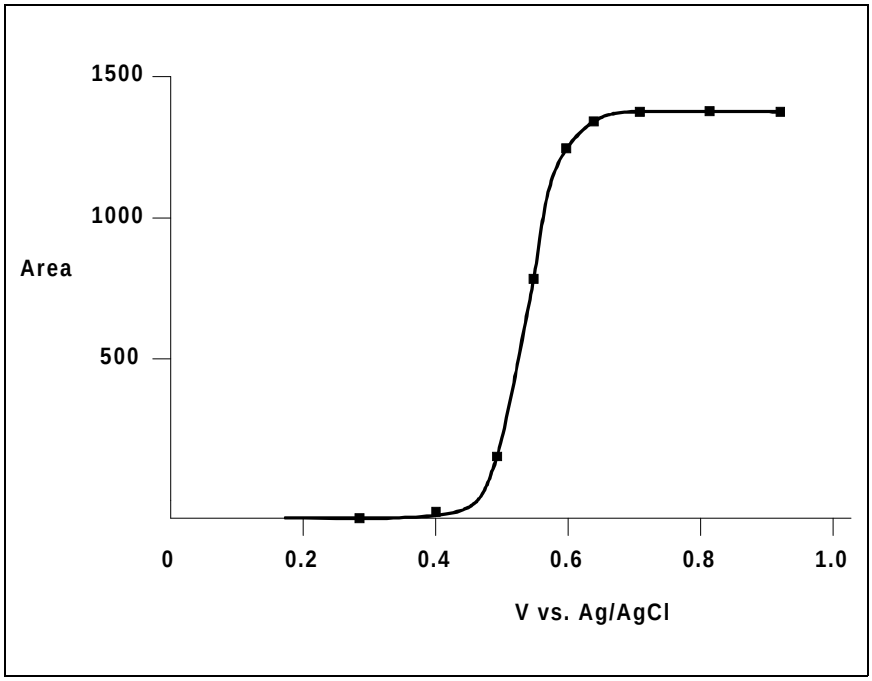


Figure 6-7. Peak Area vs. Potential for Serotonin

Species Detected by DC Amperometry

The major application is the detection of molecules containing phenol or catechol functional groups. Numerous molecules of interest in pharmaceutical or biochemical analyses are in this category. The most important application is the detection of catecholamines such as epinephrine and dopamine and other biogenic neurotransmitters, usually in urine or plasma. An example chromatogram of catecholamines in an alumina extract of urine is shown in Figure 6-8. DHBA is added as an internal standard.

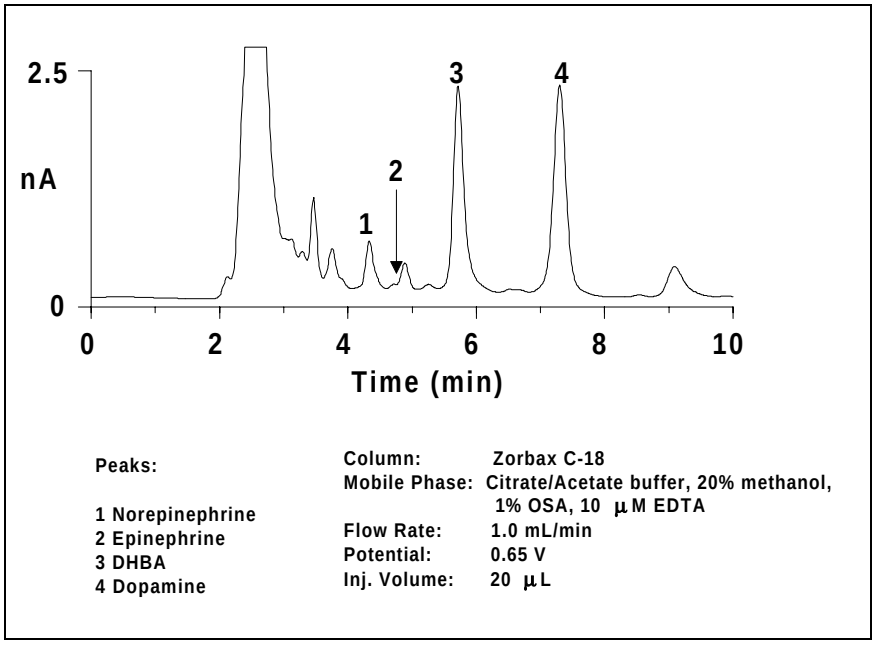


Figure 6-8. Catecholamines in Urine

The analgesic acetaminophen contains a phenol functional group and is easily detected. The amino acid tyrosine also contains a phenol functional group. Not only can this amino acid be detected, but any peptides containing tyrosine also can be detected. Some other detectable organic molecules are aromatic amines, thiols, and sulfides. Sulfonamide antibiotics are detected since they are aromatic amines. In addition to these organic molecules, inorganic anions forming complexes with silver such as cyanide, sulfide, and iodide are detected at a silver electrode.

Table 6-3 lists species most commonly detected by DC Amperometry and the working electrode material used. In addition, species determined by Pulsed Amperometric Detection (PAD) and Integrated Amperometry are listed.

Analyte or Analyte Class	Working Electrode	Method	References*
Alcohols, glycols, and aldehydes	Pt, Au	PAD	26, 30
Aliphatic amines and amino acids	Au, Pt	Int. Amp., PAD	28
Alkanolamines	Au	PAD	25, 29
Aromatic amines	G.C.	DC	13
Aromatic nitro compounds	G.C.	DC	13
Ascorbic acid	Pt	DC	12
Bromide	Ag	DC	19
Carbohydrates	Au	PAD	20-23, 27
Catecholamines and other biogenic amines	G.C.	DC	13-17
Cyanide	Ag	DC	10, 19
Hydroquinones	G.C.	DC	13
Iodide	G.C.	DC	18, 19
Phenols	G.C.	DC	13
Sulfide	Ag	DC	19
Sulfite	Pt	DC	11
Sulfur species	Pt	Int. Amp.	24
Uric acid	G.C.	DC	13

Table 6-3. Analytes Detected by Amperometry

*References are listed in Appendix F.

6.4.3 Pulsed Amperometry

Pulsed amperometry is a specific application of integrated amperometry, described in Section 6.4.4.

The development of pulsed amperometric detection grew from the need to detect carbohydrates. Since most carbohydrates contain no UV chromophore, UV absorbance detection can only be used at very low wavelength. The detection of carbohydrates at 210 nm is insensitive and nonselective. Refractive index detection, also insensitive and nonselective, had been the most commonly used detection method. Pulsed amperometric detection is replacing these optical methods for carbohydrates and is now being used for other nonchromophoric molecules containing alcohol, aldehyde, amine, or sulfur functional groups.

Although carbohydrates can be oxidized at gold and platinum electrodes, the products of the oxidation reaction poison the surface of the electrode, inhibiting further analyte oxidation. By repeatedly pulsing between high positive and negative potentials, a stable and active electrode surface can be maintained. Pulsed amperometric detection at a gold electrode is a reproducible and sensitive method for all carbohydrates of molecular weight up to approximately 10,000.

Carbohydrates can only be detected by pulsed amperometry in high pH solutions (above pH 11). They are also very weak acids (with pKa's around 12) and are easily separated on high efficiency anion exchange columns such as Dionex CarboPac™ columns. Mobile phase pH's of 11 to 14 are used (1 mM to 1 M NaOH). Sodium acetate is often added to elute oligo- and polysaccharides by increasing the strength of the mobile phase. The combination of anion exchange separation with pulsed amperometric detection is a powerful method for the determination of carbohydrates. Although there can be some interference from amines and certain sulfur species, the technique is generally very sensitive and selective.

Principles of Pulsed Amperometry

To understand the mechanism of pulsed amperometry, it is first necessary to study the oxidation of an analyte using a conventional electrochemical technique such as voltammetry. The cyclic voltammetry of glucose in 0.1 N KOH using a gold working electrode and an Ag/AgCl reference electrode is shown as an example in Figure 6-9.

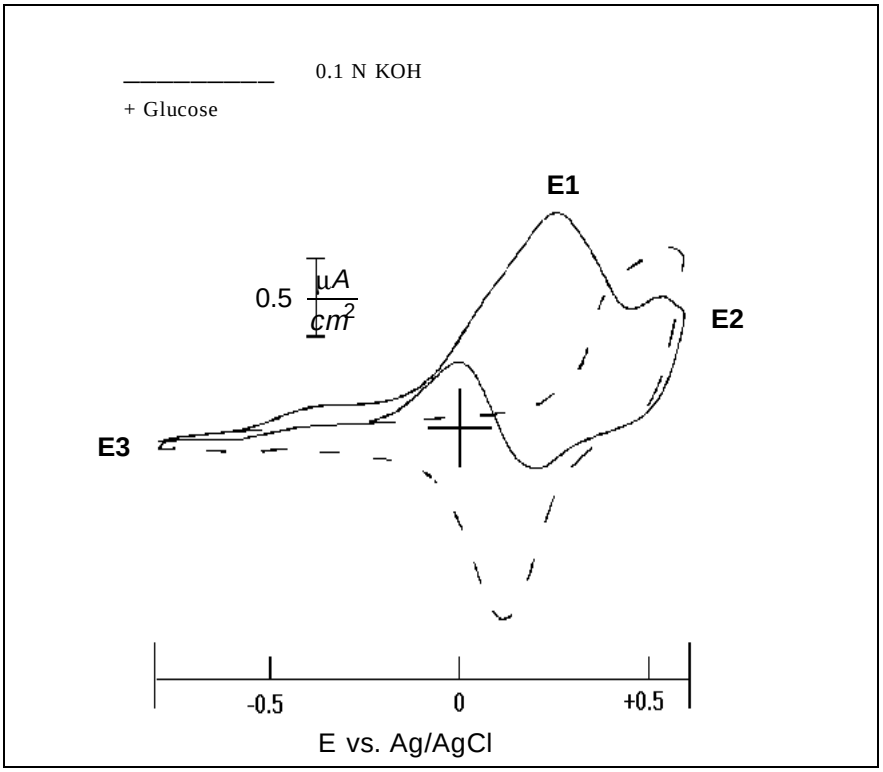


Figure 6-9. Cyclic Voltammetry of Glucose, Gold W.E.

The dashed line in the figure is the current resulting from the 0.1 N KOH supporting electrolyte in the absence of glucose analyte; that is, the background current. Beginning at -0.8 V and sweeping in a positive direction, the background current is nearly flat until approximately 0.25 V, where oxidation of the surface of the gold electrode to gold oxide begins. Following reversal of the potential sweep direction at 0.6 V, the gold oxide is reduced back to gold, with the negative peak current at 0.1 V.

With glucose added to the solution, the current rises slightly as the potential is swept in a positive direction from -0.8 V and remains unchanged until glucose oxidation begins. This causes the current to rise at -0.15 V towards a peak at about 0.25 V. The current then decreases because (1) the concentration of glucose at the electrode surface has been depleted because much of it has been oxidized and (2) the formation of gold oxide inhibits further glucose oxidation. On the reverse scan, the current actually reverses from reducing to oxidizing (positive) at the onset of the gold oxide reduction; as soon as the reduction of gold oxide back to gold begins, oxidation of glucose also begins.

If DC amperometric detection were used, the appropriate applied potential would be approximately 0.2 V. This is the potential at which the glucose oxidation current is the highest and the background current the lowest. However, the use of a single potential results in rapidly decreasing sensitivity as an oxide layer forms and products from the oxidation reaction coat and poison the electrode surface. This problem is solved by first measuring the glucose oxidation current near the peak at 0.25 V, pulsing the potential to a high positive value to form gold oxide, and then back to a potential negative of the gold oxide reduction peak. The action of repeatedly forming and removing the metal oxide surface layer cleans the electrode surface and maintains an active and stable surface. A three-step waveform useful for detecting carbohydrates is shown in Figure 6-10.

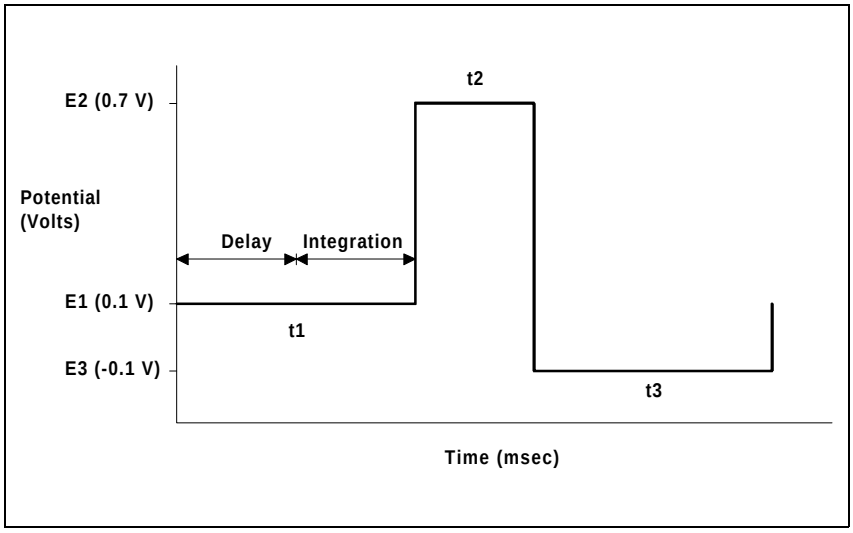


Figure 6-10. Three-Step Pulsed Amperometry Waveform

The potentials are labeled E1, E2, and E3 and are applied for durations of t1, t2, and t3, respectively. (E1 is +0.1 V, below the peak current at 0.25 V. This minimizes noise caused by gold oxide formation background current, which begins at approximately 0.2 V. It also provides a margin of safety if the event of any shifts in reference electrode potential.) The signal is measured at E1 by integrating the current for a fixed time and storing the resulting charge in a sample-and-hold amplifier until the next measurement. Current integrated for a fixed time is charge, and the units are coulombs. The ED40 reports the charge directly in coulombs. The step from E3 back to E1 is from -0.1 V to +0.1 V, which charges up the electrode/solution interfacial capacitance.

The carbohydrate oxidation current is integrated after a delay which allows the charging current to decay. Similar triple-potential waveforms are used to detect other species, especially alcohols and aldehydes.

Optimizing the Waveform Parameters

To find the optimum potentials and times for pulsed amperometric detection, begin with a basic waveform and then optimize the parameters. Choose the basic waveform from the voltammogram, or from published parameters for similar compounds (see Appendix F for a listing).

First, E1 is chosen to be the potential providing the largest ratio of analyte oxidation current to background current. Good starting values would be 0.4 second for t1, containing a 0.2-second delay period and a 0.2-second integration period. Set E2 and E3 near the positive and negative potential limits. Start with durations of 0.1 second for both. To optimize all the parameters, each one must be varied independently and the effect on reproducibility, signal magnitude, baseline level, and noise determined. For example, Figure 6-11 shows the dependence of background current on delay time for carbohydrate determinations using a gold working electrode.

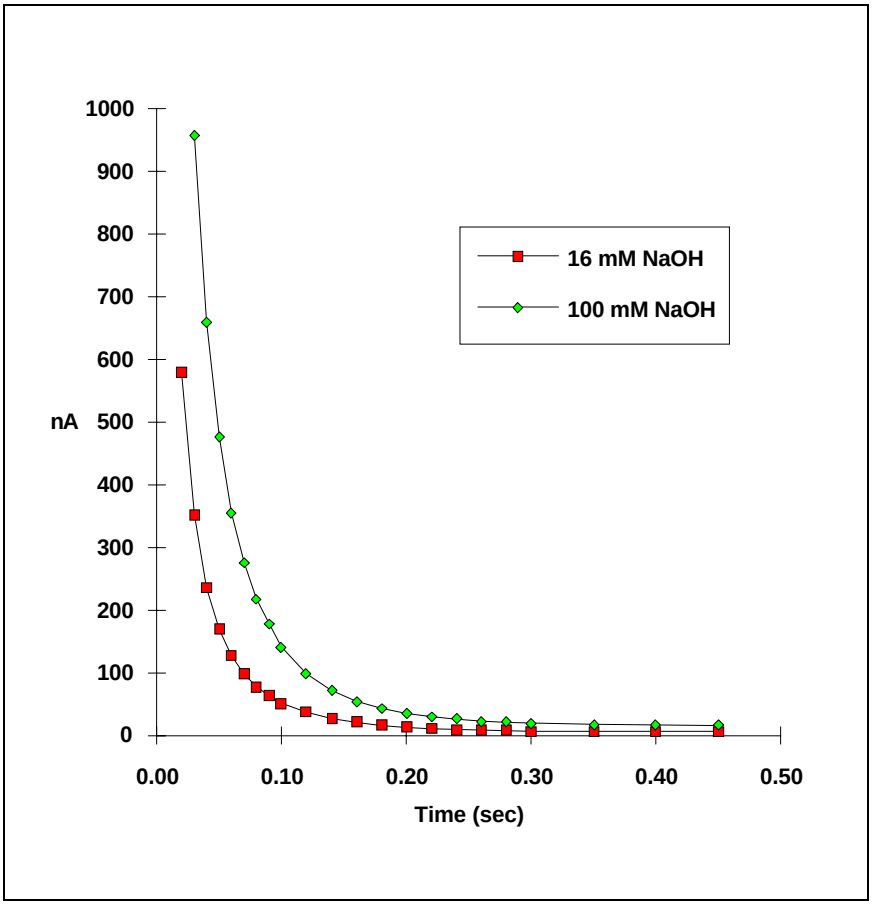


Figure 6-11. Charging Current vs. Delay Time, Au W.E.

This plot was developed by making repeated measurements while moving the integration period to later times. A delay time of 0.2 second is sufficient to allow the charging current to decay to near zero. The dependence of analyte peak height on delay time, using a gold working electrode, is shown in Figure 6-12.

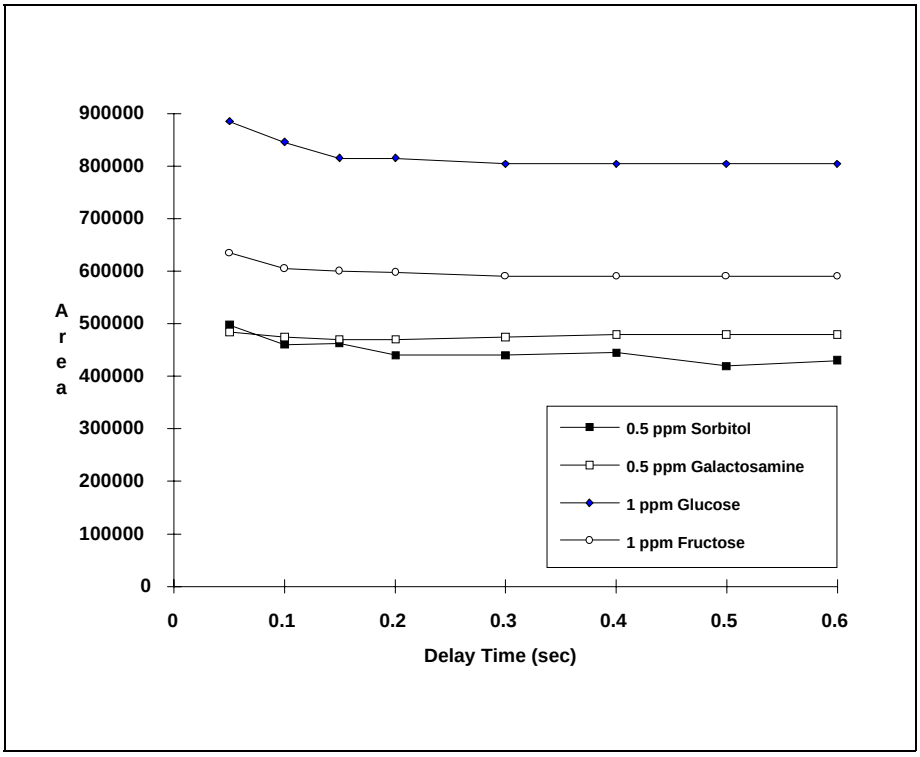


Figure 6-12. Peak Height vs. Delay Time

Since only a minor decrease in response occurs between zero and 0.2 second, a delay time of at least 0.2 second provides the optimum signal-to-background ratio. Similar plots are then created for E1 and for the positive and negative cleaning times and potentials by making repeated injections while varying their values. The cleaning potentials should be the least positive and negative potentials that produce both maximum detector response and reproducible response. (Check reproducible response with real samples and not just with standards, to ensure that sample components which may coat and poison the working electrode do not interfere with electrode cleaning.) To maintain an accurate reproduction of the eluting peak, a rapid sample rate is obtained using entire

waveforms that are as short as possible. The resulting waveform is usually a compromise of each of these goals. For example, doubling the integration period will increase the signal-to-noise ratio, but will decrease reproducibility of early eluting peaks by increasing the total waveform period. The program shown in Figure 6-10 was developed using this optimization method.

Species Detected by Pulsed Amperometry

Pulsed amperometric detection has emerged as the most sensitive and selective method for the detection of carbohydrates. High-pH anion-exchange with pulsed amperometric detection (HPAE-PAD) is becoming the principal method for analyzing the carbohydrate portions of glycoproteins (Figure 6-13).

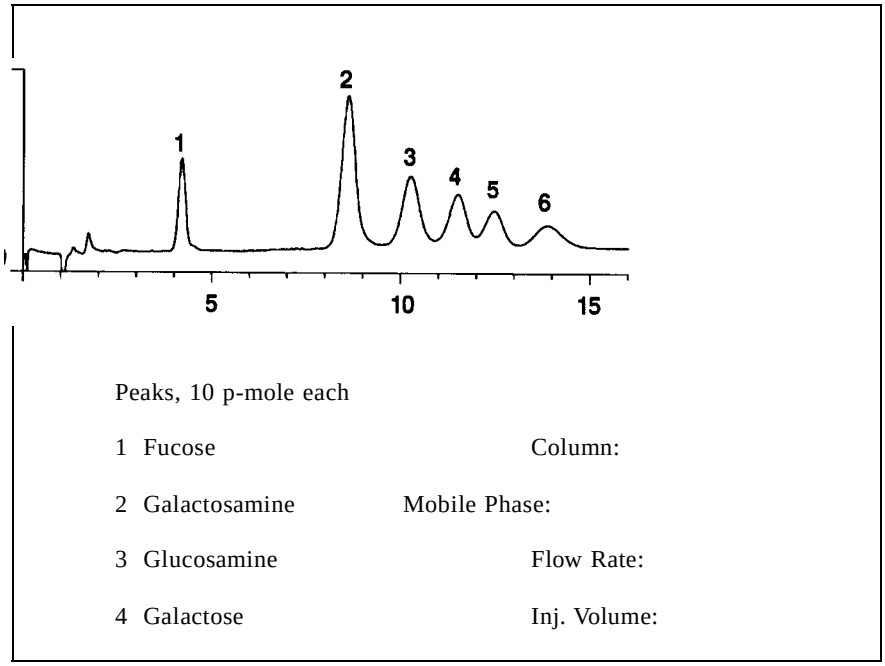


Figure 6-13. Monosaccharides from Glycoproteins Standard

Many new applications are being developed for the determination of carbohydrates in foods and beverages and in biological tissues and fluids.

Other species which can be detected by pulsed amperometry include alcohols, aldehydes, amines (primary, secondary, and tertiary, including amino acids), and many organic sulfur species. Thiols, sulfides, and mercaptans can be detected, but fully-oxidized sulfur species such as sulfates, sulfonates, and sulfones cannot be detected. Refer to Appendix F for the names of articles describing the use of pulsed amperometry to detect these species.

6.4.4 Integrated Amperometry

Integrated amperometric detection employs a repeating potential vs. time waveform. The cell current is integrated during a specific section of the waveform. It is a more general technique than pulsed amperometric detection in that pulsed amperometry employs a repeating sequence of three potentials, while integrated amperometry can be any waveform. This method is useful for amines and sulfur species because their oxidation at metal electrodes is catalyzed by the formation of metal oxide, and an integrated amperometry waveform can be used that minimizes background effects caused by metal oxide formation. Also, integrated amperometry can be used to decrease the effect of changes in pH, allowing moderate pH gradients to be performed without large shifts in the baseline. The principle of integrated amperometry is illustrated in Figure 6-14.

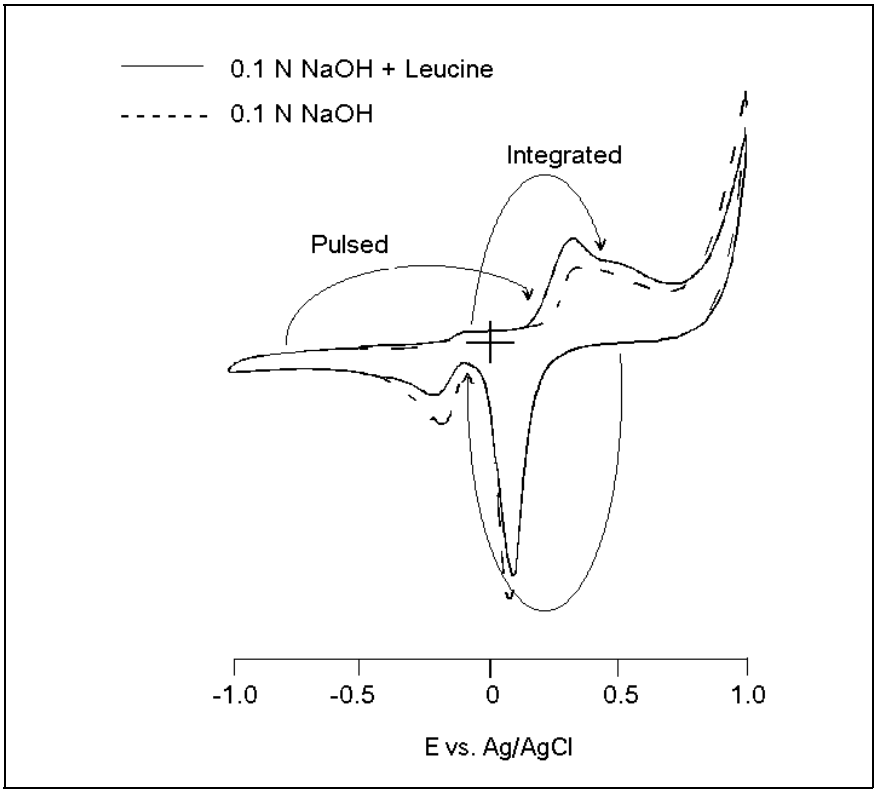


Figure 6-14. Pulsed vs. Integrated Amperometry

Figure 6-14 compares pulsed vs. integrated amperometry for leucine on a gold working electrode in 0.1 N NaOH. With pulsed amperometry, current is integrated at a single potential after a potential step. With integrated amperometry, current is integrated during a cyclic potential sweep.

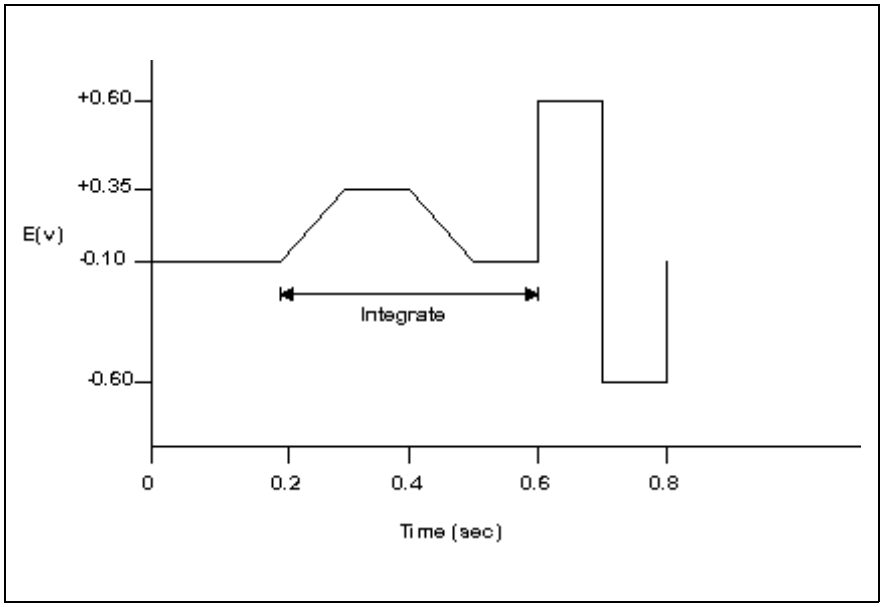


Figure 6-15. Example Integrated Amperometry Waveform

With the integrated amperometry waveform shown in Figure 6-15, the current is integrated twice—first, while the potential is swept across the metal oxide formation wave, and then during the reverse sweep across the oxide reduction wave. Without the presence of analyte molecules, the net charge is around zero. Positive and negative cleaning pulses are added to the potential vs. time waveform following the integration period. Waveforms are optimized using a technique similar to the method used for pulsed amperometry.

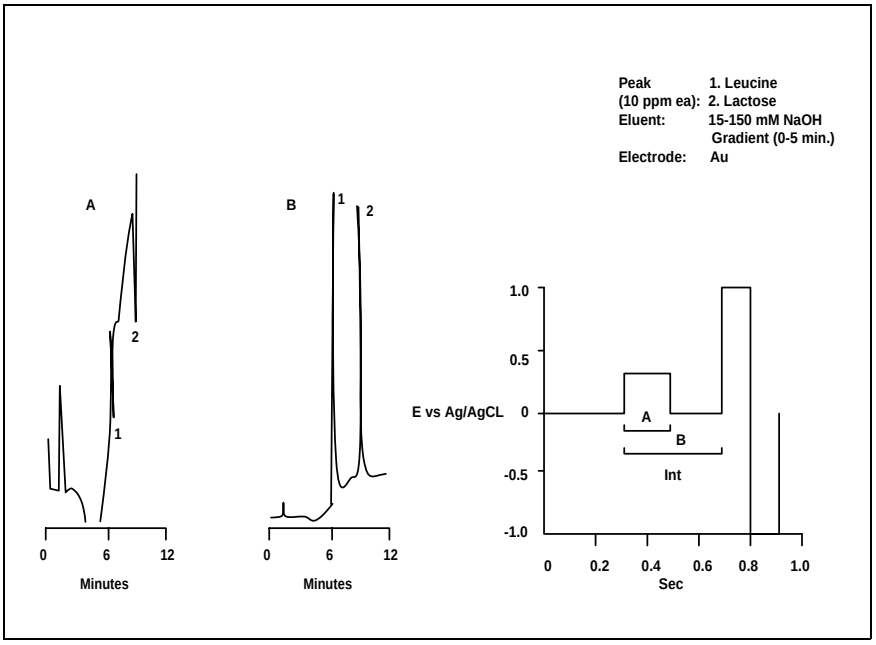


Figure 6-16. Comparison of Integration Periods

The advantage of integrated amperometry is that canceling the charge from oxide formation and reduction greatly minimizes the effect on the baseline. For an example of the effect of canceling the working electrode oxidation current, see Figure 6-16. The figure compares of two integration periods for the detection of 10 ppm leucine (peak 1) and lactose (peak 2). The gradient is 15 to 150 mM NaOH.

When the current is integrated only during the forward step across the metal oxide formation wave, the baseline shift during the gradient is very large (Figure 6-16A). Also, the eluting analytes cause dips instead of peaks because their effect on surface oxide suppression is greater than the detector response from the analytes. Integrating the current for both the forward and reverse steps greatly minimizes the baseline shift and eliminates the dips, resulting in good peaks (Figure 6-16B).

Integrated amperometry is a new technique developed in Professor Dennis Johnson's laboratory at Iowa State University. Dr. Johnson has published several recent papers showing the utility of integrated amperometry. These papers are listed in Appendix F.

6.4.5 Working Electrodes

The ED40 can be used with four working electrode materials: gold, silver, platinum, and glassy carbon. The precious metal electrodes are very high purity solid metals, while glassy carbon is a hard graphitic substance. The analytes determined using these working electrodes are listed in Table 6-3.

The choice of working electrode material for a given application depends on four factors:

- Potential limits for the working electrode in the mobile phase.
- Involvement of the electrode in the electrochemical reaction.
- Kinetics of the electron transfer reaction.
- Long-term stability of the electrode.

Potential Limits

The negative potential limit is the potential at which the mobile phase or supporting electrolyte is reduced. At the positive potential limit, the mobile phase, the supporting electrolyte, or the electrode itself can be oxidized. Because these reactions produce current far in excess of the analytical redox reaction, the potential used to detect the analyte must be within these limits. Table 6-4 lists the potential limits for the four electrode materials in acidic and basic solutions. The potential limits are strongly affected by the pH of the mobile phase. Negative potential limits are more negative in base and more positive in acid. Conversely, positive limits are more positive in acid and more negative in base. In other words,

the usable potential window shifts negative in basic solutions and positive in acidic solutions.

As the applied potential approaches the potential limit, the noise will increase as the background current increases. On metal electrodes, there is a sharp increase in background current as the potential limit is approached. On glassy carbon, the increase in background current is more gradual. Because the maximum applied potential that can be used is determined by the required signal-to-noise ratio, the values listed in Table 6-4 are only a rough guide. For some applications using glassy carbon, it may be necessary to exceed these limits.

Working Electrode	Solution (0.1 N)	Negative Limit (V)	Positive Limit (V)
Glassy Carbon (G.C.)*	KOH	(-1.5)	(+0.6)
	$HClO_4$	(-0.8)	(+1.3)
Gold (Au)	KOH	-1.25	+0.75
	$HClO_4$	-0.35	+1.50
Silver (Ag)	KOH	-1.20	+0.10
	$HClO_4$	-0.55	+0.40
Platinum (Pt)	KOH	-0.90	+0.65
	$HClO_4$	-0.20	+1.30

*Unlike metallic electrodes, the potential limits for the glassy carbon electrode do not cut off sharply. The noise and background level differ from application to application and must be determined experimentally.

Table 6-4. Potential Limits vs. Ag/AgCl Electrode

The largest positive potential limits are obtained on glassy carbon and platinum. Accordingly, oxidations are often performed using one of these two materials. The largest negative potential limits, listed in order, are obtained on glassy carbon, silver, and gold. Because of the ease of reducing hydrogen ion to hydrogen gas on a platinum electrode, platinum has a poor negative potential limit and is generally not used for reductions.

When potentials are used that are more negative than approximately -0.1 V vs. an Ag/AgCl reference electrode, high background current is caused by the reduction of molecular oxygen dissolved in the mobile phase. Degassing the mobile phase greatly reduces this background current.

Involvement of the Electrode in the Redox Reaction

The reaction mechanism for the oxidation of many analytes is the transfer of electrons from the analyte molecules to the electrode. The electrode acts as an inert electron sink, but is otherwise not involved in the oxidation reaction. With this reaction mechanism, carbon is often the preferred electrode material. Examples include the detection of catecholamines and aromatic amines.

In contrast, silver and gold can be oxidized in the presence of complex or precipitate-forming ions. For the detection of these ions, the working electrode material is directly involved in the reaction and is actually slowly consumed, although so slowly that no degradation in performance is observed. For example, silver can be oxidized to silver cyanide in the presence of cyanide ion. This reaction takes place at a much lower potential than the oxidation of cyanide to cyanate at a platinum electrode. The ability to use a lower applied potential increases the selectivity of the analysis, as fewer other species will be oxidized. Also, the oxidation of trace contaminants in the mobile phase causes less noise at the lower applied potential. A silver electrode can also be used to detect other complex or precipitate-forming ions. These

include bromide and iodide, as well as numerous sulfur-containing species such as sulfide, sulfite, thiosulfate, and organic thiols.

One disadvantage to the use of silver or gold for oxidations is that the presence of halides in the mobile phase greatly decreases the positive potential limit. Halides can usually be replaced by nonreacting anions such as acetate, methanesulfonate, perchlorate, nitrate, phosphate, or sulfate.

Kinetics of the Electron-Transfer Reaction

For a kinetically fast electron-transfer reaction at E°, the ratio of the oxidized to the reduced form of the analyte species will be described by the Nernst equation (see Section 6.4.1). The reaction is said to be *reversible*. If E_{app} is set 0.118 V positive of E°, then (for $n = 1$) the ratio of the oxidized to the reduced form of the analyte at the electrode surface will be 100:1. Nearly all of the analyte that reaches the electrode will be oxidized. The limiting current will have been attained, and no advantage will be gained from a further increase in the applied potential.

More often than not, redox reactions are *irreversible*, in that the electron transfer reaction rate is slow at E°. Irreversible reactions require a potential considerably in excess of E° to drive the reaction at a fast rate. This excess voltage is called *overpotential*. The more irreversible the redox reaction, the greater the applied potential must be. This results in more noise and less selectivity. The oxidation or reduction of many species is more facile on one electrode material than on another. This is particularly true for small inorganic species, many of which can be oxidized or reduced much more easily on platinum than on carbon. For example, platinum electrodes are used to detect arsenite, iodide, and sulfite.

Long-Term Stability of the Electrode

A major consideration when choosing an electrode material is its ability to maintain an active surface. Electrodes will develop a layer of surface oxide at positive applied potentials. This build-up inhibits oxidation of the analyte, which often results in decreasing response over repeated injections. Polishing the electrode will renew the active surface.

Glassy carbon electrodes are more resistant to poisoning by oxide formation than are metallic electrodes, and do not need to be polished as often. This is one of the reasons glassy carbon is used far more extensively than any other electrode material for DC amperometry.

Pulsed amperometry and integrated amperometry solve this problem for platinum and gold electrodes by pulsing to high positive and negative potentials after each current measurement to clean the electrode.

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A • Specifications

A.1 Electrical

Main Power 85 to 270 Vac, 47/63 Hz; 40 W Max, 25 W typical. The ED40 power supply is auto-sensing and requires no voltage adjustment.

Fuse Two 3.15 amp fast-blow IEC127 fuses (P/N 954745)

Analog Output User-selectable full-scale output of 10, 100, or 1000 mV

A.2 Environmental

Operating Temperature 4 ºC to 40 ºC (40 ºF to 104 ºF)

Operating Humidity 5 to 95% relative humidity, non-condensing

A.3 Physical

Dimensions 22.5 cm wide x 17.0 cm high x 42.0 cm deep
(8.8 in wide x 6.6 in high x 16.4 in deep)
6 cm (2.4 in) clearance required behind the detector

Weight 8.2 kg (18 lb)

A.4 Display and Keypad

Display Liquid crystal display with adjustable backlighting

Keypad 26-button keypad for entering commands and numerical values for screen parameters

A.5 Detector

Range	0.01 µS to 3000 µS, full scale (Conductivity) 50 pC to 200 µC (Integrated Amperometry) 50 pA to 300 µA (DC Amperometry)
Temperature Compensation	0.0 to 3.0% per ºC (Conductivity)
Cell Drive	Variable 8 kHz square wave (Conductivity) ±2.04 V (DC and Integrated Amperometry)
Local Operation	Front panel controls and display status of all functions.
Remote Operation	Control of 4 of 7 functions via TTL or Relay contacts.
DX LAN Operation	All functions controlled by PeakNet Software on a PC connected to the ED40 via the DX LAN interface (optional).

A.6 Conductivity Cell

Cell Body	PEEK
Active Volume	1.0 µL
Maximum Pressure	2.0 mPa (300 psi)
Electrodes	316 stainless steel

A.7 Amperometry Cell

Cell Body	Titanium (counterelectrode)
Active volume	~ 0.2 µL
Maximum Pressure	0.7 mPa (100 psi)
Working Electrodes	Gold, Silver, Platinum, Glassy Carbon
Reference Electrode	Combination pH–Ag/AgCl

A.8 SRS Power Supply

Supply Current	50, 100, 300, 500 mA @ 1.5 to 7.5 V
Over-Voltage Alarm	8.5 V
Over-Temperature Alarm	40 ºC

A.9 DS3 Detection Stabilizer

Operating Temperature	25 ºC to 40 ºC (50 ºF to 104 ºF)
Warm-up Time	10 minutes (typical)
Under- or Over-Temperature Alarms	Message displayed when not at set temperature

B • Installation

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B • Installation

B.1 Facility Requirements

- Make sure the ED40 installation site meets the electrical and environmental specifications listed in Appendix A.
- Install the ED40 on a sturdy table or workbench, at a height that ensures convenient viewing of the front panel display.
- Allow at least 5 cm (2 in) clearance behind the ED40 for power connections and ventilation.

B.2 System Configuration

Dionex DX 500 modules are designed to be stacked on top of each other, up to a maximum height of four units (see Figure B-1). The ED40, for example, is one unit high. Stacking the interlocking modules creates a compact system that requires a minimal amount of bench space; it also allows the length of tubing and cable connections to be minimized. All cables are routed through the front slots and rear chases under the modules.

NOTE

The Ship Kits for the DX 500 pumps and chromatography modules contain a shoe (P/N 046478) and two ties (P/N 046476). These items help secure the ED40 to other DX 500 modules, preventing the modules from tilting or sliding apart. Installation of the shoe and ties is optional; to install them, refer to the pump or chromatography module manual for instructions.

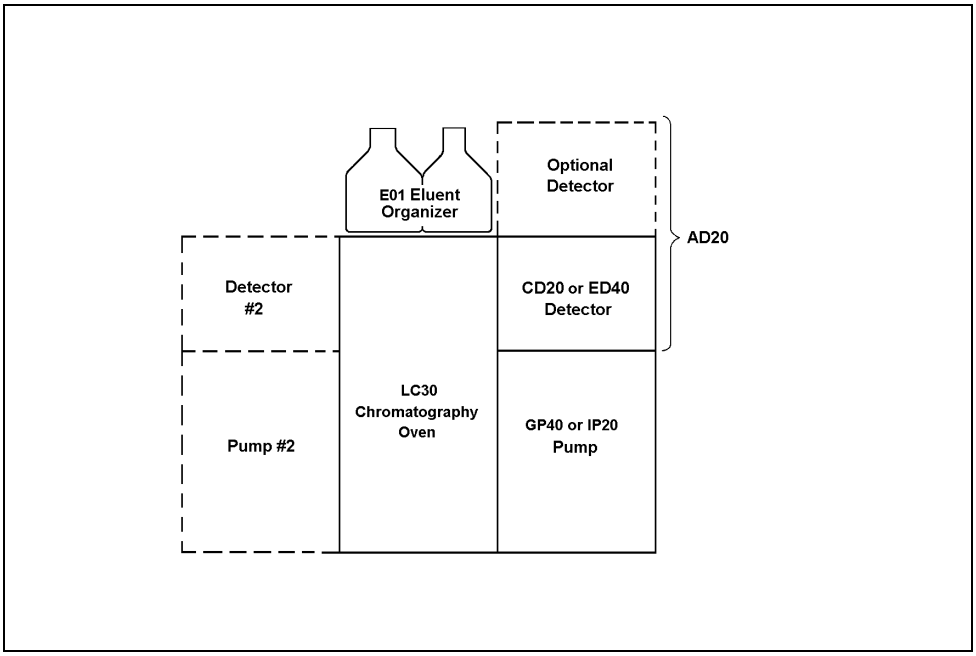


Figure B-1. Recommended DX 500 System Configuration

B.3 Installation



Lift the detector only from the bottom or side surfaces. Lifting with the front panel doors will damage the door hinges.

B.3.1 Power Connection

You can control power to the ED40 from the main power switch on the ED40 or, if present, the LC30 Chromatography Oven. In either case, no adjustment is required to select the line voltage.

For on/off control from the ED40, connect the modular power cord (IEC 320 C13) provided with the ED40 from the main power receptacle on the ED40 rear panel (Figure B-2) to a grounded, single-phase power source.

The LC30, when properly connected to other modules in the chromatographic system, can function as the main power source for the system. The LC30 Ship Kit provides IEC jumper power cables (P/N 960748) for this purpose. Connect a jumper cable from the ED40 main power receptacle to one of the four IEC auxiliary receptacles on the LC30 rear panel. Leave the ED40 power switch on continuously and use the LC30 main power switch to turn the detector on and off.



SHOCK HAZARD—To avoid electrical shock, a grounded receptacle must be used. Do not operate or connect to AC power mains without an earthed ground connection.



Operation of the ED40 at AC input levels outside of the specified operating voltage range may damage the detector.

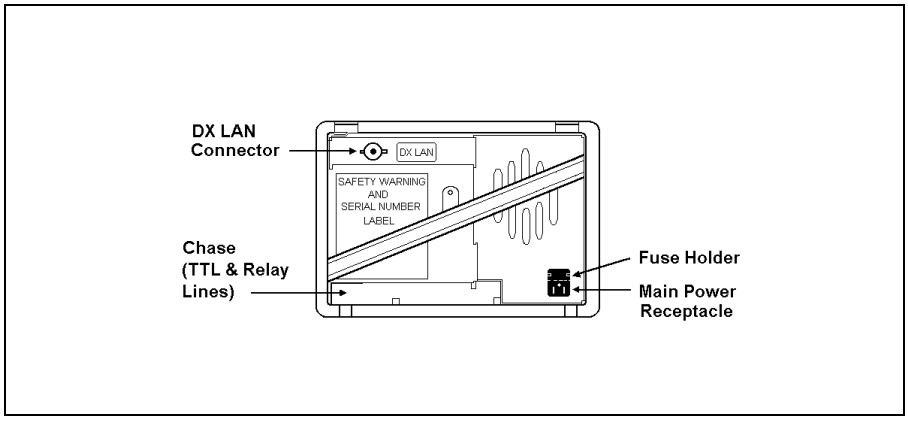


Figure B-2. ED40 Rear Panel

B.3.2 DX-LAN Network Connection (Optional)

For the ED40 to communicate with PeakNet Software, you must install a detector interface card (P/N 044196) in the ED40 and connect the DX-LAN cable (P/N 960404) to the rear panel of the detector.

IMPORTANT

STATIC—The ED40 electronics are not user-serviceable and the detector interface card should be installed by qualified personnel only. Observe standard anti-static procedures when installing the interface card or handling the CPU card.

CAUTION

To prevent damage to the detector, turn off the main power before installing the interface card. After confirming that the LED on the CPU card is off (not green or red), unplug the power cord from the mains. Do not rely on the front panel power switch.

1. Remove any TTL/Relay plugs from the connectors at slot 4 (the SP card) in the electronic chassis. (For an illustration of card locations, see Figure 2-3 in this manual or refer to the label on the inside of the front door.)

2. Disconnect the 60-pin ribbon cable from the front panel by first closing the tilt panel to expose the connector and its ejector latches. Remove the cable by opening the ejector latches.
3. Using a screwdriver as a lever, open the white ejector latch at the bottom of the CPU card. Remove the CPU card, cable, and Relay card as a single unit.
4. Insert the detector interface card (P/N 044196) into slot 4. Slide the card to the rear. Verify that the BNC connector is aligned with the hole at the rear and the card is aligned with the connector. Press firmly on the card until it mates fully with the connector on the rear panel.
5. Reinstall the CPU/Relay card. Press firmly until the CPU card is inserted into the connector on the rear panel.
6. Reconnect the ribbon cable to the 60-pin connector on the front panel. The header and connector are key-polarized near the center. The ejector latches should be partially open to accept the cable connector.
7. Attach a BNC tee connector (P/N 921914) to the BNC connector on the rear panel.
8. If the detector is the last module in the network to be connected, install a terminator plug (P/N 921034), shipped with PeakNet Software, on the remaining port of the BNC tee connector. If this is not the last module, connect the cable from the next module to the BNC tee.

Terminator resistor plugs must be installed at each end of the DX-LAN. Verify that both ends of the DX-LAN have resistor plugs installed.

9. Connect the DX-LAN cable (P/N 960404) to the BNC tee connector by twisting the rotating sleeve until a "snap" is heard (or felt).



The DX-LAN cable is a 50 ohm coaxial impedance cable. (Fifty ohm cables are imprinted with "RG58U.") Do not substitute an inferior cable, such as a 75 ohm television coaxial cable. Failure to use the correct cable or to correctly install the BNC connector will cause the module to lose communication with PeakNet.

B.3.3 DS3 Detection Stabilizer Installation

The DS3 with the conductivity cell (P/N 044130) can be installed on a workbench or in one of the DX 500 chromatography modules: the LC10 Chromatography Organizer, LC20 Chromatography Enclosure, or LC30 Chromatography Oven.

The DS3 can be plumbed for three operating modes:

- AutoSuppression™ Recycle mode (SRS required), illustrated in Figure B-3.
- AutoSuppression with External Regenerant mode (SRS required), illustrated in Figure B-4. Plumb the system in this way for the AutoSuppression Chemical Suppressor and MMS Pressurized Regen System configurations, also.
- Nonsuppressed mode, illustrated in Figure B-5. Note that conductivity measurements made when no suppressor is in-line may show noticeable baseline drift.

The procedures for installing the DS3 in an LC20 and LC30 are identical. The LC10 installation instructions are slightly different, and thus are presented separately.

Backpressure Requirements

All HPLC detector cells require backpressure to prevent mobile phase in the cell from degassing due to abrupt volume changes between the small ID of the connecting tubing and the larger volume of the cell. Degassing creates bubbles in the cell and disrupts detector responsiveness.

Before plumbing a system for one of the AutoSuppression modes, refer to the SRS manual for backpressure requirements.

NOTE

The backpressure generated by the DS3, cell, and backpressure tubing is applied to the SRS. For example, with 1 meter of 0.25-mm (0.01-in) ID tubing at a flow rate of 1.0 mL/min, the backpressure is about 200 kPa (30 psi).

Table B-1 lists backpressure requirements for the Nonsuppressed mode. The minimum backpressure required is about 0.21 to 0.34 mPa (30 to 50 psi) per minute.

Flow Rate (mL/min)	Tubing ID	Coil Length	Number of Coils
0.5 to 1.5	0.25-cm (0.010-in)	0.76 M (2.5 feet)	2
1.5 to 3.0	0.25-cm (0.010-in)	0.76 M (2.5 feet)	1
0.12 to 0.37	0.12-cm (0.005-in)	0.30 M (1.0 feet)	2
0.37 to 0.75	0.12-cm (0.005-in)	0.30 M (1.0 feet)	1

Table B-1. Coils for Backpressure Requirements

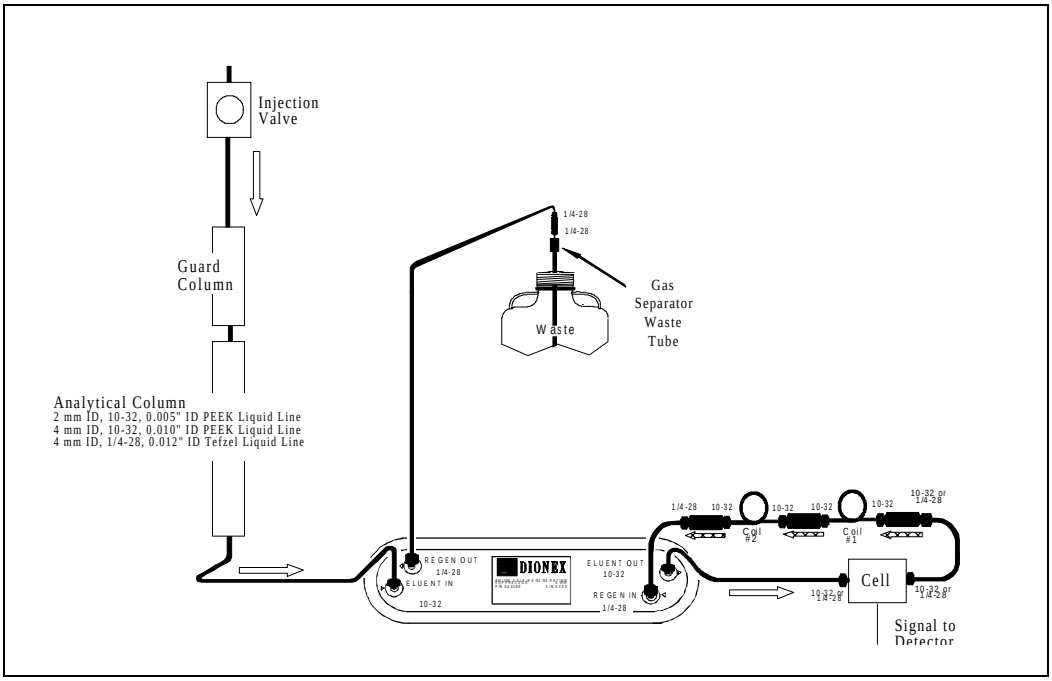


Figure B-3. AutoSuppression Recycle Mode

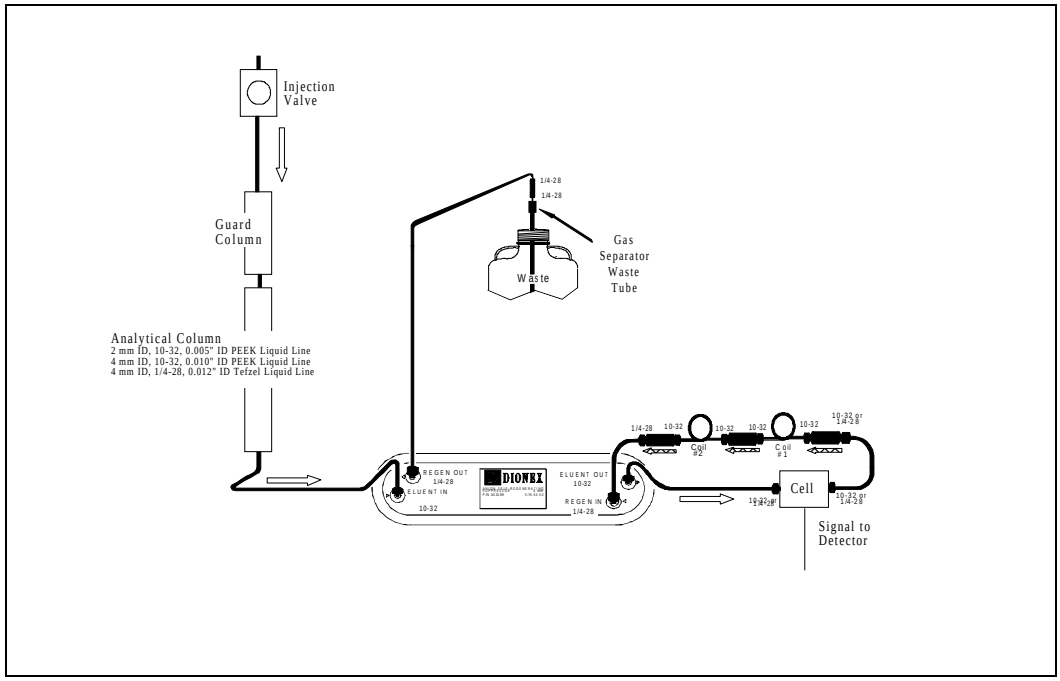


Figure B-4. AutoSuppression Mode, External Regenerant

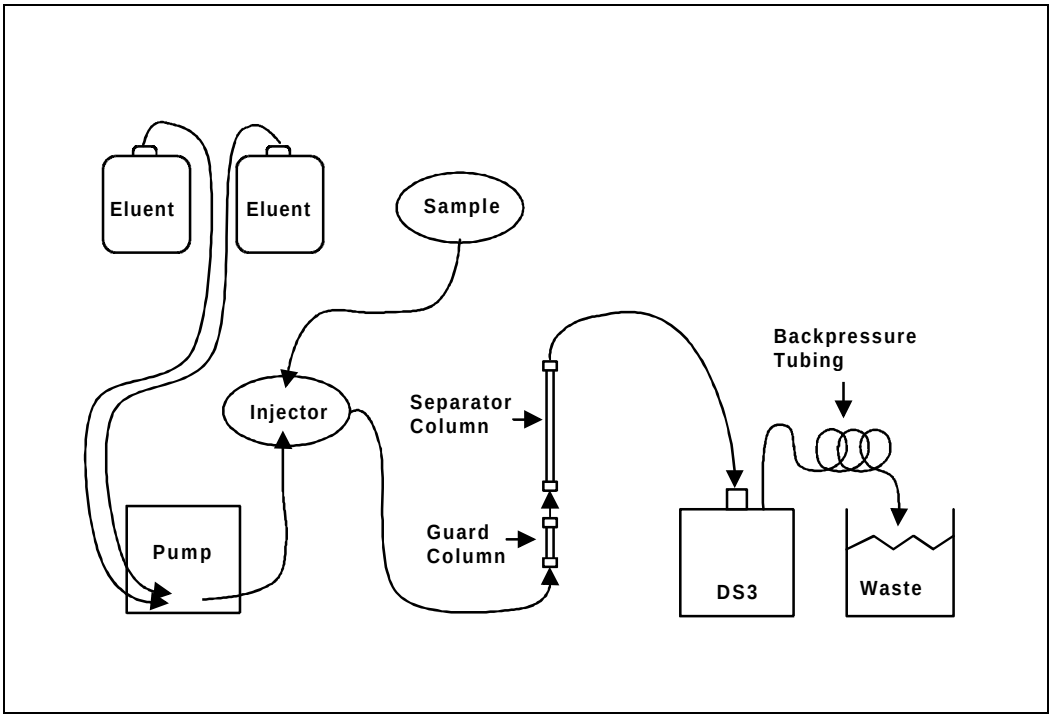


Figure B-5. Nonsuppressed Mode

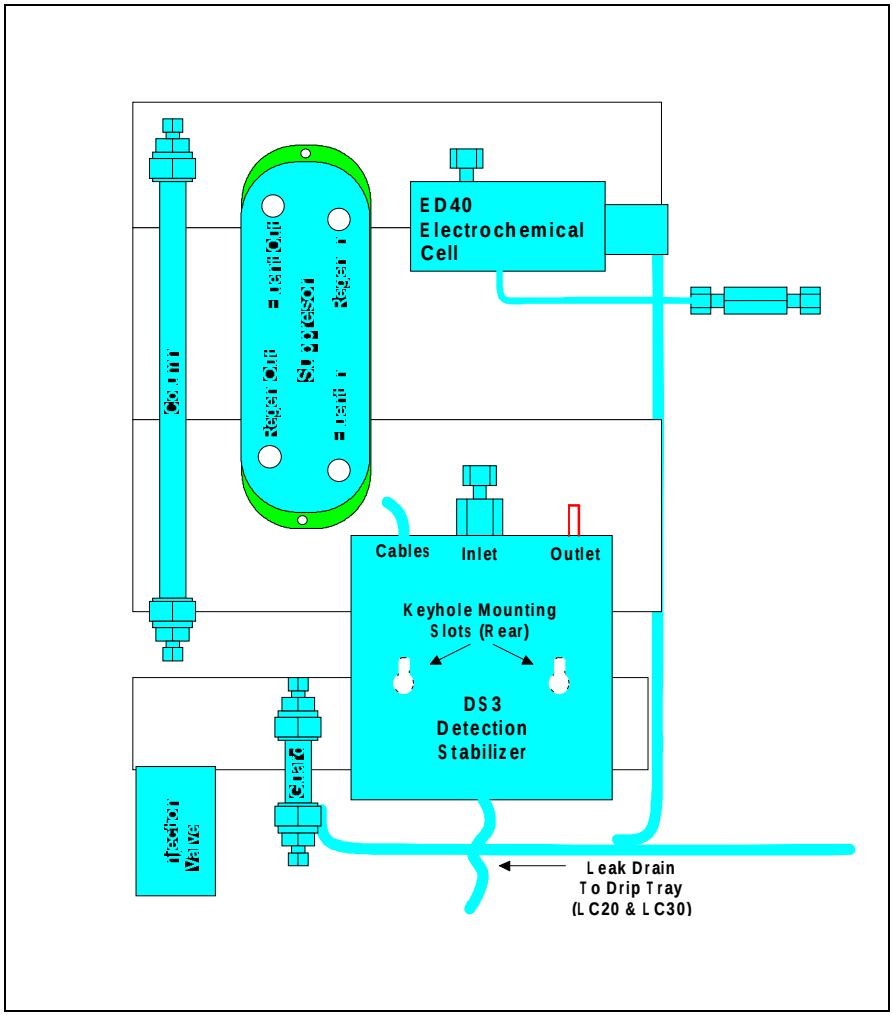


Figure B-6. Typical DS3 Installation in LC20 or LC30

Installation of a DS3 in an LC20 or LC30

1. Position the keyhole slots on the rear of the DS3 over the shoulder washers on the LC20 or LC30 component mounting panel(s). Pull the DS3 down into position.

2. Route both DS3 cables out the hole located at the back of the LC20 or LC30 and into the cable chase the base of the detector. Then route the cables through the cable chase to the front of the detector. (You can also route the cables into the detector through the slots on the sides of the detector.)
3. Plug the connectors into the appropriate jacks on the SP and SCR cards. (For an illustration of card locations, see Figure 2-3 in this manual or refer to the label on the inside of the front door.)
4. Connect the eluent outlet of the column or suppressor to the DS3 inlet, using 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).
5. Connect the outlet of the DS3 (the 10-32 coupler) to the SRS REGEN IN port (for the AutoSuppression Recycle mode) or to the backpressure line leading to waste (for the AutoSuppression with External Regenerant mode or Nonsuppressed mode).
6. Direct the spill/overflow tubing to the spill tray at the bottom front of the LC20 or LC30.

Installation of a DS3 in an LC10

1. Cut the spill/overflow tubing within 2.0 cm (3/4 in) of the DS3 enclosure.
2. Position the keyhole slots on the rear of the DS3 enclosure over the shoulder washers located on the LC10. Push the DS3 down to secure it in place.
3. Route both DS3 cables through the hole located at the base of the detector cable chase. Then route the cables through the cable chase to the front of the detector. (You can also route the cables into the detector using the slots on the sides of the detector.)

4. Plug the connectors into the appropriate jacks on the SP and SCR cards. (For an illustration of card locations, see Figure 2-3 in this manual or refer to the label on the inside of the front door.)
5. Connect the eluent outlet of the column or suppressor to the DS3 inlet, using 0.25-mm (0.010-in) ID PEEK tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276). See Table 3-1 for backpressure information.
6. Connect the outlet of the DS3 (10-32 coupler) to either the SRS REGEN IN port (for the AutoSuppression Recycle mode) or to the backpressure line leading to waste (for the AutoSuppression with External Regenerant mode or Nonsuppressed mode).

B.3.4 Shielded Conductivity Cell Installation

The shielded conductivity cell (P/N 044132) is installed in the same location as the DS3. The keyhole slots on its base plate fit on the LC10, LC20, and LC30 in the same way as the DS3. Except that the shielded cell does not have a heater power cord, installation instructions are identical.

B.3.5 Amperometry Cell Installation

Install the ED40 amperometry cell directly after the column. (A suppressor is not typically used with the amperometry cell.) A second detector (an AD20 Absorbance Detector, for example) may be installed before the amperometry cell. However, if a second detector is installed ***after*** the cell, make sure the pressure at the amperometry cell outlet remains below 700 kPa (100 psi).

Because of the volume within the reference electrode section of the cell, you may note some band broadening at the second detector. However, this is minimized by the precision flat bottomed reference electrode.

1. Inspect the cell gasket; if it is scratched or damaged, replace the gasket (P/N 045972).
2. Rinse the gasket with deionized water. Clean the polished surface of the cell with a damp paper towel.
3. Install the gasket over the alignment studs on the cell body. When correctly installed, one end of the gasket extends beyond the cell body, to facilitate gasket installation and removal.
4. Polish the working electrode block (see Section 5.5) and rinse its surface with deionized water. Wipe it with a damp paper towel.

NOTE

Polishing the working electrode is recommended only before initial installation and if the electrode ever becomes seriously fouled.

5. Install the polished working electrode block over the alignment studs so that the electrode type stamped on the top of the working electrode faces up.

6. Fasten the working electrode in place with the wing screws (P/N 045937). The electrode material stamped on the block is visible with the cover on.
7. Remove the pH reference electrode (P/N 046333) from its box within the amperometry cell shipping container. Remove the electrode from the soaker bottle in which it is shipped by partially unscrewing the lid of the soaker bottle and pulling the electrode out of the O-ring groove within the lid.
8. Rinse the reference electrode thoroughly in deionized water to remove any precipitated salt. Do not discard the soaker bottle and lid; they are required for safe storage of the electrode when the amperometry cell is not in use.
9. Following the instructions on the **pH CALIBRATION** screen, calibrate the reference electrode.
10. To install the calibrated electrode, remove the reference electrode compression nut (P/N 045968) from the cell body.
11. Fill the reference electrode cavity within the cell body with enough deionized water to saturate the O-ring within the reference electrode cavity.
12. Feed the reference electrode cable (P/N 045868) through the slot in the compression nut. Carefully guide the reference electrode with the nut into the reference electrode cavity. After making sure the electrode is properly aligned, screw the compression nut into the cell body until it completely bottoms out. **If the electrode is not aligned when you tighten the compression nut, the side of the electrode may crack.** As you tighten the compression nut, the reference electrode will slip into position past the O-ring.
13. Connect the reference electrode cable to Junction J2 on the cell pre-amp PC board.
14. Verify that the white working electrode lead wire is connected to junction J1 on the cell pre-amp PC board.

15. Slide the cell cover back over the cell body and tighten the cell cover thumbscrew.

16. Make sure the detector cell is turned off. Connect the amperometry cell cable to connector #1 on the SP card. (For an illustration of card locations, see Figure 2-3 in this manual or refer to the label on the inside of the front door.)

    a. If the chromatography module is an LC20 or LC30, feed the amperometry cell cable under the card cage and out the back of the ED40, or directly out the side of the ED40. Connect the cable to the amperometry cell, allowing a service loop to slide the chromatography component card in and out of the module.

    b. If the chromatography module is an LC10, first remove the amperometry cell insulation plate (P/N 046617), cell mounting screws (P/N 045796), and cell mounting washers (P/N 045973) from the cell body. Store these components in a safe place; they are necessary insulation and mounting components for the LC20 and LC30 if the system is upgraded. Connect the amperometry cell cable to the amperometry cell.

    c. To install the cell outside the chromatography module, use the amperometry cell bracket (P/N 048749). Locate the bracket as close as possible to the column outlet, to minimize dead volume. When properly installed, the amperometry cell hangs on the bracket at an angle that minimize bubble formation.

17. Attach the tubing from the column to the cell inlet.

NOTE

The short length of titanium tubing at the cell inlet is an integral part of the cell. DO NOT REMOVE IT. This tube extends the counterelectrode, thereby shielding the working electrode from electrical noise.

18. Connect the waste line to the cell outlet.
19. Secure the plumbed amperometry cell to its appropriate mounting location in the chromatography module.

NOTE

When properly mounted, the outlet fitting on the amperometry cell is on the uppermost surface of the cell. This ensures proper orientation of the reference electrode within the amperometry cell and helps sweep out bubbles.

B.3.6 Recorder/Diagnostic Connection

Connecting a strip chart recorder or integrator to the ED40 allows you to record or monitor several parameters, in addition to the cell analog output. For a list of the pinouts for the cable, see Table E-1 in Appendix E.

Use the twisted black and red wires (P/N 043598) provided in the ED40 Ship Kit (P/N 046297) to connect a recorder or integrator to the SCR card. (For an illustration of card locations, see Figure 2-3 in this manual or refer to the label on the inside of the front door.)

Strip the ends of the wires and insert into the plugs. The signal wire (red) goes on top and the ground wire (black) goes on the bottom of each plug. Use the screwdriver (P/N 046985) provided in the ED40 Ship Kit to tighten the locking screws.

B.3.7 Relay/TTL Control Connections

Eight 2-pin connector plugs (P/N 921019) are provided in the ED40 Ship Kit (P/N 046297): four input and four output. Attach individual wires, twisted pairs, or coaxial cables to the plugs.

Strip the end of the wire(s) and insert into the plugs. The signal wire (red) goes on top and the ground wire (black) goes on the bottom of each plug. Use the screwdriver (P/N 046985) provided in the Ship Kit to tighten the locking screws.

Connections 1 and 2 can be programmed to switch any low-voltage control. Switched current must be less than 200 mA and 42 V peak.

CAUTION

The relay output, TTL output, and TTL inputs use the same style of connector. Connection of relay loads and their power sources to the TTL outputs will damage the TTL output stage. If the relay load can supply more than 200 mA at 5 V or higher, damage to the CPU module may result.

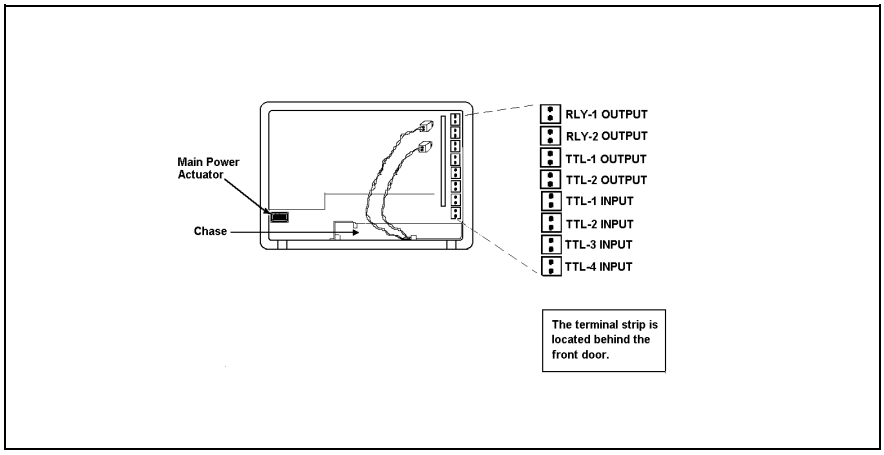


Figure B-7. TTL Connections

Table B-2 describes the Relay/TTL connector pinouts. TTL inputs 1, 2, 3, and 4 can be used to control any four of the functions listed in the table. Select the functions from the **TIME FUNCTION IN** screen.

- OFFSET Recorder
- HOLD/RUN Program
- SRS OFF/ON
- METHOD NUMBER INCRement
- METHOD NUMBER DECRement
- MARK Recorder
- Increase Recorder RANGE x10

Connector Number	Pin Number	Description
Relay 1	1 and 2	Solid State Relay Contacts Out
Relay 2	1 and 2	Solid State Relay Contacts Out
Relay 3	1 2	TTL Out (1 kΩ pull up to +5, 100 mA sink) Ground
Relay 4	1 2	TTL Out (1 kΩ pull up to +5, 100 mA sink) Ground
Relay 5	1 2	Input TTL 1 Ground
Relay 6	1 2	Input TTL 2 Ground
Relay 7	1 2	Input TTL 3 Ground
Relay 8	1 2	Input TTL 4 Ground

Table B-2. Relay/TTL Connector Pinouts

B.4 Automatic SRS Power Control

By using TTL connections, the power to a Self- Regenerating Suppressor (SRS) can be automatically switched off by a signal from the GP40 Gradient Pump or IP20 Isocratic Pump when the flow stops.

1. Go to the **PUMP OPTIONS** screen and set **TTL2 OUTPUT USAGE** to **0 Flow**. For detailed instructions, see the operator's manual for the GP40 Gradient Pump or IP20 Isocratic Pump.
2. Go to the ED40 **TTL FUNCTION IN** screen and assign **TTL3** to **SRS OFF/ON**.
3. Locate the twisted black and red wires (P/N 043598) and two green TTL connector plugs (P/N 921019) provided in the ED40 Ship Kit.
4. Strip the ends of the wires and insert them into the plugs. The signal wire (red) goes on top and the ground wire (black) goes on the bottom of each plug. Use the screwdriver (P/N 046985) provided in the Ship Kit to tighten the locking screws.
5. Plug one end of the cable into TTL2-OUT on the rear panel of the pump. The TTL connectors are located behind the upper door. Route the cable through the upper chases of the pump and the ED40. Plug the other end into TTL3 IN on the ED40 (see Figure B-6).
6. Turn off the flow, from the pump, and confirm that the SRS is automatically turned off by the detector.

C • User Interface

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C • User Interface

This appendix illustrates and describes all of the screens available for display on the front panel of the ED40 Electrochemical Detector. There are two categories of screens: operational and diagnostic (see Figure C-1).

- ***Operational*** screens enable you to create, edit, and run methods that control ED40 operation, and to select default parameters for the detector.
- ***Diagnostic*** screens provide access to diagnostic information and tests, including some screens that enable you to calibrate various ED40 functions.

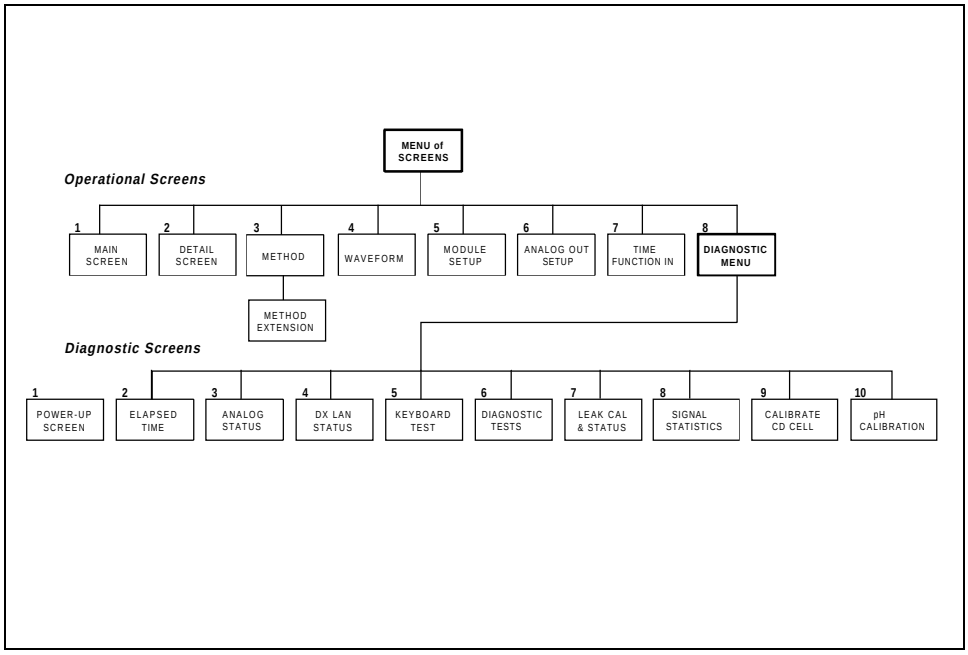


Figure C-1. ED40 Menu Structure

Operational screens are selected from the **MENU of SCREENS**. Diagnostic screens are selected from the **DIAGNOSTIC MENU**, an option on the **MENU of SCREENS**.

The detection mode is selected from the **MAIN** or **DETAIL** screen. The **MENU of SCREENS**, the **MAIN** screen, the **DETAIL** screen, the **METHOD** screen, and the **WAVEFORM** screen are unique to each of the detection modes. The remaining screens are common to all detection modes.

C.1 Operational Screens

C.1.1 Menu of Screens—Conductivity

The **MENU of SCREENS** provides top-level access to the menu structure.

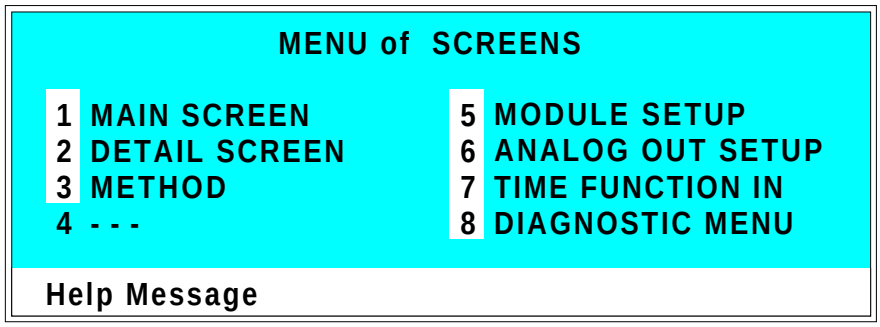


Figure C-2. Menu of Screens—Conductivity

There are two ways to select a screen from this menu:

- Use the cursor directional buttons to move the cursor to the field containing the screen number and press **Enter**.
- Press the number button on the front panel keypad that corresponds to the screen.

To display a brief description of each menu, press the **Help** button.

C.1.2 Main Screen—Conductivity

The **MAIN** screen displays the measured conductivity (Siemens) and other primary functions in large characters to make viewing easier from a distance.

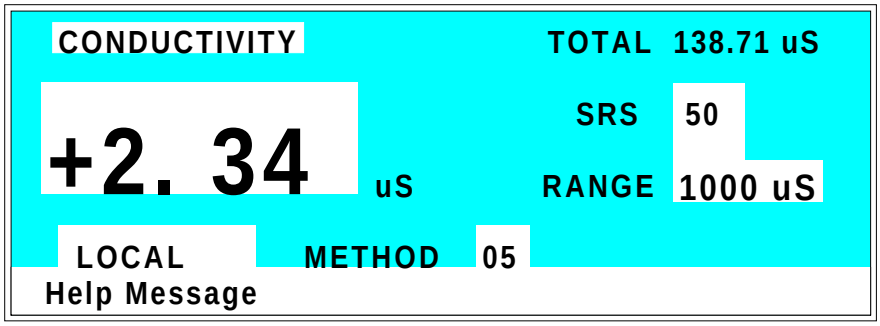


Figure C-3. Main Screen—Conductivity

Detector Operating Mode	Selects the detector operating mode: Conductivity, DC Amperometry, Integrated Amperometry, or Voltammetry.
TOTAL	Displays total conductivity (without an offset).
SRS	Selects the amount of current sent to the suppressor. Current selection depends on the eluent concentration, flow rate, etc. The discrete settings are Off, 50, 100, 300, and 500 mA.
RANGE	Sets the analog output scale factor. Select a range between 0.01 and 3000 µS.
METHOD	Sets the method number. Pressing **Select** and **Enter** sets the detector to Direct control.
Control Mode	Sets the detector to Local, Remote, or Locked Remote control.
MIN	Shows the method clock elapsed time.

C.1.3 Detail Screen—Conductivity

The **DETAIL** screen includes all the fields contained on the **MAIN** screen, as well as the TTL and Relay fields and fields to control detection. Refer to the **MAIN** screen for a description of fields that are common to both screens.

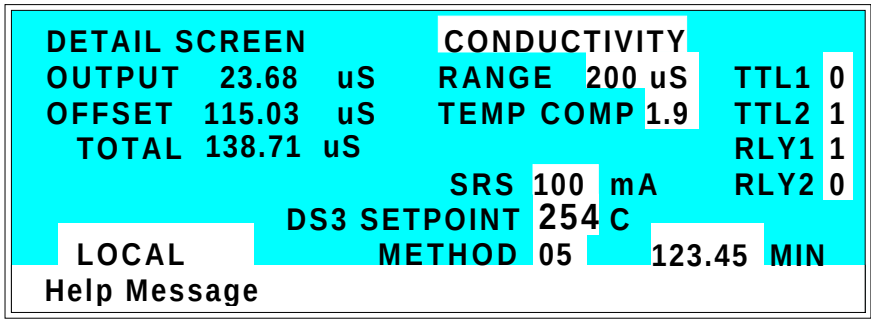


Figure C-4. Detail Screen—Conductivity

TEMP COMP — Sets the temperature compensation factor. The range is 0 to 3%. 1.7% is appropriate for most eluents.

DS3 SETPOINT — Sets the temperature of the DS3 Detection Stabilizer.

TTL1
TTL2 — Provides TTL control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

RLY1
RLY2 — Provides relay contact closure control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

C.1.4 Method—Conductivity

A method consists of a series of timed steps. Each step has a set of parameters associated with it.

Initial conditions are applied when a method is invoked. A lower case **v** next to the last step on the display indicates that the method contains more steps.

Blank fields denote no change from the previous step. The **Delete** button blanks the field.

NOTE

TEMP COMP, DS3 TEMP, and SRS CURRENT are not time-programmable.

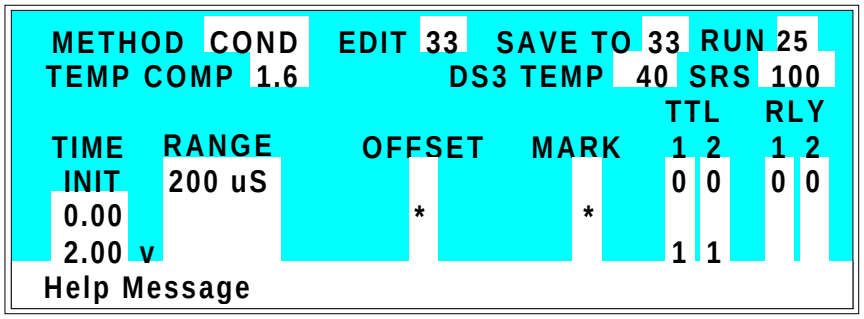


Figure C-5. Method Screen—Conductivity

Detector Operating Mode — Selects the detector operating mode: Conductivity, DC Amperometry, or Integrated Amperometry.

EDIT — Specifies the method number (0 through 99) to edit.

SAVE TO — Specifies the method number (0 through 99) to save the current method to.

RUN — Specifies the method number (0 through 99) to run. The **Hold/Run** button controls the running of the method.

TEMP COMP	Sets the Temperature Compensation factor.
SET TEMP	Sets the temperature for the DS3 Detection Stabilizer.
SRS	Sets the Self-Regenerating Suppressor current.
TIME	Specifies the start time for each step. Times are displayed chronologically.
RANGE	Sets the analog output range.
OFFSET	Stores the offset value. The baseline is set by subtracting the offset measured when this step is executed from all subsequent measurements. An asterisk (*) indicates that OFFSET will occur at this time.
MARK	Sends a positive pulse to the analog output (recorder) as an event marker. An asterisk (*) indicates that a MARK will occur at this time.
TTL	Sets TTL1 and TTL2 to off (0) or on (1).
RLY	Sets RLY1 and RLY2 to off (0) or on (1).

C.1.5 Menu of Screens—Integrated Amperometry

Figure C-6 shows the **MENU of SCREENS** for the Integrated Amperometry mode.

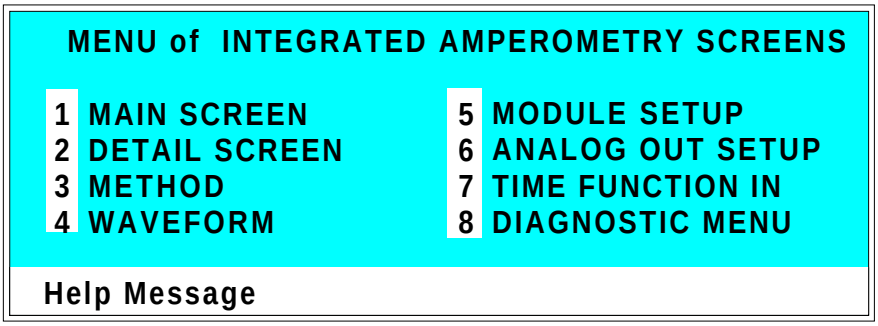


Figure C-6. Menu of Screens—Int. Amperometry

There are two ways to select a screen from this menu:

- Use the cursor directional buttons to move the cursor to the field containing the screen number and press **Enter**.
- Press the number button on the front panel keypad that corresponds to the screen.

To display a brief description of each menu, press the **Help** button.

C.1.6 Main Screen—Integrated Amperometry

The **MAIN** screen displays the measured charge (coulombs) and other primary functions in large characters to make viewing easier from a distance.

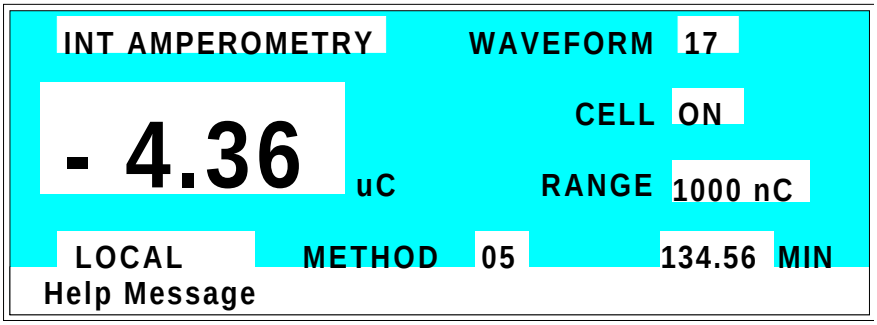


Figure C-7. Main Screen—Integrated Amperometry

Detector Operating Mode	Selects the detector operating mode: Conductivity, DC Amperometry, Integrated Amperometry, or Voltammetry.
WAVEFORM	Sets the waveform number to run. Program the waveform from the **WAVEFORM** screen.
CELL	Turns the amperometry cell off and on, disabling or enabling detection.
RANGE	Sets the detector sensitivity between 50 pC and 200 µC.
METHOD	Sets the method number. Pressing **Select** and **Enter** sets the detector to Direct control.
Control Mode	Sets the detector to Local, Remote, or Locked Remote control.
MIN	Shows the waveform clock elapsed time.

C.1.7 Detail Screen—Integrated Amperometry

The **DETAIL** screen includes all the fields contained on the **MAIN** screen, as well as the TTL and Relay fields and other fields to control detection. Refer to the **MAIN** screen for a description of fields common to both screens.

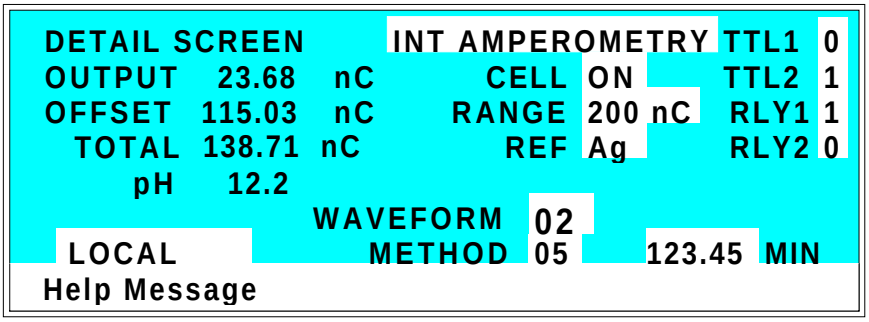


Figure C-8. Detail Screen—Integrated Amperometry

REF — Sets the reference electrode to use either the pH or Ag/AgCl half of the cell.

pH — Displays the mobile phase pH.

TTL1
TTL2 — Provides TTL control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

RLY1
RLY2 — Provides relay control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

C.1.8 Method—Integrated Amperometry

A method consists of a series of timed steps. Each step has a set of parameters associated with it.

Initial conditions are applied when a method is invoked. A lower case **v** next to the last step on the display indicates that the method contains more steps.

Blank fields denote no change from the previous step. The **Delete** button blanks the field.

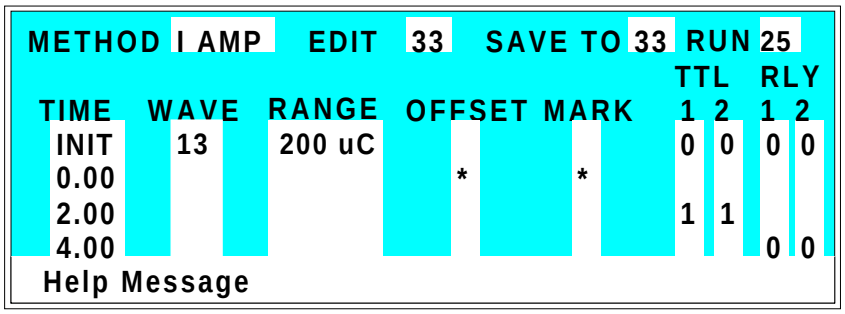


Figure C-9. Method Screen—Integrated Amperometry

Detector Operating Mode	Selects the detector operating mode: Conductivity, DC Amperometry, or Integrated Amperometry.
EDIT	Specifies the method number (0 through 99) to edit.
SAVE TO	Specifies the method number (0 through 99) to save the current method to.
RUN	Specifies the method number (0 through 99) to run. The **Hold/Run** button controls the running of the method.
TIME	Specifies the start time for each step. Times are displayed chronologically.

WAVE	Sets the waveform number (0 through 19) to run.
RANGE	Specifies the detection range.
OFFSET	Stores the offset value. The baseline is set by subtracting the offset measured when this step executes from all subsequent measurements. An asterisk (*) indicates that OFFSET will occur at this time.
MARK	Sends a positive pulse to the analog output (recorder) as an event marker. An asterisk (*) indicates a MARK will occur at this time.
TTL	Sets TTL1 and TTL2 to off (0) or on (1).
RLY	Sets RLY1 and RLY2 to off (0) or on (1).

C.1.9 Waveform—Integrated Amperometry

Figure C-10 shows the **WAVEFORM** screen for Integrated Amperometry. These entries form points on a plot of potential vs. time.

When the last step displayed is not the last step in the waveform, a lower case **v** is displayed beside the time digits in the last line.

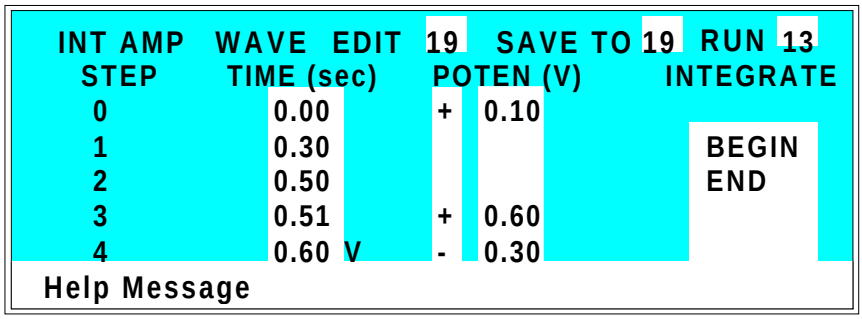


Figure C-10. Waveform Screen—Integrated Amperometry

WAVE EDIT — Specifies the waveform number (0 through 19) to edit.

SAVE TO — Specifies the waveform number (0 through 19) to save the current waveform definition to.

RUN — Specifies the waveform number (0 through 19) to run.

TIME — Specifies the time for this step. Times are displayed chronologically.

POTENTIAL — The left field sets the polarity; the right field is the point in the waveform for this voltage. No entry means the values from the preceding step remain in effect.

INTEGRATE	Sets the begin and ending times for the integration period. Integration cannot begin on the first waveform step, or end on the last step.

C.1.10 Menu of Screens—DC Amperometry

Figure C-11 shows the **MENU of SCREENS** for the DC Amperometry mode.

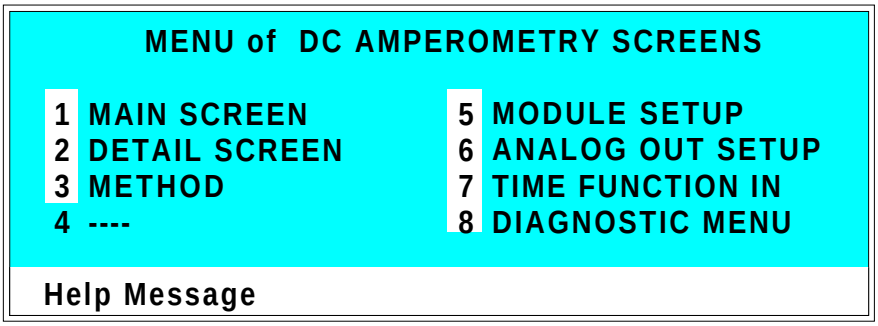


Figure C-11. Menu of Screens—DC Amperometry

There are two ways to select a screen from this menu:

- Use the cursor directional buttons to move the cursor to the field containing the screen number and press **Enter**.
- Press the number button on the front panel keypad that corresponds to the screen.

To display a brief description of each menu, press the **Help** button.

C.1.11 Main Screen—DC Amperometry

The **MAIN** screen displays the measured current (Amperes) and other primary functions in large characters to make viewing easier from a distance.

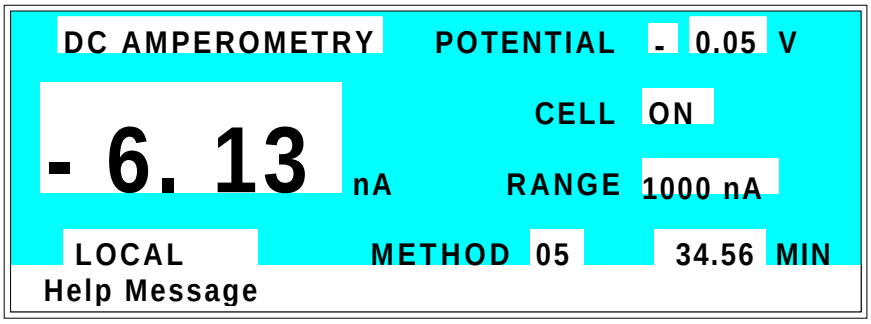


Figure C-12. Main Screen—DC Amperometry

Detector Operating Mode	Selects the detector operating mode: Conductivity, DC Amperometry, Integrated Amperometry, or Voltammetry.
POTENTIAL	The field on the left sets the polarity. The field on the right sets the applied potential.
CELL	Turns the amperometry cell off or on, disabling or enabling detection.
RANGE	Sets the detector sensitivity between 50 pA to 300 µA.
METHOD	Sets the method number. Pressing **Select** and **Enter** sets the detector to Direct control.
Control Mode	Sets the detector to Local, Remote or Locked Remote control.
MIN	Shows the method clock elapsed time.

C.1.12 Detail Screen—DC Amperometry

The **DETAIL** screen includes all the fields contained on the **MAIN** screen, as well as the TTL and Relay fields and other fields to control detection. Refer to the **MAIN** screen for a description of fields that are common to both screens.

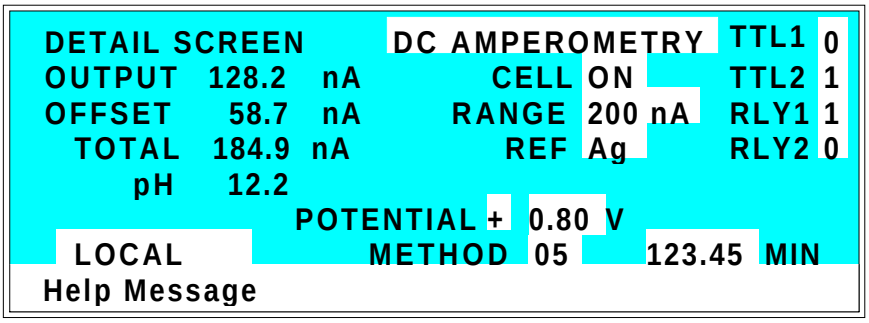


Figure C-13. Detail Screen—DC Amperometry

REF — Sets the reference electrode to use either the pH or Ag/AgCl half of the cell.

pH — Displays the mobile phase pH.

TTL1
TTL2 — Provides TTL control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

RLY1
RLY2 — Provides relay contact closure control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

C.1.13 Method—DC Amperometry

A method consists of a series of timed steps. Each step has a set of parameters associated with it.

Initial conditions are applied when a method is invoked. A lower case **v** next to the last step on the display indicates that the method contains more steps.

Blank fields denote no change from the previous step. The **Delete** button blanks the field.

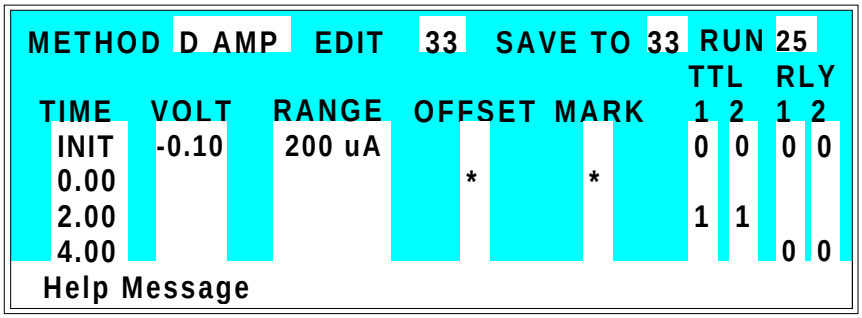


Figure C-14. Method Screen—DC Amperometry

Detector Operating Mode — Selects the detector operating mode: Conductivity, DC Amperometry, or Integrated Amperometry.

EDIT — Specifies the method number (0 through 99) to edit.

SAVE TO — Specifies the method number (0 through 99) to save the current method to.

RUN — Specifies the method number (0 through 99) to run. The **Hold/Run** button controls the running of the method.

TIME — Specifies the start time for each step. Times are displayed chronologically.

VOLT	Specifies the voltage applied to the cell.
RANGE	Specifies the detection range.
OFFSET	Stores the offset value. The baseline is set by subtracting the offset measured when this step executes from all subsequent measurements. An asterisk (*) indicates that OFFSET will occur at this time.
MARK	Sends a positive pulse to the analog output (recorder) as an event marker. An asterisk (*) indicates that a MARK will occur at this time.
TTL	Sets TTL1 and TTL2 to off (0) or on (1).
RLY	Sets RLY1 and RLY2 to off (0) or on (1).

C.1.14 Menu of Screens—Voltammetry

Figure C-15 shows the **MENU of SCREENS** for the Voltammetry mode.

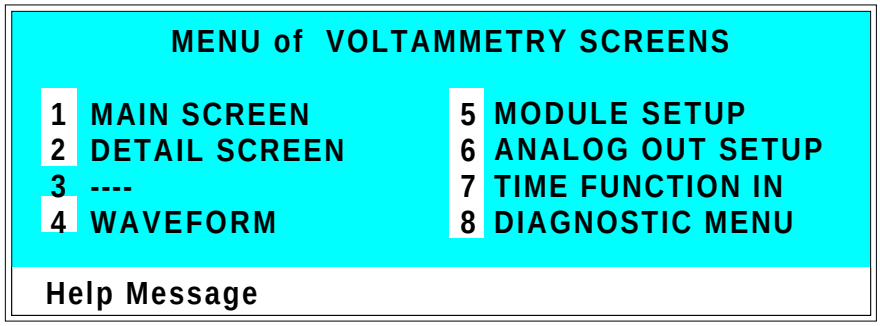


Figure C-15. Menu of Screens—Voltammetry

There are two ways to select a screen from this menu:

- Use the cursor directional buttons to move the cursor to the field containing the screen number and press **Enter**.
- Press the number button on the front panel keypad that corresponds to the screen.

To display a brief description of each menu, press the **Help** button.

C.1.15 Main Screen—Voltammetry

The **MAIN** screen displays the measured current (Amperes) and other primary functions in large characters to make viewing easier from a distance.

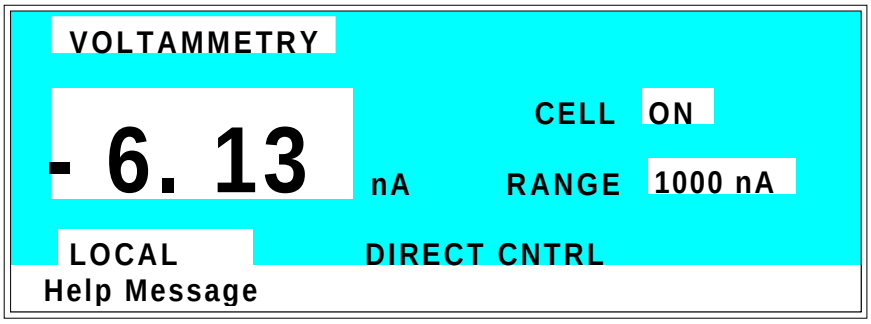


Figure C-16. Main Screen—Voltammetry

Detector Operating Mode	Selects the detector operating mode: Conductivity, DC Amperometry, Integrated Amperometry, or Voltammetry.
CELL	Turns the amperometry cell off or on, disabling or enabling detection.
RANGE	Sets the detector sensitivity between 1 nA to 100 µA.
DIRECT CNTRL	In Voltammetry mode, the detector is always in Direct control mode. Because methods do not apply to Voltammetry, this field cannot be changed.
Control Mode	Sets the detector to Local, Remote, or Locked Remote control.

C.1.16 Detail Screen — Voltammetry

The **DETAIL** screen includes all the fields contained on the **MAIN** screen, as well as the TTL and Relay fields and other fields to control detection. Refer to the **MAIN** screen for a description of fields that are common to both screens.

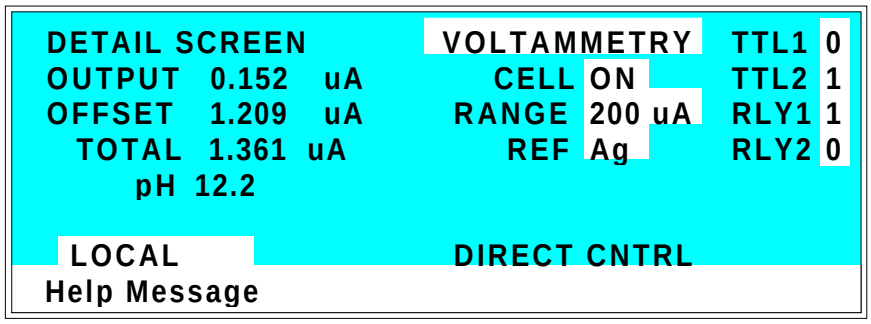


Figure C-17. Detail Screen—Voltammetry

REF — Sets the reference electrode to use either the pH or Ag/AgCl half of the cell.

pH — Displays the mobile phase pH.

TTL1
TTL2 — Provides TTL control of accessories. In a method, these values occur according to the method timing. In Direct control, select off (0) or on (1).

RLY1
RLY2 — Provides relay control of accessories. Select off (0) or on (1).

C.1.17 Waveform—Voltammetry

Figure C-18 shows the **WAVEFORM** screen for Voltammetry. These entries form points on a plot of potential vs. time.

When the last step displayed is not the last step in the waveform, a lower case **v** is displayed beside the time digits in the last line.

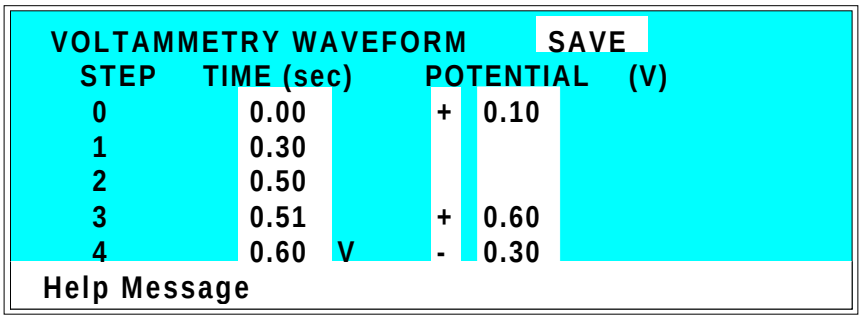


Figure C-18. Waveform Screen—Voltammetry

TIME — Specifies the time for this step to begin. Times must be entered chronologically.

POTENTIAL — The left field sets the polarity; the right field is the point in the waveform for this voltage. No entry means the values from the preceding step remain in effect.

SAVE — Move the cursor to this field and press **Enter** to save the waveform.

C.1.18 Module Setup

The backlight intensity, key beep, and error beep are configured from the **MODULE SETUP** screen.

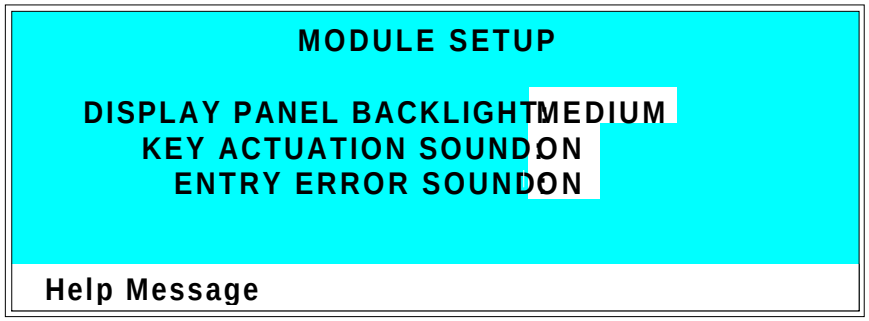


Figure C-19. Module Setup Screen

DISPLAY PANEL BACKLIGHT	Sets the display panel backlight to **LOW**, **MEDIUM**, **HIGH**, or **OFF**.
KEY ACTUATION SOUND	Toggles the keypad touch sound. When this option is selected, the detector sounds a beep when a button is pressed.
ENTRY ERROR SOUND	Toggles the error sound. When this option is selected, the detector sounds a beep when an invalid entry is made.

C.1.19 Analog Out Setup

The **ANALOG OUT SETUP** screen contains parameters for setting the analog output, such as for a recorder or oscilloscope.

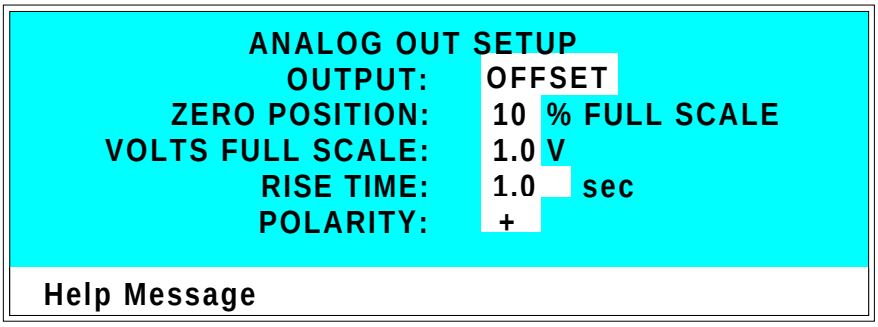


Figure C-20. Analog Out Setup Screen

OUTPUT — Sets the analog output to one of the following:
OFFSET uses the offset level value.
TOTAL sets the detector output to the total cell conductivity, charge or current, disabling the offset function.
ZERO sets the detector output to zero volts.
FULL SCALE sets the output to the full- scale setting.

ZERO POSITION — Sets the analog (recorder) offset level (0 to 100%).

VOLTS FULL SCALE — Sets the full-scale voltage for a signal equal to the range setting. The discrete settings are 1.0, 0.1, and 0.01 volts.

RISE TIME — Sets the analog output filter rise time to 0.05, 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, or 10.0 seconds. Rise time is a measure of how quickly the detector responds to a change in signal. The filter is a Bessel filter that ensures optimal noise rejection and low signal distortion.

POLARITY	Determines the output voltage polarity. The output is usually set to positive polarity. Negative polarity is used to reverse peaks from indirect detection.

C.1.20 Time Function In

Four input TTL controls may be assigned to the time input functions via the **TIME FUNCTION IN** screen. Control is enabled when Local mode is selected.

Use the **Select** buttons to assign TTL1 through TTL4 to any four of the seven time functions. TTL input ports can also be assigned to the **INCR** and **DECR** fields, to increase or decrease the method number.

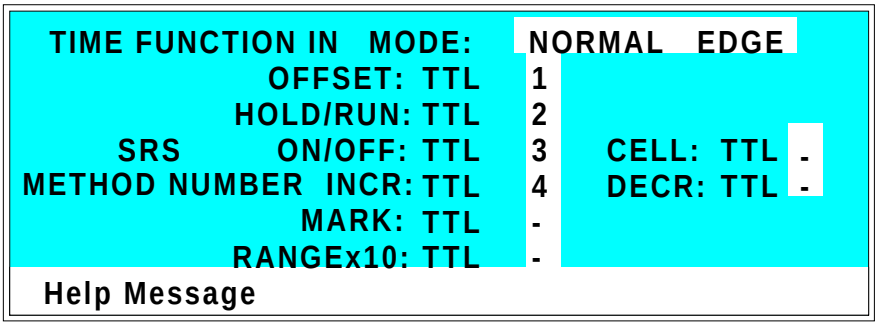


Figure C-21. Time Function In Screen

C.2 Diagnostic Screens

To access the ED40 diagnostics, select the **DIAGNOSTIC MENU** from the **MENU of SCREENS**. While a diagnostic screen is displayed, pressing the **Menu** button returns you to the **DIAGNOSTIC MENU**. From the **DIAGNOSTIC MENU**, pressing **Menu** returns you to the **MENU of SCREENS**.

C.2.1 Diagnostic Menu

The **DIAGNOSTIC MENU** lists the available diagnostic screens. Figure C-22 shows all of the possible menu options; however, the options actually displayed will depend on which detection mode is selected.

- Option 7, **LEAK CAL(IBRATION) & STATUS**, is available only in Conductivity mode and when a leak detector is connected.
- Option 9, **CALIBRATE CD CELL**, is available only in Conductivity mode.
- Option 10, **pH CALIBRATION**, is available only in Integrated Amperometry, DC Amperometry, and Voltammetry modes.

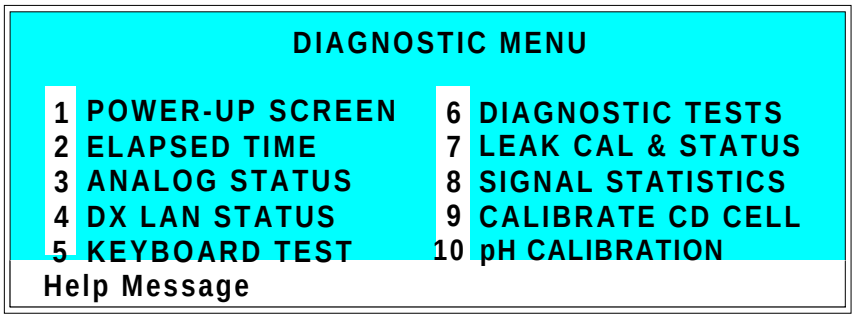


Figure C-22. Diagnostic Menu Screen

C.2.2 Power-Up Screen

This screen displays the revision numbers for the Moduleware and BIOS code. It also displays the identification number of the optional DX LAN interface, if connected.

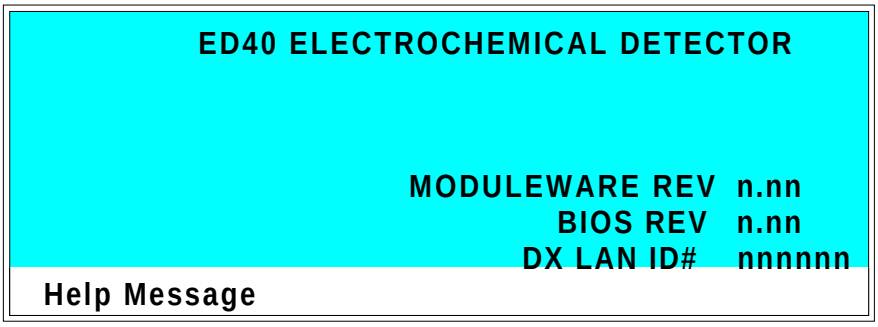


Figure C-23. Power-Up Screen

C.2.3 Elapsed Time

This screen reports for how long various ED40 parameters have been in use. The status of each parameter updates in real time.



Figure C-24. Elapsed Time Screen

MODULE ON — Reports the total time the detector has been powered up in its lifetime.

BACKLIGHT — Reports the total time the LCD backlight has been on in its lifetime.

SRS ON — Reports the total time the SRS has run. Reset this field to zero after changing the SRS.

EC CELL ON — Reports the total time the amperometry cell has run. Reset this field to zero after replacing the cell.

RESET — Allows a timer to be reset to zero to restart the counter. Reset the appropriate timer after installing a new component.

C.2.4 Analog Status

This screen reports the status of several analog test points. If a run is in progress, the status updates are from the last idle state. If no run is in progress, the values are in real time.

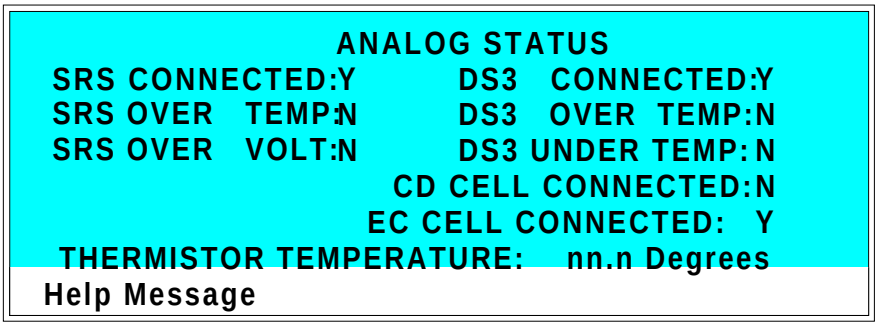


Figure C-25. Analog Status Screen

SRS CONNECTED	Indicates whether a Self-Regenerating Suppressor is connected to the detector.
SRS OVER TEMP	Indicates whether the SRS is over the temperature specified.
SRS OVER VOLT	Indicates whether the SRS is over the voltage range specified.
DS3 CONNECTED	Indicates whether a DS3 Detection Stabilizer is connected to the detector.
DS3 OVER TEMP	Indicates whether the DS3 is over the temperature specified.
DS3 UNDER TEMP	Indicates whether the DS3 is under the temperature specified.
CD CELL CONNECTED	Indicates whether a conductivity cell is connected to the detector.
EC CELL CONNECTED	Indicates whether an amperometry cell is connected to the detector.

THERMISTOR TEMPERATURE	Indicates the temperature recorded from the conductivity cell thermistor.

C.2.5 DX LAN Status

The DX LAN driver monitors several types of errors that may occur on the network. The detector reads the DX LAN error counts and displays them on this screen, along with the DX LAN addresses. Most errors are caused by a defective cable, missing or incorrectly terminated cable. The DX LAN cable must be RG58U or RG58AU.

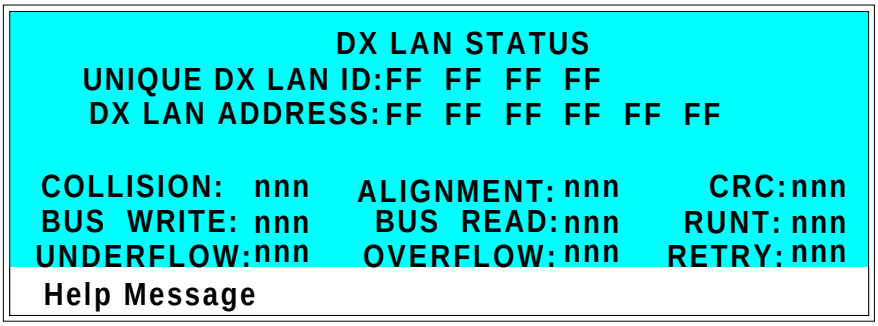


Figure C-26. DX LAN Status Screen

UNIQUE DX LAN ID — Displays the three-byte DX LAN ID programmed into the detector Moduleware (in HEX). The ID is unique to the detector and never changes.

DX LAN ADDRESS — Displays the six-byte DX LAN address assigned by the PC (in HEX). The first three bytes are the system assignment and the last three bytes are the assignment within the system.

COLLISION — Indicates that 16 unsuccessful transmissions of the same packet occurred, due to collisions.

BUS WRITE — Indicates that a ready response could not be issued within 2.4 microseconds after the WR signal was asserted. This occurs when the transmit buffer memory is full.

UNDERFLOW	Indicates that data from the transmit section of the hardware buffer memory is not available for serial transmission. The DX LAN will continue to send out this data frame.
ALIGNMENT	Indicates that a packet was received with an alignment error, meaning that there were one to seven extra bits at the end of the packet. This is usually caused by a collision or a faulty transceiver.
BUS READ	Indicates that a ready response could not be issued within 2.4 microseconds after the ready signal was asserted. This occurs when reading an empty buffer.
OVERFLOW	Indicates that the DX LAN hardware receive buffer became full and had to reject a packet for lack of space.
CRC	Indicates that a packet was received with a CRC error. This usually means that a collision has corrupted the packet.
RUNT	Indicates that a “runt” packet (one less than 15 bytes in length) was received. This usually occurs after a collision has truncated the original length.
RETRY	Indicates the number of retries required to transmit the last packet.

C.2.6 Keyboard Test

You can conduct an interactive test of the ED40 front panel keypad from this screen. Pressing any front panel button changes the corresponding button indicator on the screen to reverse video. This confirms proper operation of that button. When you release the button, the display returns to normal video.

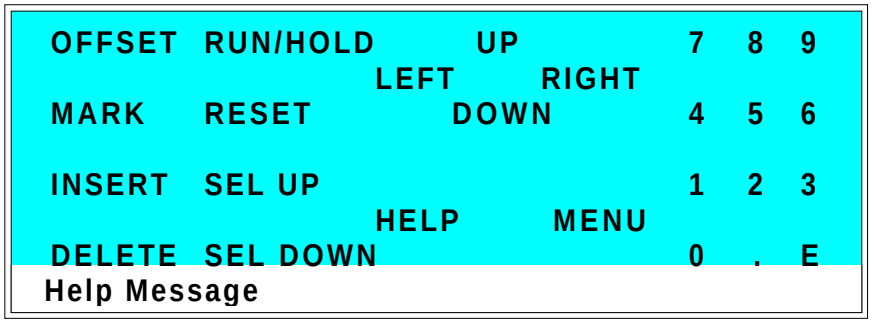


Figure C-27. Keyboard Test Screen

C.2.7 Diagnostic Tests

These tests verify the integrity of the detector electronics and functions.

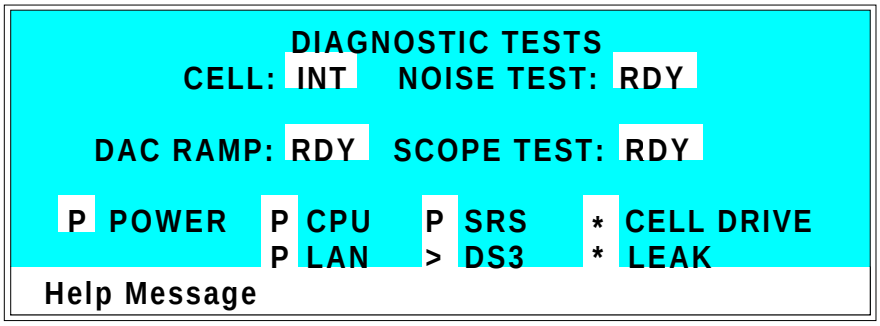


Figure C-28. Diagnostic Tests Screen

CELL — Specifies whether the cell to be tested is internal (INT) or external (EXT).

NOISE TEST — Selects a cell for noise testing. The test modes are:

RDY—The system is idle and ready to begin a test.

CD—Selects a conductivity cell for testing.

EC—Selects an amperometry cell for testing.

DAC RAMP — Selects the DAC test mode. The test linear ramps the output of the selected DA on the SP or SCR card from 0 to full-scale and can be plotted from the recorder output or from PeakNet. The ramp test modes are:

RDY—The system is idle and ready to begin a test.

CDO—Tests the CD Offset DAC (eight iterations, one for each gain; the eighth iteration is flat on top). Disconnect the conductivity cell before running this test.

REC—Tests the Recorder Output DAC.

ECO—Tests the EC Offset DAC (two iterations; one for the ADC fine channel and one for the ADC coarse channel). Disconnect the amperometry cell before running this test.

ECD—Tests the EC Drive DAC. A full-scale ramp is repeated over and over until you exit the test or the screen. Disconnect the amperometry cell before running this test.

SCOPE TEST Generates test waveforms that can be monitored on test points on the SCR and SP cards, using an oscilloscope or chart recorder. The test modes are:

RDY—The system is idle and ready to begin a test.

CDF—Tests the CD Offset DAC output (TP9 on the SP card) and the CD fine filter circuit (TP19 on the SP card). TP9 outputs a square wave with a frequency of 200 ms and an amplitude from 0 to -0.1 V. TP19 outputs a sine wave with a frequency of 200 ms. The peak-to-peak amplitude is 4.40 V.

CDT—Tests the CD Temperature Compensation DAC (TP26 on the SP card). The output is a square wave in which the bottom and top amplitudes increase and decrease from 11 V to 12 V. Before running this test, connect the DS3 and set the temperature to 45 °C, or connect a dummy thermistor value of 42 kΩ to pins 2 and 3 of the conductivity cell connector.

SCR—Tests the SCR DAC for the DS3 set point temperature (TP13 on the SCR card) and SRS current set point (TP17 on the SCR card). The output on each test point is a linear ramp that goes from 0 to 10 V in 2.5 seconds. Disconnect the DS3 and SRS before running this test.

PHD—Tests the pH DAC (TP10 on the SP card). The output is a waveform with an increasingly linear ramp that goes from approximately 0 to 0.87 VDC in 31 seconds, with a steep slope for the first 5.5 seconds of the ramp. The decreasing side of the ramp is 25.5 seconds in duration.

The following tests can be run from the **DIAGNOSTIC TESTS** screen. To begin a test, select the asterisk (*) in the field beside the test name and press **Enter**. When the test is complete, the entry field displays either **P** (pass) or **F** (fail).

NOTE

Running the following tests disturbs the equilibration of the detector as services cycle on and off and the system runs through its test modes. After testing, allow the system to equilibrate before resuming operation.

- POWER—Checks the +5, ±15, and +24 volt monitor on the Relay card.
- CPU—Checks the CPU internal configuration and the Moduleware checksum.
- DX LAN—Checks the DX LAN hardware configuration and loop-back.
- SRS—Varies the SRS power requirements and checks the flags.
- DS3—Varies the DS3 temperature set point and checks the flags.
- CELL DRIVE—Switches in a resistor in place of the cell to provide a calibrated signal to read.
- LEAK—Checks the detector's leak sensor for a correct, open circuit, or short circuit condition.

C.2.8 Leak Sensor Calibration and Status

This screen reports the status of leak sensor parameters.

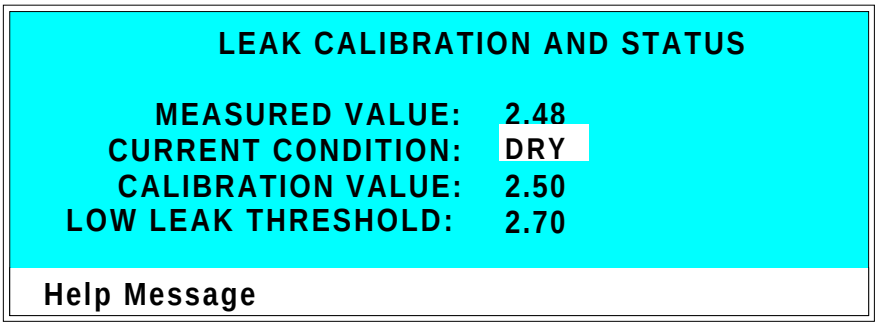


Figure C-29. Leak Calibration and Status Screen

MEASURED VALUE	Reports the measured voltage from the leak sensor.
CURRENT CONDITION	Reports the current (error) condition of the leak sensor: WET, DRY, or ERR. Error indicates an open or short circuit. To calibrate a leak sensor, select **CAL** and press **Enter**. After calibration, the field will revert to DRY, unless an error condition exists.
CALIBRATION VALUE	Reports the value saved when the leak sensor was last calibrated.
LOWER THRESHOLD	Reports the threshold value below which a leak is indicated. This is based on the calibration value.

C.2.9 Signal Statistics

This screen enables you to monitor the selected input to the A/D circuitry. When the screen is first displayed, the **MAX** and **MIN** status values are equal to the **INPUT** value, and **DURATION** reads 0. Status values are reported in A/D volts and are updated dynamically.

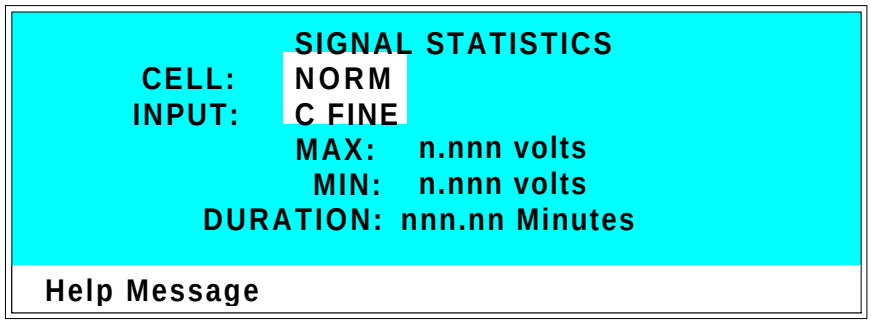


Figure C-30. Signal Statistics Screen

CELL — Selects the cell test mode:
NORM—Uses input data from the cell.
FIXED—Uses a fixed dummy cell for input.

INPUT — The measured data from the A/D circuit:
C FINE—Conductivity cell fine input
C COARSE—Conductivity cell coarse input
C THER—Conductivity cell thermistor input
E FINE—Amperometry cell fine input
E COARSE—Amperometry cell coarse input
E pH—Amperometry cell pH input
LEAK—Leak detector input
DRIVE—Cell drive circuit input

MAX — The maximum input data value during the duration of the test.

MIN — The minimum input data value during the duration of the test.

DURATION	The duration (in minutes) of the test. The test starts when you enter this screen and terminates when you exit.

C.2.10 Calibrate Conductivity Cell

This screen allows you to calibrate the conductivity cell with 1 mM KCl.

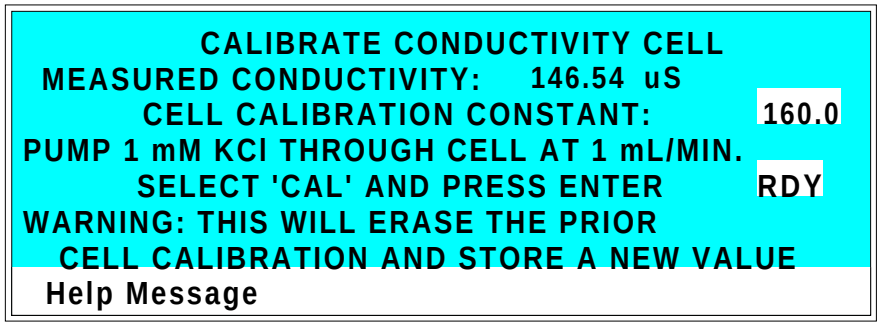


Figure C-31. Calibrate Conductivity Cell Screen

CONDUCTIVITY — Reports the measured conductivity from the conductivity cell.

Calibrate — Select **CAL** and press **Enter** to calibrate the conductivity cell to the value displayed in the conductivity field. The calibration assumes that the cell is full of 1 mM KCl. The new value replaces the previous calibration value.

C.2.11 pH Calibration

This screen allows you to calibrate the pH reference electrode in the amperometry cell with either Na correction or linear (no correction). The steps in this screen must be performed in sequence. Two calibrations are required.

- Do the first calibration at pH 7.0. With the electrode in pH 7.0 buffer solution, verify that the **MEASURED pH** field displays 7.0. If not, press **Select** and then **Enter**.
- Do the second calibration with another buffer solution of known pH; for example, pH 10.0.

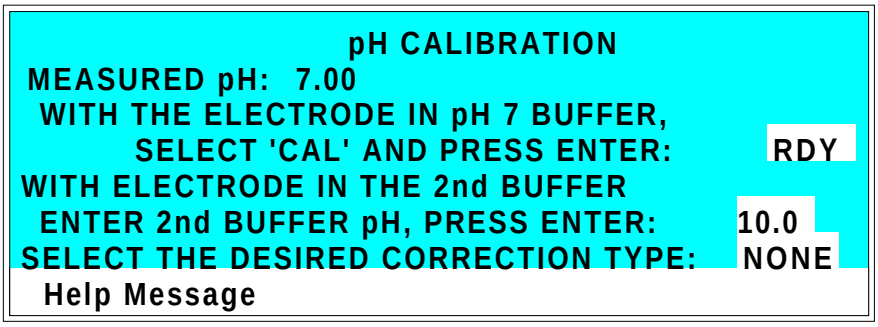


Figure C-32. pH Calibration Screen

MEASURED pH	Reports the pH measured from the electrode.
SELECT CAL	**RDY** indicates no selection has been made. Calibration will not occur. Select **CAL** and press **Enter** to calibrate the electrode for pH 7.0.
2nd BUFFER	Enter the known pH of the second buffer solution. With the reference electrode in the second buffer solution, press **Enter** to perform the second calibration.
CORRECTION TYPE	Select **Na** to apply a correction for sodium.

D • Signal Processor Functions

D • Signal Processor Functions

Table D-1 lists the functions of the Signal Processor (SP) card.

Function	Cond.	DC Amp.	Int. Amp.	Volt.
Temperature compensation digital-to-analog converter	X			
Cell chopper, driver	X			
Offset digital-to-analog scaling switch	X			
Conductivity signal receiver	X			
Second-stage amplifier and gain switch	X			
Synchronous rectifier	X			
5 mS noise filter	X	X		X
DC amplifier 100 mS filter	X	X		
Bipolar cell drive analog-to-digital filter		X	X	X
Amperometry cell drive smoothing filter		X	X	X
pH slope and offset corrector		X	X	X
Cell receiver/driver		X	X	X
Fine and coarse integrators			X	
Signal selection (MUX)	X	X	X	X
16-bit analog-to-digital converter	X	X	X	X
Digital interface	X	X	X	X

Table D-1. SP Card Functions

E • Connector Pinouts

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E • Connector Pinouts

E.1 Recorder/Diagnostic Signal Pinouts

This section describes the Recorder/Diagnostic Signal pinouts. The connector is supplied plugged into the socket. The pins are numbered consecutively from 1 to 10 (top to bottom).

Pin	Signal
1	Recorder Negative
2	Recorder Positive
3	SRS Supply Voltage
4	DS3 Supply Current
5	Conductivity Cell Flow Stream Temperature
6	Amperometry Cell Flow Stream pH
7	Amperometry Cell Voltage, Working to Reference Electrode (polarity is reversed).*
8	+10 V Reference from SCR Card
9	Integrated Amperometry Scope Sync. Pulse
10	Common Ground for Signals 3 through 9

Table E-1. Recorder/Diagnostic Cable Pinouts

E.1.1 Signal Electrical Parameters

Pin 1 Attach the negative input of the chart recorder to pin 1.

Pin 2 The full-scale output range setting (0 to 0.01 V, 0 to 0.1 V, or 0 to 1.0 V) is selected from the **ANALOG OUTPUT** screen.

The output resistance is 1 to 2 kΩ.

Pin 3 The voltage is equal to that developed across the SRS. The SRS is driven by a regulated current source.

The output resistance is 10 kΩ.

Pin 4 The voltage is proportional to the DS3 heater current. The maximum heater power produces approximately 1.2 V.

The output resistance is 5 kΩ.

Pin 5 The voltage is related to the inverse exponential of the temperature. Table E-2 lists the conductivity flow stream temperatures.

The output resistance is 1 kΩ.

°C	Volts	Slope (-mV/°C)
0	2.00	88
5	1.71	75
10	1.42	63
15	1.18	52
20	0.97	43
25	0.80	35
30	0.65	28
35	0.53	23
40	0.45	20
45	0.35	15
50	0.30	13

Table E-2. Conductivity Flow Stream Temperatures

Pin 6 The voltage is proportional to the pH, after calibration, as indicated in Table E-3.

The output resistance is 900 Ω.

pH	Volts
0	-0.7
1	-0.6
2	-0.5
3	-0.4
4	-0.3
5	-0.2
6	-0.1
7	0.0
8	0.1
9	0.2
10	0.3
11	0.4
12	0.5
13	0.6
14	0.7

Table E-3. Amperometry Cell Flow Stream Temperatures

Pin 7 The voltage is equal to the applied amperometry cell voltage.

The output resistance is 790 Ω. (The polarity is reversed.)

Pin 8 The signal is 10.00 ± 0.01 V.

The output resistance is 0 Ω.

The maximum load current capability is 10 mA. This output may be divided with a resistor network to provide an offset to a monitoring device. Shorting or overloading this output will disrupt operation of the SCR module.

Pin 9 0 to 5 V logic pulse of 1 mS duration at the start of each integrated amperometry waveform cycle (time zero on the **WAVEFORM** screen).

The output resistance is approximately 100 Ω.

The maximum current is approximately 10 mA.

Pin 10 A signal ground (0 volts) for monitoring only. Avoid connecting this pin to any grounds or sources of AC or DC current. This ground may be used in common for any of the signals on pins 3 through 9.

E.2 TTL/Relay Pinouts

The TTL and Relay connectors are on the DX LAN/Relay card. The TTL and Relay connectors all have the same pinout configuration; be careful to use the correct connector.

Connector Number	Pin Number	Description
Relay 1	1 and 2	Solid State Relay Contacts Out
Relay 2	1 and 2	Solid State Relay Contacts Out
Relay 3	1 2	TTL Out (1 kΩ pull up to +5, 100 mA sink) Ground
Relay 4	1 2	TTL Out (1 kΩ pull up to +5, 100 mA sink) Ground
Relay 5	1 2	Input TTL 1 Ground
Relay 6	1 2	Input TTL 12 Ground
Relay 7	1 2	Input TTL 3 Ground
Relay 8	1 2	Input TTL 4 Ground

Table E-4. TTL/Relay Connector Pinouts

E.3 DS3 Connector Pinouts—SCR

The DS3 Detection Stabilizer connects to the SCR card. The connector is a double-row, 1.6 cm (0.63 in) latching displacement connector.

Pin Number	Description
1	DS3 Ground (body)
2	+24 V
3	Base Drive
4	Emitter
5	Emitter
6	Thermistor Ground
7	Thermistor
8	Leak +5 V
9	Leak
10	Lead Ground
11	Disconnect
12	Disconnect Ground (24-gauge)

Table E-5. DS3 Connector Pinouts

E.4 SRS Connector Pinouts—SCR

The SRS (Self-Regenerating Suppressor) connects to the SCR card. The connector is a double-row, 0.84 cm (0.33 in) latching displacement connector.

Pin Number	Description
1	Ground
2	Thermistor
3	SRS Positive
4	SRS Negative
5	Disconnect
6	Disconnect (Ground)

Table E-6. SRS Connector Pinouts

E.5 Amperometry Cell Connector Pinouts—SP

The amperometry cell connects to the SP card. The amperometry cell connector is a single-row, shielded BERG-type latching connector. With the connector on the cable facing you and the open metal shield on the left, pin 1 is at the top.

Pin Number	Description	Color (inside cable)
1	Ground, Counterelectrode	Black
2	Working Electrode	Red
3	pH Reference Electrode pre-amp out	Brown
4	Ag/AgCl Reference Electrode pre-amp out	Orange
5	+15 V	Yellow
6	-15 V	Blue
7	Amperometry Cell Disconnect	Green
8	Spare	Purple

Table E-7. Amperometry Cell Connector Pinouts

E.6 Conductivity Cell Connector Pinouts—SP

The conductivity cell connects to the SP card. The connector is a single-row, shielded, BERG-type latching connector.

Pin Number	Description	Color (inside cable)
1	Cell Drive	Red COAX
2	Ground (Thermistor and Shield)	All COAX
3	Thermistor	Black COAX
4	Cell Return	Red COAX
5–8	Grounded (Plug Body Shield)	On receptacle only, not on plug

Table E-8. Conductivity Cell Connector Pinouts

F • Further Reading

F • Further Reading

Ion Chromatography and Conductivity Detection

1. Weiß, J. Ionen Chromatographie 2nd ed., VCH, Weinheim, 1991.
2. Dasgupta, P. K. "Ion chromatography: the state of the art." *Anal. Chem.* **64** (1992), 775A.
3. Dietrich, A. M. et al. "Determination of chlorite and chlorate in chlorinated and chloraminated drinking water by flow injection analysis and ion chromatography." *Anal. Chem.* **64** (1992), 496.
4. Henshall, A.; Rabin, S.; Statler, J.; Stillian, J. "A recent development in ion chromatography detection: The self-regenerating suppressor." *American Lab* **24** (Nov. 1992), 20R.
5. Slingsby, R. W.; Rey, M. A. OmniPac Guidebook: Methods Development and Troubleshooting. Dionex, Sunnyvale, CA 1991 (Dionex LPN 034517).
6. Haddad, P. R.; Jackson, P. E. Ion Chromatography: Principles and Applications. Elsevier, Amsterdam, 1990.
7. Small, H. Ion Chromatography. Plenum, New York, 1989 (Dionex LPN 034327).

Voltammetry and Principles of Electrochemistry

8. Kissinger, P. T.; Heineman, W. R. Laboratory Techniques in Electroanalytical Chemistry. Marcel Dekker, New York, 1984.
9. Plambeck, J. A. Electroanalytical Chemistry: Basic Principles and Applications. Wiley, New York, 1982.

DC Amperometry

10. Liu, Y.; Rocklin, R. D.; Joyce, R. J.; Doyle, M. J. "Photodissociation/gas diffusion/ion chromatography system for determination of total and labile cyanide in waters." *Anal. Chem.* **62** (1990), 766.

11. Kim, H. "Determination of sulfites in foods by ion exclusion chromatography with electrochemical detection: a collaborative study." *J. Assoc. Off. Anal. Chem.* **73** (1990), 216.

12. Kim, H. "Determination of total vitamin C by ion exclusion chromatography with electrochemical detection." *J. Assoc. Off. Anal. Chem.* **72** (1989), 681.

13. Radzuk, D. M.; Lunte, S. M. "Application of liquid chromatography/electrochemistry in pharmaceutical and biochemical analysis: a critical review." *CRC Crit. Rev. Anal. Chem.* **20** (1989), 317.

14. Kissinger, P. T. "Biomedical applications of liquid chromatography/electrochemistry." *J. Chromatogr.* **488** (1989), 31.

15. Ehrenström, F. "Determination of catechols in small volumes of plasma using ion-pair reversed-phase liquid chromatography/ electrochemistry." *Life Sci.* **43** (1988), 615.

16. Parvez, H. et al, eds. *Progress in HPLC, Vol. 2* Electrochemical Detection in Medicine and Chemistry. VNU Science Press, Utrecht, Neth. 1987.

17. Selavka, C. M.; Krull, I. S. "The forensic determination of drugs of abuse using liquid chromatography with electrochemical detection: a review." *J. Liq. Chromatogr.* **10** (1987), 345.

18. Han, K.; Koch, W. F.; Pratt, K. W. "Improved procedure for the determination of iodide by ion chromatography with electrochemical detection." *Anal. Chem.* **59** (1987), 731.

19. Rocklin, R. D.; Johnson, E. L. "Determination of cyanide, sulfide, bromide and iodide by ion chromatography with electrochemical detection." *Anal. Chem.* **55** (1983), 4.

Pulsed and Integrated Amperometry

20. "Analysis of carbohydrates by high-performance anion-exchange chromatography with pulsed amperometric detection (HPAE-PAD)." Technical Note 20, Dionex Corp., 1993 (LPN 032857).

21. "Optimal settings for pulsed amperometric detection of carbohydrates using Dionex pulsed electrochemical and amperometric detectors." Technical Note 21, Dionex Corp., 1993 (LPN 034889).

22. "Monosaccharide and Oligosaccharide analysis of glycoproteins electrotransferred onto polyvinylidene fluoride (PVDF) membranes." Technical Note 30, Dionex Corp., 1993 (LPN 034883).

23. LaCourse, W. R.; Johnson, D. C. "Optimization of waveforms for pulsed amperometric detection of carbohydrates based on pulsed voltammetry." *Anal. Chem.* **65** (1993), 50.

24. Vandeberg, P. J.; Johnson, D. C. "Comparison of pulsed amperometric detection and integrated voltammetric detection for organic sulfur compounds in liquid chromatography." *Anal. Chim. Acta* **290** (1994), 317.

25. Jackson, W. A.; LaCourse, W. R.; Dobberpuhl, D. A.; Johnson, D. C. "The voltammetric response of ethanolamine at gold electrodes in alkaline media." *Electroanalysis* **3** (1991).

26. LaCourse, W. R.; Johnson, D. C.; Rey, M. A.; Slingsby, R. W. "Pulsed amperometric detection of aliphatic alcohols in liquid chromatography." *Anal. Chem.* **63** (1991), 134.

27. Townsend, R. R.; Hardy, M. R. "Analysis of glycoprotein oligosaccharides using high-pH anion exchange chromatography." *Glycobiology* **1** (1991), 139.

28. Johnson, D. C.; LaCourse, W. R. “Liquid chromatography with pulsed electrochemical detection at gold and platinum electrodes.” *Anal. Chem.* **62** (1990), 589A.

29. LaCourse, W. R.; Jackson, W. A.; Johnson, D. C. “Pulsed amperometric detection of alkanolamines following ion-pair chromatography.” *Anal. Chem.* **61** (1989), 2466.

30. Rocklin, R. D. “Ion Chromatography with pulsed amperometric detection: Simultaneous determination of formic acid, formaldehyde, acetaldehyde, propionaldehyde, and butyraldehyde.” *Adv. in Chem. Series 210*, Ch. 2, p. 13, Formaldehyde: Analytical Chemistry and Toxicology, V. Turoski, Ed. ACS, 1985.

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