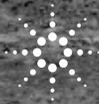


# Agilent 1049A Electrochemical Detector

## Operator's Handbook



Agilent Technologies

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## WARNING

For details of safety,  
see Safety Information  
on page 181.

### Warning Symbols Used In This Book



The apparatus is marked  
with this symbol when  
the user should refer to  
the instruction manual  
in order to protect the  
apparatus against  
damage.

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**Operator's Handbook**

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# Using This Handbook

This handbook is for the Agilent 1049A electrochemical detector. It describes installation, operation, maintenance and troubleshooting of your detector and is designed to be used as both a beginner's self-teaching guide and a day-to-day reference guide.

Chapter 1 "Preparing Your Liquid Chromatograph" describes how to prepare mobile phases for use with your detector and how to passivate the other parts of your chromatographic system.

Chapter 2 "Installing Your Detector" describes how to install your detector and how to verify that it is working correctly.

Chapter 3 "Running An Analysis" provides you with an overview of how to run an analysis. The chapter gives clear, precise instructions without unnecessary details. A one-page checklist is included for day-to-day reference.

Chapter 4 "Keyboard Reference" describes each of the detector's functions in detail. The functions are arranged logically according to the keyboard for easy access.

Chapter 5 "Automating Your Detector" gives you several detailed examples of how to automate operation of your detector.

Chapter 6 "Troubleshooting Your Detector" helps you to investigate problems, find the cause and take appropriate action.

Chapter 7 "Maintaining Your Detector" describes the tasks you can perform to keep your detector in good working order.

Chapter 8 "Installing Accessories" describes how to install accessories that are available for your detector.

Chapter 9 "Using the GPIB Interface" explains the communication protocol of the General Purpose Interface Bus that you can use to control the detector from a compatible computer.

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## Contents

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## **Preparing Your Liquid Chromatograph**

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# Preparing Your Liquid Chromatograph

Read the information in this chapter on mobile phases and solvents. The quality of the solvents you use and the way you prepare them can greatly influence your chromatographic results.

It is important that you prepare your chromatographic system by passivating all parts in contact with the mobile phase. Passivation is necessary because electroactive oxides on internal surfaces gradually dissolve in the mobile phase, resulting in a continuously drifting baseline and even contamination of electrodes.

---

**NOTE**

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To save time we recommend that you passivate your chromatographic system at the same time as you are installing your detector.

## Preparing mobile phases and solvents

### Quality

Always use LC-quality solvents.

### Filtering

Always filter your solvents. When filtering, discard the first 200 ml of solvent that could contain residual particles from the filtering apparatus.

Solvent filtering/degassing apparatus is available from Agilent Technologies. For ordering details see the *Analytical Supplies Catalog* or ask your Agilent Technologies representative.

### Degassing

Always degas your solvents thoroughly. If possible, use a closed system to prevent air from redissolving in the mobile phase.

### Recommended buffers

Always use freshly prepared mobile phases, particularly salts and modifiers.

Use a buffer in the mobile phase, such as phosphate, acetate or ammonium hydroxide, for conductance.

Use quaternary ammonium salts as the positive pairing ion. Use sulfonic acids as the negative pairing ion.

Use EDTA as a complexing agent to prevent free metal-ions from interfering with detection.

Do not use higher concentrations than 1 mM of chloride ions ( $\text{Cl}^-$ ) when using the reference electrode with internal electrolyte.

Do not use tri-ethylamine (TEA) for buffering or ion-pairing.

Do not use potassium permanganate  $\text{KMnO}_4$  to passivate your system and take care when using any substances containing manganese. Manganese and its oxides are strongly adsorbed onto electrode surfaces.

## **Storage**

Do not keep solvents at room temperature for long periods. Store the solvents in a refrigerator.

## Passivating your liquid chromatograph

You must passivate all parts of your chromatographic system that come into contact with the mobile phase, *except* the column and detector cell.

---

**NOTE**

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Do not use potassium permanganate  $\text{KMnO}_4$  to passivate your system—manganese and its oxides are strongly adsorbed onto electrode surfaces.

### Stage 1: Setting-up

Allow *10 min* to complete this stage.

- 1 Connect your solvent pump, injector, solvent thermostat (if installed) and all capillaries in series.
- 2 Lead the capillaries away to a safe waste container.
- 3 *Do not* connect your column.
- 4 *Do not* connect the detector cell.

### Stage 2: Cleaning and degreasing

Allow *60 min* to complete this stage.

- 1 Set your solvent pump to deliver maximum flow rate.
- 2 Flush the chromatographic system with water for 20 min.
- 3 Flush with isopropanol for 15 min.
- 4 Flush with water for 15 min.

---

**NOTE**

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Remember to flush all bypass capillaries, for example, in the injection valve.

### Stage 3: Passivating

Allow *60 min* to complete this stage.

**Passivating your liquid chromatograph**

---

**NOTE**

If you have non-Agilent Technologies instruments or accessories, check with the manufacturer that these will not be damaged by passivating with concentrated nitric acid.

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**WARNING**

**This procedure uses concentrated nitric acid. Wear appropriate safety clothing (gloves and lab coat) and eye protection (goggles or face shield).**

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**WARNUNG**

**In dieser Vorschrift wird Salpetersäure verwendet. Tragen Sie deshalb angemessene Sicherheitskleidung (Handschuhe und Labormantel) und einem Augenschutz (Sicherheitsbrille oder Gesichtsschild).**

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**CUIDADO**

**En este procedimiento se usa ácido nítrico concentrado. Utilice ropa adecuada de laboratorio, guantes y gafas de seguridad.**

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**ATTENTION**

**Dans cette procédure vous utilisez de l'acide nitrique concentré. Portez les vêtements de sécurité appropriés (gants et blouse) et protections oculaires (lunettes de sécurité ou visière).**

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**ATTENZIONE**

**Questa operazione impiega acido nítrico concentrato: indossare gli indumenti adatti (guanti e camice) e proteggere gli occhi (occhiali o schermo protettivo).**

---

- 1 Set your solvent pump to deliver maximum flow rate.
- 2 Flush the chromatographic system with 6M (37.8%) nitric acid for 30 min.
- 3 Flush with water until the pH is about 7 (use pH meter or pH paper).

---

**NOTE**

Replace the water in the solvent reservoir several times to ensure that the nitric acid is flushed out of the inlet filters.

---

### **Stage 4: Equilibration**

Allow *70 min* to complete this stage.

By this stage you should have installed your detector.

### Passivating your liquid chromatograph

- 1 Set your solvent pump to deliver maximum flow rate.
- 2 Flush the chromatographic system with the mobile phase for your application for 30 min.
- 3 Set your solvent pump to deliver about  $1 \text{ mlmin}^{-1}$ .
- 4 Connect your column and detector cell with the passivated capillaries.
- 5 Set your solvent pump to deliver the flow rate for your application.
- 6 Flush with mobile phase for 30 min.

### Summary

If you need to passivate your system frequently, use this summary as a quick reference.

To passivate your chromatographic system:

- 1 Connect all parts of your system, *except* column and detector cell.
- 2 Set pump to deliver maximum flow rate.
- 3 Flush with water for 20 min.
- 4 Flush with isopropanol for 15 min.
- 5 Flush with water for 15 min.
- 6 Flush with 6 M (37.8%) nitric acid for 30 min.
- 7 Flush with water until pH is about 7.
- 8 Flush with mobile phase for 30 min.
- 9 Connect column and detector cell.
- 10 Set pump to deliver flow rate for your application.
- 11 Flush with mobile phase for 30 min.

Preparing Your Liquid Chromatograph  
**Passivating your liquid chromatograph**

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## **Installing Your Detector**

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# Installing Your Detector

This chapter describes how to install your detector and verify that it is functioning correctly.

---

## NOTE

To save time, we recommend that you passivate your chromatographic system at the same time as you are installing your detector; see Chapter 1 “Preparing Your Liquid Chromatograph”.

Check carefully before you begin that you have all the necessary cables and connection points for the Agilent 1049A: read “Choosing a Suitable Place” on page 20 and “Unpacking” on page 21. If you wish to install or interface the Agilent 1049A to instruments or accessories not specified here, you should first contact your local Agilent Technologies office to check that the safety of the Agilent 1049A will not be affected by this intended connection.

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## WARNING

**The Agilent 1049A panels and cabinet must be grounded to protect yourself during operation. The Agilent 1049A, and any peripheral instruments connected to it, must be operated from a three-contact power source, with the ground contacts securely connected to ground. Get an electrician to check and verify ground at the source.**

**High voltages: Disconnect the line-power cord before removing any of the Agilent 1049A panels.**

---

## WARNUNG

**Um den Benutzer zu schützen, muß das Gerät geerdet sein. Der Agilent 1049A und alle Peripherie müssen mit einem 3-adrigen Netzkabel ausgestattet sein, das den Agilent 1049A mit der Schutz Erde verbindet, sobald der Stecker in eine passende Steckdose gesteckt wird. Überprüfen Sie die Schutz Erde, bevor der Agilent 1049A installiert wird.**

**Hochspannung: Bevor ein Gehäuseteil entfernt werden soll, muß das Netzkabel abgezogen werden.**

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**CUIDADO**

Para proteger al operador, todos los paneles del instrumento deben estar conectados a tierra. Tanto el Agilent 1049A como los periféricos deben trabajar con todas las tomas de tierra conectadas correctamente. Verifique su toma de tierra antes de instalar el Agilent 1049A.

**Alta tensión:** Desconecte el instrumento de la línea antes de sacar el panel posterior del instrumento.

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**ATTENTION**

Pour protéger l'opérateur, les panneaux de l'instrument et le four doivent être raccordés à la terre; le Agilent 1049A ainsi que tout périphérique associé doivent être branchés à une prise électrique à 3 contacts dont celui de terre est relié correctement à la terre du bâtiment. Vérifiez la prise de terre avant d'installer le Agilent 1049A.

**Tensions élevées:** Débranchez le cordon secteur avant d'ôter le panneau arrière.

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**ATTENZIONE**

Per proteggere l'utilizzatore, i pannelli dello strumento e la struttura esterna devono essere collegati a terra. Sia il vostro Agilent 1049A che le varie periferiche devono lavorare con un cavo di alimentazione a 3 conduttori con il filo di terra adeguatamente collegato a terra. Verificate la vostra linea di terra prima di installare lo strumento.

**Alte tensioni:** Scollegare il cavo di alimentazione prima di rimuovere il pannello posteriore.

---

## **Choosing a Suitable Place**

Check carefully before you begin that the place you have chosen to install your detector meets requirements below. Check each item as you read through the list.

### **Dimensions**

430 mm × 370 mm × 150 mm

(17 in × 14.5 in × 6 in)

(width × depth × height)

Leave adequate space to the right (or left, as you prefer) of the Agilent 1049A for capillary connections, and at least 8 cm (3 in) at the rear for drainage of any leaks to waste, and access to the ~LINE switch.

### **Weight**

8.2 Kg (18 lb)

### **Environment**

Temperature 0 to 55°C (50 to 131°F)

Humidity 5 to 95% (40°C)

### **Line Voltage**

100 to 120 V AC, ±10% or 220 to 240 V AC, ±10%

### **Line Frequency**

47 to 63 Hz

### **Power Consumption**

100 VA maximum

## Unpacking

Inspect the carton. If you find signs of external damage, contact your local Agilent Technologies office. After you have removed the plastic straps and staples, open the top of the carton. Lift out the accessory box. Check the accessories against the packing list below.

**Table 1**

### Checklist of Accessories

Description	Part Number	Check
Power Cord	as ordered	...
Signal cable(s)	as ordered	...
Remote cable(s)	as ordered	...
Instruction kit	01049-90000	...
Electrochemical cell, including:	01049-68700	...
5 Electrode spacers	01049-24705	...
Cell holder	-	...
Platinum working electrode (if ordered as option #031)	01049-28801	...
Gold working electrode (if ordered as option #030)	01049-28802	...
Reference electrode with internal electrolyte (if ordered as option #035)	01049-62902	...
Solvent thermostat (if ordered as option #025)	79845A	...
Waste tubing	0890-1711	...
Accessory kit, including:	01049-68701	...
Polishing kit	01049-67001	...

**Table 1**

<b>Checklist of Accessories, continued</b>		
<b>Description</b>	<b>Part Number</b>	<b>Check</b>
3 Ferrules	5041-2121	...
3 Grippers	5041-2122	...
3 Male fittings	5041-2123	...
3 Buffer discs	5041-2124	...
Inlet capillary	01048-87302	...
Outlet tubing	01040-67602	...
2 fuses, 1 Amp	2110-0007	...
2 fuses, 0.5 Amp	2110-0202	...
2 Pins	1251-3911	...
1 Plug	1251-4782	...
Wrench, 5/16in and 1/4in	8710-0510	...
Pozidriv screwdriver #1	8710-0899	...
Slot screwdriver	8730-0019	...

Each Agilent 1049A is identified by a 10-digit serial number on a label attached to the rear panel.

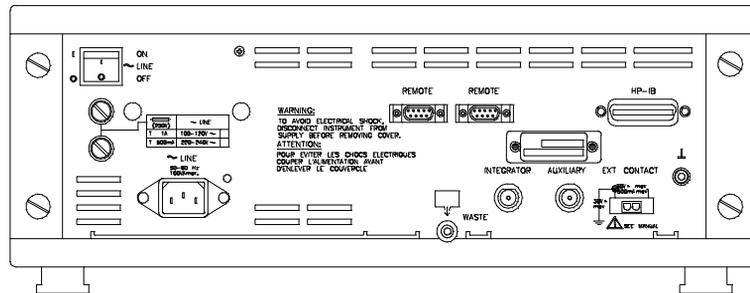
The serial number of my Agilent 1049A is:

.....

## Cables

Figure 1

Rear Panel of Detector



### INTEGRATOR and AUXILIARY Connectors

The INTEGRATOR and AUXILIARY connectors provide signal output. You can vary the scaling range of these outputs according to your application, see “Changing the Full-Scale Range” on page 24 and see “SCALING PARAMETERS” on page 83.

If you want to use the detector's **SWEEP** mode, see “The SWEEP Mode” on page 70, connect the INTEGRATOR output to the X-axis (for potential) and connect the AUXILIARY output to the Y-axis (for current) on your plotter. To control pen-up and pen-down movement on your plotter, use the EXT CONTACT function of your detector, see “EXT CONTACT Connector” on page 27.

Check that you have the correct cable from the detector to your integrator or plotter, see Table 2. The part number is in a sheath around the cable.

**Cables**

Screw the metal cap of the signal cable onto the connector. Push the other end of the signal cable into the connector for analog input on your integrator or plotter.

**Table 2****Signal cables**

<b>From Agilent 1049A to:</b>	<b>Requires signal cable:</b>
HP 3390A Integrator	01040-60101
HP 3392A Integrator	01040-60101
HP 3393A Integrator	01040-60101
HP 3394A Integrator	35900-60750
Agilent 3396A Integrator	35900-60750
HP 18652A Interface	01046-60103
Agilent 35900A Dual Channel Interface	35900-60750
HP 1082B Liquid Chromatograph	8120-1840
HP 1084B Liquid Chromatograph	8120-1840
General purpose	01046-60105

If you are using a non-Agilent device, connect the spade lugs on the end of the general purpose signal cable 01046-60105 to the contacts of your instrument.

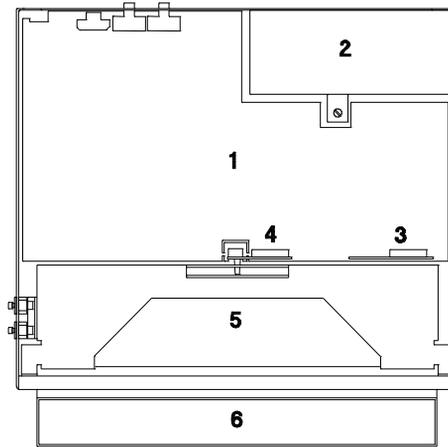
**Changing the Full-Scale Range**

The full-scale range of the INTEGRATOR and AUXILIARY outputs is set at 0 to 1 V. You can change this setting to 0 to 100 mV by moving a slide-switch on the detector's ECC board.

- 1** Remove the 2 screws at each side of the rear panel.
- 2** Remove the 2 screws at both the left and right side of the detector.
- 3** Lift off the top cover.
- 4** Find the full-scale definition switch—it is located on the ECC board (item 7 in Figure 2) near to the INTEGRATOR and AUXILIARY connectors.

Figure 2

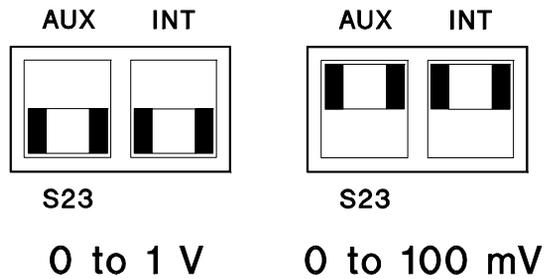
Location of Full-scale Range Switch



- 5 Insert tip of small screwdriver into slot of switch and slide to correct position, see Figure 3.

Figure 3

Setting the Full-scale Range Switch



- 6 Put the top cover back into place.
- 7 Secure the side panels and top cover with the 2 screws on each side and the 2 screws on the rear panel.

### REMOTE Connectors

These connectors communicate start, stop, prepare, error and not-ready signal inputs and outputs, in a chain arrangement between your liquid

**Cables**

chromatograph and integrator. The connectors are identical: you can connect your liquid chromatograph or integrator to either one. The Agilent 1049A simply passes on the signals it receives, in either direction. If the flow cell leaks, a shut-down signal is generated, which you can use to stop your solvent pump.

Check that you have the correct cable from the Agilent 1049A to your instrument, and from your instruments to the Agilent 1049A if necessary, see Table 3 and Table 4. The part number is in a sheath around the cable.

**Table 3****Remote Control Cables From Agilent 1049A**

<b>From Agilent 1049A to:</b>	<b>Requires remote control cable:</b>
HP 3390A Integrator	01046-60203
HP 3392A Integrator	01046-60206
HP 3393A Integrator	01046-60206
HP 3394A Integrator	01046-60210
Agilent 3396A Integrator	03394-60600
HP 18652A Interface	01046-60204
Agilent 35900A Dual Channel Interface	5061-3378
General Purpose	01046-60201

**Table 4****Remote Control Cables To Agilent 1049A**

<b>To Agilent 1049A from:</b>	<b>Requires remote control cable:</b>
HP 1050 Series of LC modules	5061-3378
HP 1081B Liquid Chromatograph	01046-60200
HP 1082B Liquid Chromatograph	01046-60201
HP 1084B Liquid Chromatograph	01046-60201
HP 1090 Liquid Chromatograph	01046-60202

**Cables**

Push the metal plug of the remote control cable onto one of the REMOTE connectors. There is only one way to position the plug. Tighten the two screws to secure the connector. Connect the other end of the cable to the contacts for remote start, stop or prepare on your instrument.

If you are using a non-Agilent device, connect the crimp contacts on the end of the general purpose remote cable 01046-60201 to the contacts of your instrument. To help you make the correct connections, the signals carried on each pin are listed below, the colors refer to the wires of remote cable part number 01046-60201.

**Table 5****Signals Carried by Remote Cable**

Pin	Signal	Wire color	Activity
1	Digital ground	white	
2	Prepare run	brown	low
3	Start	gray	low
4	Shut down	blue	low
5	Not used	pink	
6	Not used	yellow	
7	Ready	red	high
8	Stop	green	low
9	Start request*	black	low

\* The Agilent 1049A does not use the start request signal.

**EXT CONTACT Connector**

The EXT CONTACT provides a fused 500 mA 30 V AC/DC rated contact closure.

You can use the EXT CONTACT:

- To control the pen of an X-Y plotter. In **SWEEP** mode the detector closes the contact when you press   and opens the contact when the sweep has finished or when you press  . Use this to control the pen-down and pen-up functions of your X-Y plotter.

## Cables

- To start another device. In all modes, *except* **SWEEP** mode, the detector closes the contact for 1 s when you press  .
- To start your system. Connect the cable from the EXT CONTACT connector to pin 3 (start) or to pin 9 (start request, if you are using an HP 1050 Series module) of one of the REMOTE connectors (connect the other REMOTE connector to the other devices in your system). In all modes, *except* **SWEEP** mode, the detector closes the contact for 1 s when you press  . This will start the detector and pass on the start signal through the remote cables to other devices in your system. Use this method of operation when you want the detector to be the "controller" of your system.

The accessory kit contains a brown plastic plug (part number 1251-4782) and two metal pins (part number 1251-3911) to adapt your cables for connection to the EXT CONTACT socket.

To attach a plug to your cable:

- 1 Remove plug or connector on free end of your cable.
- 2 Remove 2 cm (1 in) of insulation material from end of cable.
- 3 Remove 0.5 cm (0.25 in) of insulation material from input wire of cable. Twist exposed wire(s) tightly to give a good contact surface.
- 4 Break off a metal pin from metal strip.
- 5 Place exposed end of wire inside open end of pin.
- 6 Using pliers, pinch open end of pin over wire.
- 7 Repeat steps 3 through 6 for ground wire of cable.
- 8 Push pins through holes in plug.
- 9 Push plug into EXT CONTACT connector.

## Chassis Terminal

If you have a measuring instrument that has a safety ground connector, connect grounding cable from your instrument to chassis terminal on rear panel of your Agilent 1049A.

## **Installing the Electrochemical Cell**

Before you install the electrochemical cell you must:

- Remove the front panel of your detector.
- Fit the working and reference electrodes of your choice.
- Fit the cell clamp to the mounting panel.

### **Removing the Front Panel**

- 1** Loosen the 2 screws in bottom-left and bottom-right corners of front panel.
- 2** Pull bottom edge of front panel towards you and then pull it down away from detector.
- 3** Using the 2 clips on the inside of the front panel, hang front panel on front edge of detector's bottom panel, or place front panel on top or at the side of the detector.

### **Fitting Electrodes of Your Choice**

The electrochemical cell is shipped with a glassy carbon working electrode and a solid state reference electrode installed.

If you want to use the electrochemical cell as supplied, go to “Fitting the Cell Clamp” on page 31.

If you want to use the optional gold or platinum working electrode, or the optional reference electrode with internal electrolyte, Figure 4 will help you identify the parts of the electrochemical cell.

Figure 4

Exploded Diagram of Electrochemical Cell

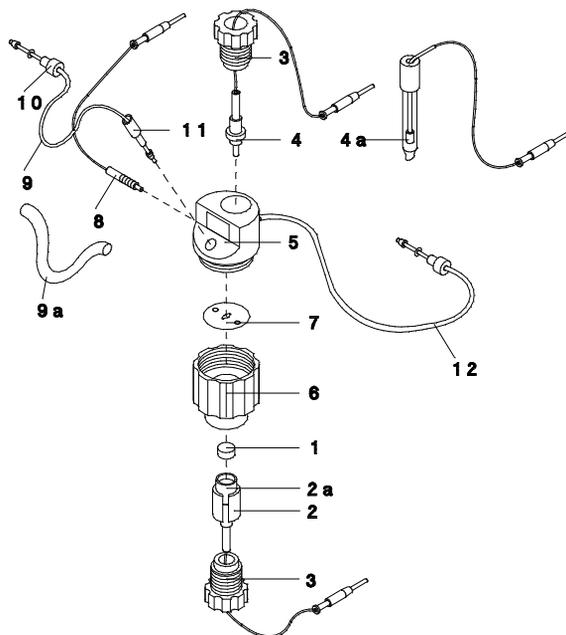


Figure legend:

1. Working electrode
2. Working electrode retaining ring
- 2a. Working electrode holder
3. Electrode assembly retaining nut
- 4a. Solid state reference electrode
- 4b. Reference electrode with internal electrolyte
5. Cell body (upper part)
6. Cell body (lower part)
7. Electrode spacer
8. Auxiliary electrode connector
9. Inlet capillary
10. Inlet capillary fitting (from column)

## Installing the Electrochemical Cell

11. Inlet capillary fitting (to cell)

12. Outlet capillary

If you want to use the optional gold or platinum working electrode, go to “Fitting Gold or Platinum Working Electrodes” on page 31.

If you want to use the optional reference electrode with internal electrolyte, go to “Fitting the Reference Electrode” on page 31.

### Fitting Gold or Platinum Working Electrodes

- 1 Unscrew nut (item 3 at bottom of Figure 4) and remove working electrode assembly (items 1, 2 and 2a in Figure 4).
- 2 Hold shaft of electrode assembly and pull down retaining ring (item 2 in Figure 4).
- 3 Using tip of flat-bladed screwdriver, prise off glassy carbon working electrode (item 1 in Figure 4).
- 4 Insert gold or platinum working electrode and push up retaining ring.
- 5 Clean surface of working electrode with acetone or methanol.
- 6 Insert working electrode assembly in cell body and tighten nut.

### Fitting the Reference Electrode

- 1 Fill reference electrode with potassium chloride (KCl) solution supplied with the electrode.
- 2 Unscrew nut (item 3 at top of Figure 4 on page 30) and remove solid state reference electrode.
- 3 Insert reference electrode with internal electrolyte in cell body and tighten nut.

### Fitting the Cell Clamp

You must now decide where on the mounting panel you want to fit the cell. Your decision will depend on:

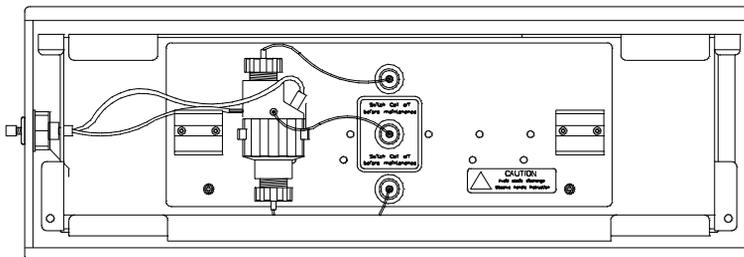
- whether you want the inlet on the left or right side of the detector, and;
- whether you have a solvent thermostat.

We will describe how to change the position of the inlet and outlet connections (on the side panel) later in this chapter.

## Installing the Electrochemical Cell

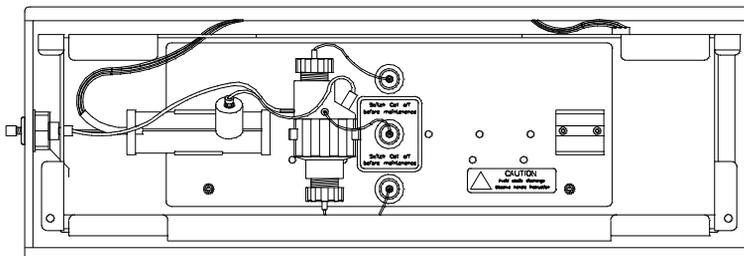
If you do not have a solvent thermostat, you must fit the cell clamp near the left or right side of the mounting panel, see Figure 5.

**Figure 5** **Fitting of Cell (Solid State Reference Electrode)**



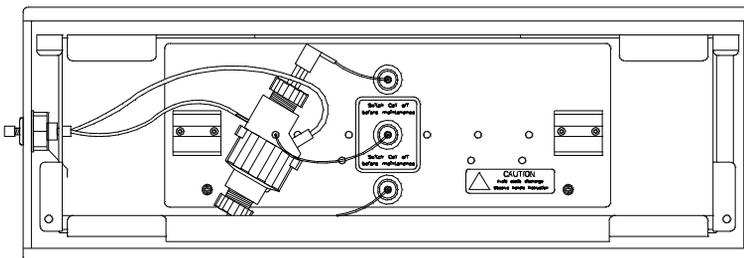
If you have a solvent thermostat, you must fit the cell clamp near the center of the mounting panel, see Figure 6.

**Figure 6** **Fitting of Cell (Solid State Reference Electrode) and Thermostat**



There is one further consideration—if you are using the reference electrode with internal electrolyte, you must fit the cell clamp at an angle (the reference electrode with internal electrolyte is longer than the solid state reference electrode and prevents you from fitting the cell vertically), see Figure 7 and Figure 8.

**Figure 7 Fitting of Cell (Reference Electrode with Internal Electrolyte)**



**Figure 8 Fitting of Cell (Reference Electrode with Internal Electrolyte) and Thermostat**

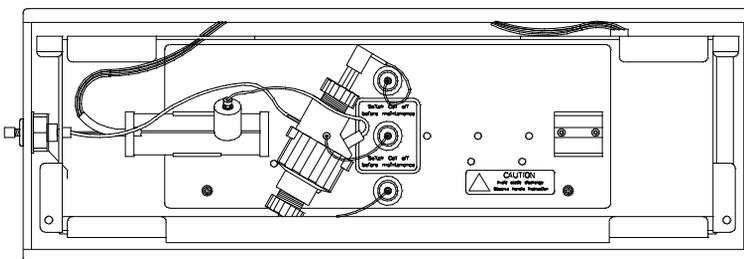
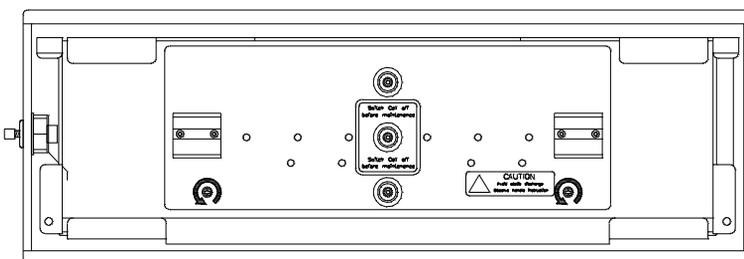


Figure 9 will help you identify the parts inside the cell and thermostat compartment.

**Figure 9 Cell Compartment**



To fit the cell clamp:

- 1 If a solvent thermostat is installed, pull the thermostat out of its clamp.
- 2 Loosen the 2 thumb screws in the bottom-left and bottom-right corners of the mounting panel.

### Installing the Electrochemical Cell

- 3** Pull the bottom edge of the mounting panel towards you and then pull it down and away from the detector.
- 4** From the rear of the mounting panel, insert the 2 screws through the appropriate holes in the panel and secure the cell clamp.
- 5** Replace the mounting panel in the detector and tighten the 2 thumb screws.
- 6** If a solvent thermostat is installed, insert it into its clamp.

### Fitting the Cell

- 1** With the reference electrode at the top (blue wire), insert the cell into its clamp.
- 2** *Do not* connect the inlet and outlet capillaries.
- 3** *Do not* connect the electrodes.

## Connecting the Hydraulic Lines

The inlet and outlet connections are factory installed in the side panel at the left of the Agilent 1049A. If you want to keep the length of the capillary connections from your liquid chromatograph to a minimum, you can exchange the left side with the right, as follows.

### Changing Position of Inlet and Outlet Connections

- 1 Remove the 4 screws at each side of the rear panel.
- 2 Remove the 2 screws at both the left and right side of the detector.
- 3 Lift off the top cover.
- 4 Lift out the right side panel and put to one side.
- 5 Lift out the left side panel and turn it over to the right side of the detector.
- 6 Turn over the other panel and place it on the left side of the detector.
- 7 Pull off leak drain from inlet and outlet connections, turn leak drain through 180°; and replace it beneath the connections.
- 8 Put the top cover back into place.
- 9 Secure the side panels and top cover with the 2 screws on each side and the 4 screws on the rear panel.

### Connecting the Detector Inlet and Outlet

The detector inlet and outlet are the 2 fittings labeled IN (in *red*) and OUT (in *blue*) on the side panel of your detector.

---

**NOTE**

By this stage you *must* have passivated your chromatographic system, *including* the detector's IN fitting, any capillaries on the inlet side *and* the solvent thermostat (if installed), see Chapter 1 “Preparing Your Liquid Chromatograph”.

---

## Connecting the Hydraulic Lines

- 1 Connect the inlet capillary (supplied in the accessory kit) from your column to the IN fitting on the side of the detector.
- 2 Connect the outlet tubing (supplied in the accessory kit) from the OUT fitting on the side of the detector to a safe waste container.

Keep the height difference between the OUT fitting and the waste container to a minimum, to prevent underpressure or overpressure in the cell.

---

**NOTE**

---

Make sure the cell outlet points upwards to prevent air bubbles from being trapped in the cell.

- If you are using the reference electrode with internal electrolyte:  
Underpressure draws electrolyte out of the reference electrode. In the worst case the Ag/AgCl wire will no longer be in contact with the KCl solution, resulting in large potential jumps of the potentiostat, because no reference potential is defined. This could damage the working electrode.  
Overpressure can push eluent into the reference electrode. This will give increased noise and/or a shifted reference electrode potential.

---

**NOTE**

---

If you are unsure about the actual potential of the reference electrode, use the REFERENCETEST under flow conditions, see “Reference Electrode Test” on page 49.

- In general, underpressure can cause air to be trapped in the reference electrode compartment, which is difficult to remove. This will increase the noise (usually as spikes).

## Connecting the Cell and Solvent Thermostat

If you have a solvent thermostat installed:

- 1 Attach the yellow label to the thermostat. The label reads:

BEFORE FIRST USE  
PURGE WITH HNO<sub>3</sub>  
(SEE INSTALLATION  
PROCEDURE IN  
MANUAL)

## Connecting the Hydraulic Lines

The passivation procedure is described in Chapter 1 “Preparing Your Liquid Chromatograph”.

- 2 Connect the male fitting on the thermostat to the IN fitting on the inside of the side panel.
- 3 Connect the inlet tube (colored *red*) between the cell and the fitting on the thermostat.
- 4 Connect the cell outlet tube (colored *blue*) between the cell and the OUT fitting on the inside of the side panel.
- 5 *Do not* connect the electrodes.

If you do not have a solvent thermostat installed:

- 1 Connect the inlet tube (colored *red*) between the cell and the IN fitting on the inside of the side panel.
- 2 Connect the cell outlet tube (colored *blue*) between the cell and the OUT fitting on the inside of the side panel.
- 3 *Do not* connect the electrodes.

## Connecting the WASTE Outlet

Push a length of corrugated tubing (supplied with your detector) onto fitting at lower center of rear panel, see Figure 1. Lead tubing to a safe waste disposal container.

## Connecting Line Power

Observe the following safety information.

---

**WARNING**

---

**Ensure that line-power cord is disconnected before changing line-voltage setting. Ensure that the line-power cord is disconnected before installing or replacing a fuse.**

---

**WARNUNG**

---

**Wenn der Netzspannungswahlschalter betätigt wird, darf das Netzkabel nicht angeschlossen sein. Während des Auswechslens einer Sicherung darf das Netzkabel nicht angeschlossen sein.**

---

**CUIDADO**

---

**Asegúrese que el cable de red está desconectado antes de cambiar el selector de voltaje. Asegúrese de tener desconectado el instrumento cuando necesite cambiar un fusible.**

---

**ATTENTION**

---

**Assurez vous que le cable secteur n'est pas connté avant de changer la tension d'alimentation. Vérifiez que le cordon secteur est déconnecté avant de mettre en place ou de remplacer le fusible.**

---

**ATTENZIONE**

---

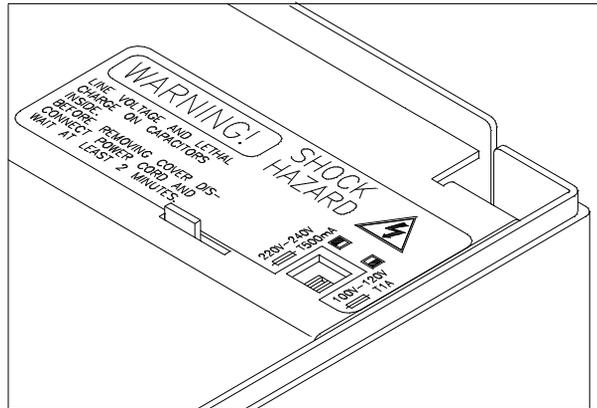
**Assicuratevi che il cavo do alimentazione sia scollegato prima di spostare li elettore di voltaggio. Assicuratevi che il cavo di alimentazione sia staccato prima di installare o sostituire il fusibile.**

## Setting the Line Voltage

Check that the line-voltage selector is set for the correct line voltage for operation in your area. The switch is set at the factory, you should not need to change it. The position of the voltage selector switch is marked on the label on top of the main power supply cover, see Figure 10.

**Figure 10**

**Setting the Line Voltage**



To change the setting: Turn-OFF the detector at the ~LINE power switch at the rear. Insert tip of small screwdriver into slot of switch and slide switch to correct position.

**Fuses**

Check that the correct fuses for the voltage operation you have selected are installed.

**Table 6**

**Fuses**

<b>Voltage</b>	<b>Fuse</b>	<b>Part number</b>
100 to 120 V	1 Amp	2110-0007
220 to 240 V	0.5 Amp	2110-0202

To install a fuse:

- 1** Use a screwdriver to push the fuse cap inwards and then turn counterclockwise.
- 2** Place new fuse in fuse cap.
- 3** Insert fuse and cap into fuse receptacle, pressing and turning clockwise until cap locks in place.

### **Connecting the Power Cord**

Connect the line power cord to the ~LINE socket at the rear of the detector.

Connect the other end of the line power cord to a suitable power source, as indicated on the rear panel.

---

**NOTE**

---

Connect the power cords of *all* units of your system to the *same* power source. This prevents current loops that can cause systematic baseline interference.

## Connecting the Electrodes

Before connecting the electrodes you must ensure that the cell is turned-OFF.

- 1 Turn-ON line power to the detector at the ~LINE switch on the rear panel.
- 2 Turn-OFF the cell:

On the keyboard, press **Cell on/off** to display:

```
■CELL      = ON
```

Press **▶** to move the cursor to ■ON.

Press **Next ▼** to change the display to:

```
CELL      = !OFF
```

Press **Enter**. The cell is now OFF and you can safely connect the electrodes.

- 3 Insert the *black* pin of the working electrode in the *black* socket on the mounting panel.
- 4 Insert the *red* pin of the auxiliary electrode in the *red* socket on the mounting panel.
- 5 Insert the *blue* pin of the reference electrode in the *blue* socket of the mounting panel.
- 6 Now replace the front panel, securing it with the 2 screws.

## Verifying the Performance of Your Detector

The performance tests described here assume that you have prepared your liquid chromatograph as described in Chapter 1 “Preparing Your Liquid Chromatograph” and that you have installed your detector correctly, according to the instructions at the beginning of this chapter.

You will perform 6 test procedures. The first 4 tests (analog output test, amperometer test, amperometer/zero test and dummy cell test) are done under static conditions with no eluent flow to check that all the electronics are functioning correctly.

The final 2 tests (baseline level and noise test and sensitivity test) are done under dynamic conditions with eluent flow.

### Static Performance Tests

During the performance tests the detector sets the correct test parameters automatically.

#### Analog Output Test

- 1 Connect the INTEGRATOR or AUXILIARY output of the detector to your integrator or recording device.
- 2 Set the full scale range of the integrator to 50  $\mu$ V.
- 3 Set the detector mode to:

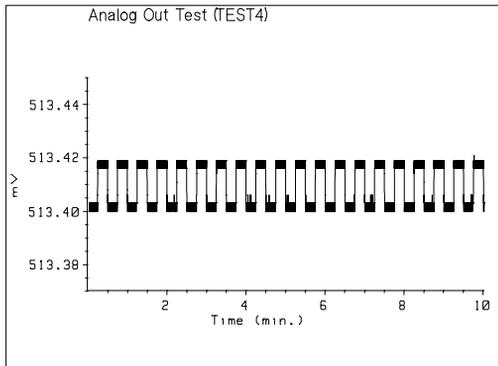
```
MODE = ■TEST4 ; analog out test
```

(several times)

- 4 Start your integrator or recording device.

You should get a plot similar to Figure 11.

Figure 11

**Analog Output Test**

When you start the analog output test the detector sets the analog output to 50% of the full scale range. At the default setting of 1 V, the output should be about 500 mV. On top of this output the detector superimposes an output level change every 15 s. The plot looks like a square wave.

The steps of the square wave should be about  $15 \mu\text{V}$  ( $\pm 2 \mu\text{V}$ ). The noise level within a step should not exceed  $4 \mu\text{V}$ . The drift should be negligible.

**Amperometer Test**

- 1 Connect the INTEGRATOR or AUXILIARY output of the detector to your integrator or recording device.
- 2 Set the full scale range on the integrator to  $50 \mu\text{V}$ .
- 3 Set the detector mode to:

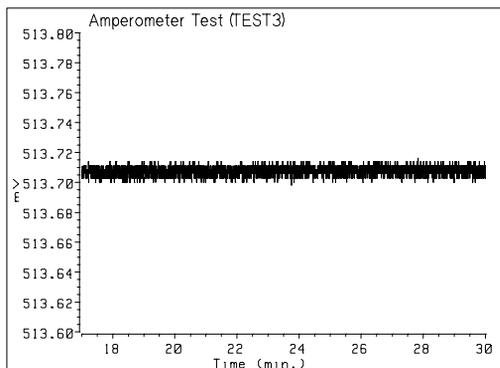
MODE = ■TEST3 ;                      amp test

Mode    Next ▼ (several times)    Enter

- 4 Start your integrator or recording device.  
You should get a plot similar to Figure 12.

Figure 12

### Amperometer Test



In this experiment the detector measures the amperometer noise. The cell is not connected electrically, and the electronic parts that allow an offset of the cell current (zero level and background current) are disabled. The full scale range is changed to 50 nA automatically. The noise should be below 20  $\mu\text{V}$  (750 fA).

### Amperometer/Zero Test

Before you do this test, the cell should have been turned-ON for at least 30 minutes.

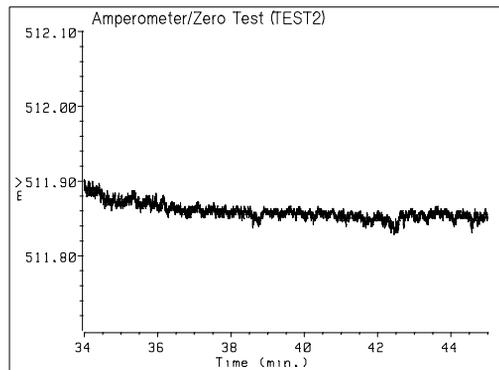
- 1 Connect the INTEGRATOR or AUXILIARY output of the detector to your integrator or recording device.
- 2 Set the full scale range on the integrator to 50  $\mu\text{V}$ .
- 3 Set the detector mode to:

```
MODE = ■TEST2 ; amp / zero test
```

(several times)

- 4 Wait about 5 minutes, then start your integrator or recording device.  
You should get a plot similar to Figure 13.

Figure 13

**Amperometer Zero Test**

The full scale range in this test is changed to 50 nA automatically. In this test the complete amperometer is tested, including the electronic parts that allow an offset of the cell current (zero level and background current). The noise should be less than 40  $\mu\text{V}$  (2 pA).

**Dummy Cell Test**

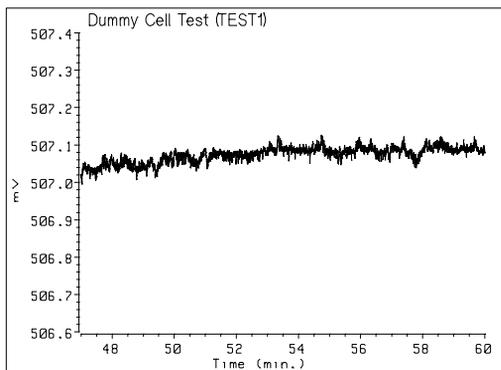
- 1 Connect the INTEGRATOR or AUXILIARY output of the detector to your integrator or recording device.
- 2 Set the full scale range on the integrator to 500  $\mu\text{V}$ .
- 3 Set the detector mode to:

```
MODE = ■TEST1 ; dummy cell test
```

Mode    Next ▼    (several times)    Enter

- 4 Start your integrator or recording device.  
You should get a plot similar to Figure 14.

Figure 14

**Dummy Cell Test**

In this test the complete electrochemical detector is checked, with a dummy resistance of 1 MOhm. If the noise values you obtained in the previous tests were correct, the noise in this test should be less than 60  $\mu\text{V}$ . The noise is mainly caused by an amplification of the DC noise of the potentiostat and of the amperometer. As the full scale range is 500 nA in this test, a noise level of 60  $\mu\text{V}$  is equal to 30 pA through the dummy cell.

**Dynamic Performance Tests**

For the dynamic performance tests you need to prepare a test mobile phase and test sample.

To prepare the test mobile phase:

- 1 Put 1000 ml pure water in a beaker.
- 2 Add 5.73 ml acetic acid (gives 0.1 M)
- 3 Add 746 mg potassium chloride (gives 0.01 M)
- 4 Add 372 mg sodium EDTA salt (gives 1 mM)
- 5 Mix solution thoroughly.

To prepare the test sample:

- 1 Put 100 ml of the mobile phase in a beaker.
- 2 Add 422 mg potassium ferrocyanate (gives 10 mM).
- 3 Mix solution thoroughly—this is the stock solution.
- 4 Take a small volume of stock solution and dilute 1000 times—this is the test sample.

## Verifying the Performance of Your Detector

---

### NOTE

---

Always prepare the test sample immediately before use.

To prepare your chromatographic system:

- 1 Connect your solvent pump and injector *directly* to the detector. *Do not* connect your column.
- 2 Pump the test mobile phase for about 30 min at a flow rate of 1.5 ml/min. This ensures that the working electrode is not contaminated by any materials still present in the system.

Set the detection parameters to the following values (the keystrokes are shown under each display):

INSTRUMENT FULLSCALE = ■ 0.5 uA

Ctrl Enter Next ▼ ▶ Next ▼ Enter

MODE = ■ AMPEROMETRY

Mode Next ▼ ... Enter

RESPONSETIME = ■ 1.00 ; sec

Resptime ▶ Next ▼ ... Enter

For solid state reference electrode:

POTENTIAL = ■ 0.300 ; Volt

Pot ▶ 0 ● 3 Enter

For reference electrode with internal electrolyte:

POTENTIAL = ■ 0.600 ; Volt

Pot ▶ 0 ● 6 Enter

### Baseline Level and Noise

Turn-ON the detector cell:

CELL = ■ ON

Cell on/off ▶ Enter

## Verifying the Performance of Your Detector

Press **Monitor**. The cell current will be very high and unstable. Eventually you might see an **over** message that stands for amperometer overflow or analog output overflow. This is normal when you turn-ON the detector cell.

Press **Next ▼** to display the drift monitor. When all conditions are stable the drift will reach a value of about zero. The absolute cell current (in the monitor display) will be less than 10 nA under these conditions.

Set a ZEROLEVEL that is appropriate for your integrator, for example:

```
ZEROLEVEL = 10 ; % of output
```

**Zero** **Next ▼** **▶** **1** **0** **Enter**

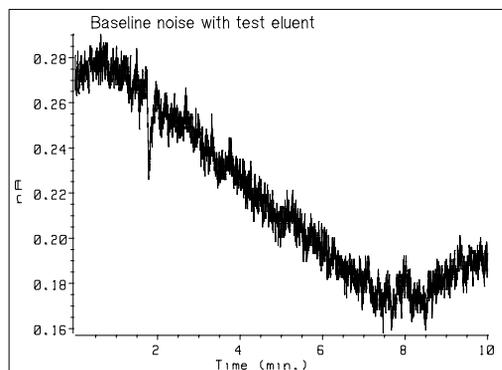
When the baseline is stable, press **Zero** and then **Enter**.

Start your integrator.

Depending on your solvent pump and solvents, you could get baseline noise as low as 15 pA as shown in Figure 15.

**Figure 15**

### Baseline Noise Test



If the signal wanders periodically with the same rhythm as the pulsations of your pump, you might have air in the pump. This might be caused by improper degassing or a blocked solvent inlet filter. It might also be caused by improper pulse dampening. You can remove trapped air from the system by flushing with isopropanol.

If you are unsure if the pump is influencing the noise, change the flow rate to 0.01 ml/min. If the noise decreases dramatically, your pump is causing the problems.

## Verifying the Performance of Your Detector

Temperature fluctuations might also increase the noise level. You might need to thermally insulate the capillary between your injector and the detector.

If the noise level remains high (greater than 100 pA) you have a chemical problem:

- The eluent is not sufficiently clean, or;
- Either the working electrode or reference electrode are deactivated or contaminated.

### Sensitivity

Set a full scale range of 1 V on your integrator.

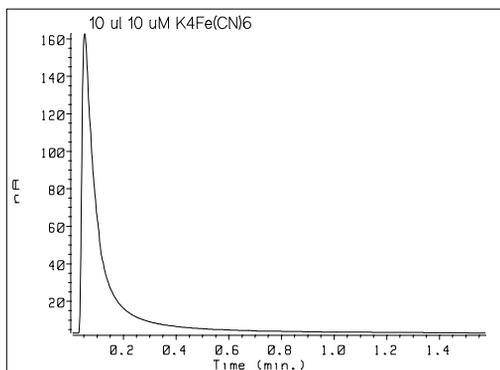
Start your integrator.

Inject 10  $\mu$ l of the 10  $\mu$ M test sample.

You should get a plot similar to Figure 16.

Figure 16

### Sensitivity Test



---

### NOTE

When you have finished the tests, reset the response time parameter to a value suitable for your application. An unsuitable response time setting can result in a large drop in sensitivity.

---

### Reference Electrode Test

The reference electrode test is a diagnostic tool to determine the real potential of the reference electrode and the stability of the potential under flow conditions.

**Verifying the Performance of Your Detector**

In general it is useful to characterize the true redox behaviour of the reference electrode with a known chemical environment. You should bear in mind that the eluent composition and especially the ion concentrations have a dominant impact on the true reference potential.

If you use the reference electrode test to determine absolute potential and drift, wait about 1 hour before starting the test—the large surface areas of the electrodes mean inherently long equilibration times. During the reference electrode test the detector uses the auxiliary electrode as the true measuring electrode and turns off the working electrode. In this way you are able to make a comparison with a working system independent of working electrode influences.

In **REFERENCETEST** mode you can calibrate the measurement by pressing  . When you return to normal (**AMPEROMETRY**) mode, recalibrate the detector with a zero balance by pressing  .

During the test press Monitor to display the absolute potential, then   to display potential drift. In this mode the INTEGRATOR output will be as follows:

**Table 7****Output at the INTEGRATOR Connector**

Reference Potential	INTEGRATOR Output
+0.5 V	1.0 V
0 V	0.5 V
-0.5 V	0 V

---

## **Running An Analysis**

---

# Running An Analysis

This chapter is intended to be a checklist to remind you of the steps you must do when running an analysis. The explanations are deliberately short—cross references to other sections are given throughout, where you will find full details.

---

## **Is Substance Detectable?**

Also supplied with your detector is the handbook *Electrochemical Detection in HPLC--A Practical Primer*. Refer to this handbook to find out *if* your substance is detectable.

This handbook lists over 500 different substances, giving the detection potential, the working electrode material and references to the chromatographic conditions.

## Preparing the System

Before you run an analysis, you *must* ensure that:

- You are using properly prepared mobile phases and solvents of adequate quality, see “Preparing mobile phases and solvents” on page 11.
- You have properly passivated *all* parts of your chromatographic system that come into contact with the mobile phase, see “Passivating your liquid chromatograph” on page 13.
- You have installed your detector and verified that it is functioning correctly, according to the instructions given in Chapter 2 “Installing Your Detector”.

## Setting Detection Parameters

When you turn-ON line power to your detector, the detection parameters are preset to values that are usable immediately—the detection mode is preset to amperometry. The only parameter you must set is the potential.

Set the potential to the required value for your application, for example, +0.6 V for ascorbic acid.

```
POTENTIAL = ■ 0.600 ; Volt
```

Pot ▶ 0 ● 6 Enter

---

## Turning On the Cell

Turn-ON the detector cell:

CELL            = ■ ON

Cell on/off   Enter   ►   Next ▼   Enter

Press Monitor. The cell current will be very high and unstable. Eventually you might see an **over** message that stands for amperometer overflow or analog output overflow. This is normal when you turn-ON the detector cell.

Press Next&down;]] to display the drift monitor. When all conditions are stable the drift will reach a value of about zero. The absolute cell current (in the monitor display) will be less than 10 nA under these conditions.

When the baseline is stable, press Zero and then Enter.

*You are now ready to start.*

## **Checklist**

- Is your substance detectable?
- Prepare mobile phases and solvents.
- Passivate your chromatographic system.
- Set the potential.
- Turn-ON the detector cell.
- Check baseline stability and drift.
- Zero the detector.
- *Start.*

Running An Analysis

**Checklist**

---

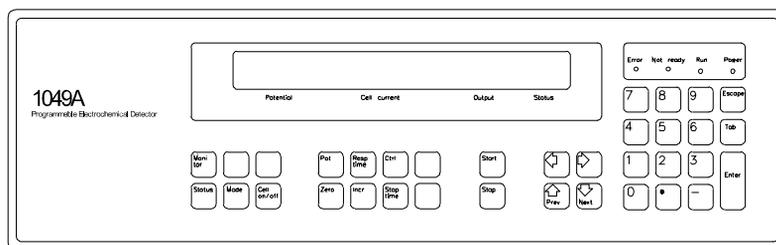
## **Keyboard Reference**

# Keyboard Reference

## The Keyboard

Figure 17

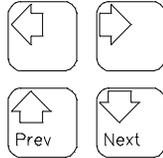
The Keyboard



## Direction Keys

Figure 18

### The Direction Keys



The direction keys, **Next ▼**, **Prev ▲**, **▶** and **◀**, allow you to select different options and move the cursor in the display.



If cursor is on left of display, **Next ▼** or **Prev ▲** displays next or previous line of list.

If cursor is in entry field, enter numeric value, press **Next ▼** or **Prev ▲** to display next or previous choice, or press **Escape** to turn off a function.



Moves cursor to the entry field on right or to the next digit in a numeric entry field.

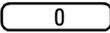
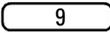


Moves cursor to previous character or entry field on left or to previous digit in numeric entry field.

---

## Numeric Keys

**Figure 19****The Numeric Keys**

Pressing the numeric keys  through  display the numbers 0 through 9 in numeric entry fields.



Pressing  displays a decimal point (.) in numeric entry fields.



Pressing  displays a minus sign (-) in numeric entry fields.

---

## Parameter and Function Entry Keys

Figure 20

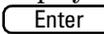
### The Parameter and Function Entry Keys



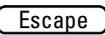
Pressing  enters value of parameter or function displayed. When you begin to edit a display the cursor will be an exclamation mark **!**. When you press  the cursor becomes a rectangle **■**.

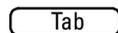
A **!** cursor means the display is being edited and the displayed value is not necessarily the same as the actual setting of the parameter.

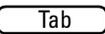
A **■** cursor means the displayed value is the same as the actual setting.

When editing a display the detector's parameter or function is not changed until you press .



Pressing  displays original value of parameter or function.



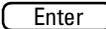
Pressing  has no function.

## Analysis Control Keys

**Figure 21**

### Analysis Control Keys



You use the analysis control keys,  and , with the  key to start and stop an analysis.



Pressing   starts an analysis. Remember that you can use the REMOTE connectors or the GPIB interface to start an analysis from another device, for example, your injector.

In **SWEEP** mode, the detector closes the contact of the EXT CONTACT connector to control the pen-down function of an X-Y plotter.

In all other modes, the detector closes the contact of the EXT CONTACT connector for 1 s so that other instruments in your system can be started by the detector.



Pressing   stops an analysis. Remember that you can use the REMOTE connectors or the GPIB interface to stop an analysis from another device, or you can use the detector's **STOPTIME** parameter to handle automation functions like **ZERO BALANCE**, **INCREMENT** or **PRETREAT**.

In **SWEEP** mode, the detector opens the contact of the EXT CONTACT connector (to control the pen-up function of an X-Y plotter).

---

**NOTE**

The detector does not send out start and stop signals through the REMOTE connectors. The start and stop lines of these connectors are for input of start and stop signals to the detector.

---

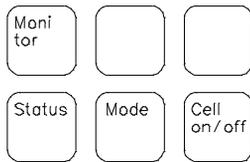
---

## Display and Command Functions

You access the display and command functions using the block of keys on the left of the keyboard.

**Figure 22**

### Display and Command Functions



#### Monitor

Pressing **Monitor** displays the potential, cell current, percentage of output at INTEGRATOR connector and status of the detector, for example:

```
0.600 V  59.92 nA  10 %  cell
```

Where:

**0.600 V** is the potential in volts;

**59.92 nA** is the cell current in nano-amps or micro-amps;

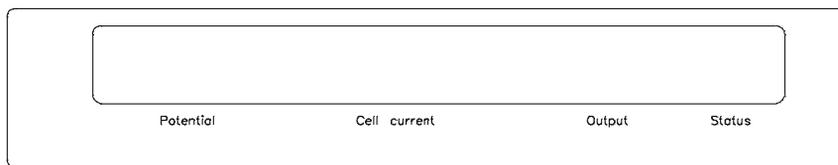
**10 %** is the percentage of output (10% means 100 mV at 1 V full scale);

**cell** is a short status message, see below.

To help you read the monitor display the front panel is labeled with the words *Potential*, *Cell current*, *Output* and *Status* below the corresponding part of the display.

Figure 23

## The Display



Short status messages that can appear in the monitor display are **leak**, **test**, **cell**, **incr**, **dyn**, **zero**, **full**, **und**, **over**, **temp**, **pret**, **prep**, **drif** or **hpib**.

In **REFERENCETEST** mode the monitor display is:

```
Reference  -180.3 mV
```

If a solvent thermostat is installed and **temp** error is detected, the monitor display is:

```
check thermostat cable
```

Pressing **Monitor** **Next** ▼ displays the drift monitor, for example:

```
Drift      19.05 nA/min
```

Where the value is the drift in  $\text{nAmin}^{-1}$  or  $\mu\text{Amin}^{-1}$  (or  $\text{mVmin}^{-1}$  in **REFERENCETEST** mode). Use the drift monitor to check the stability of the baseline before you start running analyses.

In **DIFFERENTIAL** mode the drift monitor has a different definition:

```
Cur1  72.11 nA  Cur2  32.30 nA
```

Where **Cur1** and **Cur2** are the currents measured at the differential potentials, see “The DIFFERENTIAL Mode” on page 72.

**Status**

Pressing **Status** displays the current operating condition of your detector, for example:

```
PRERUN      1049A ready
```

**Display and Command Functions**

<b>RUN 3.56</b>	<b>1049A ready</b>
-----------------	--------------------

<b>POSTRUN 2.03</b>	<b>1049A ready</b>
---------------------	--------------------

<b>WAIT</b>	<b>notready</b>
-------------	-----------------

When a time is displayed during **PRERUN** the set prepare time is running (the time runs down from the set value to zero).

During **RUN** the detector displays the time elapsed since the start of the analysis (  pressed or start signal received through REMOTE or GPIB connector).

During **POSTRUN** the detector displays the time remaining (the time runs down from the set value to zero). The posttime starts when **STOPTIME** elapsed, (  pressed or stop signal received through REMOTE or GPIB connector). When the **POSTTIME** has elapsed the detector will display the **PRERUN** status again.

**WAIT** is a special status for when the detector is working with an GPIB controller. During **WAIT** the controller is reading data from the detector which generates a **not ready** message. If you turn-OFF line power to the GPIB controller, you must reset the detector using the **RESET GPIB CONTROL** function.

The table below shows the conditions of the status lamps.

**Table 8****Conditions of Status Lamps**

<b>Status</b>	<b>Run Lamp</b>
PRERUN	OFF
RUN	ON
POSTRUN	Flashing
WAIT	Flashing

On the right of the display the detector will display an **ERROR**, **CAUTION** or **NOTREADY** message when a corresponding condition is detected (**CAUTION** messages will flash). Press  to display the full error message. Below is a list of the full error messages together with the corresponding short forms that are shown in the monitor display.

**Display and Command Functions****Status Message Short Form**

<b>ERROR:</b> leak, reset sensor	leak
<b>ERROR:</b> leak sensor failed	leak
<b>ERROR:</b> out of temperature range	temp
<b>ERROR:</b> detector overflow	gpib
<b>CAUTION:</b> test is running	test
<b>CAUTION:</b> cell is off	cell
<b>CAUTION:</b> cell not connected	cell
<b>CAUTION:</b> increment is on	incr
<b>CAUTION:</b> reduced dynamic range	dyn
<b>CAUTION:</b> analog out underflow	und
<b>CAUTION:</b> analog out underflow	over
<b>NOTREADY:</b> zero setting	zero
<b>NOTREADY:</b> out of fullscale	full
<b>NOTREADY:</b> temperature high	temp
<b>NOTREADY:</b> temperature low	temp
<b>NOTREADY:</b> pretreatment running	pret
<b>NOTREADY:</b> preparetime running	prep
<b>NOTREADY:</b> drifttrigger is on	drif

**Display and Command Functions**

If there are several error messages they will appear in the same order as in the list above.

**NOTE**

For details of what to do when the detector displays an error message, see Chapter 6 “Troubleshooting Your Detector”.

**Mode**

The **Mode** key allows you to select the mode of detection you want to use for your application. Press **Next** ▼ or **Prev** ▲ to choose mode, then press **Enter**. Modes are:

**Mode**

**AMPEROMETRY**

**Next** ▼

**PRETREAT**

**Next** ▼

**SWEEP**

**Next** ▼

**PULSE**

**Next** ▼

**DIFFERENTIAL**

**Next** ▼

**TEST1**

**Next** ▼

**TEST2**

**Next** ▼

**TEST3**

**Next** ▼

**TEST4**

**Next** ▼

**REFERENCETEST**

**The AMPEROMETRY Mode**

The **AMPEROMETRY** mode is a standard electrochemical detection mode where the detector applies a constant DC-potential across the analyte (between the working and reference electrode). The detector measures and amplifies the current generated by electrons (released by the electrolysis of the analyte) flowing between the working electrode and the analyte.

For more details of the theory of electrochemical detection, see *Electrochemical Detection in High Performance Liquid Chromatography--A Practical Primer*.

**The PRETREAT Mode**

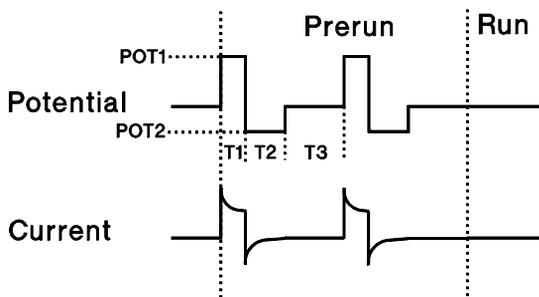
The **PRETREAT** mode allows you to clean the surface of the working electrode electrochemically before or between analyses. The detector does

this pretreatment outside of the RUN status, sending a not ready signal through the REMOTE or GPIB connectors (to inhibit your injector from injecting the sample). When pretreatment is complete and the posttime or preparetime (depending on the settings of the remote control) has elapsed, the detector removes the not ready signal, allowing your injector to inject the sample. If you have set the **DRIFTTRIGGER**, the detector will remove the not ready signal when the current drift drops below the set value. Use this mode when the electro-active species foul the working electrode relatively slowly (use the **PULSE** mode when the species rapidly foul the electrode).

For pretreatment you can specify two different potentials (for example, a positive and a negative potential), times for how long the detector should apply the potentials and the number of times (cycles) the potentials should be applied. The final (third) potential is the working potential you set before pretreatment, see Figure 24.

Figure 24

#### The Pretreatment Mode



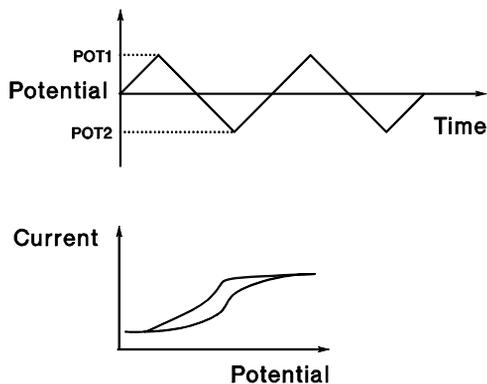
With glassy carbon electrodes very low frequencies ( $<2$  Hz) are typically applied as compared to noble metal electrodes. The time T3 is usually set to 0 in this mode.

For details of the potentials and times, see “PRETREAT PARAMETERS” on page 84.

#### The SWEEP Mode

The **SWEEP** mode allows you to do hydrodynamic voltammograms by varying the applied potential over a period of time. Use this mode when you are developing a new method and need to find the potential that gives you the best sensitivity and/or selectivity. In **SWEEP** mode you can specify two

potentials (for example, a positive and a negative potential) and a rate at which potential should change, see Figure 25.

**Figure 25****The Sweep Mode**

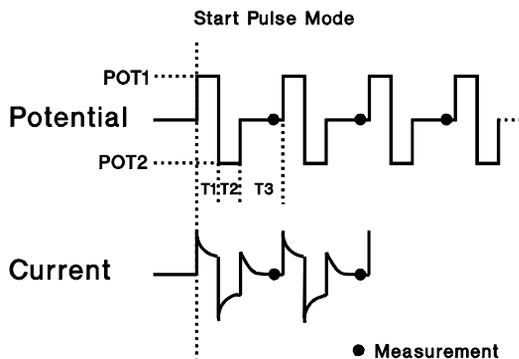
For details of the potentials and sweep rate, see “SWEEP PARAMETERS” on page 87.

**The PULSE Mode**

The **PULSE** mode allows you to clean the surface of the working electrode electrochemically during an analysis. Use this mode when the electro-active species foul the working electrode rapidly (use the **PRETREAT** mode when the species foul the electrode relatively slowly).

For pulse mode you can specify two different potentials (for example, a positive and a negative potential) and times for how long the detector should apply the potentials. The third potential is the preset working potential that is also used for sampling the data, see Figure 26.

Figure 26

**The Pulse Mode**

For details of the potentials and times, see “PULSE PARAMETERS” on page 89.

**NOTE**

In general you will achieve better results with gold or platinum working electrodes than with glassy carbon, because the electrochemical response of these electrode materials is faster and also better, depending on the application. The glassy carbon electrode has a longer response behaviour than noble metal electrodes, resulting in significantly longer time settings and therefore worse signal-to-noise performance.

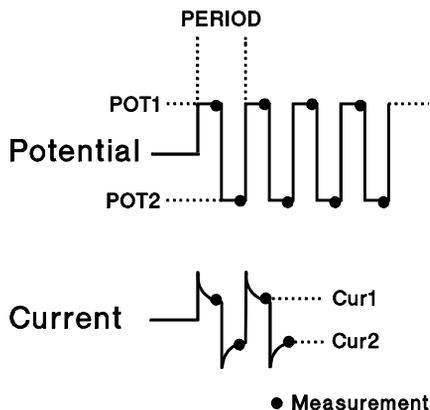
Time  $T_3$  should always be much larger than  $T_1$  or  $T_2$  to prevent discharge currents of double layer capacitance from being part of the measurement.

**The DIFFERENTIAL Mode**

In **DIFFERENTIAL** mode the detector applies square wave pulses of potential continuously throughout the analysis, see Figure 27.

Figure 27

### The Differential Mode



The detector measures the current at the end of each pulse. The output current is the difference between the two currents measured within the time period:  $\text{Output} = \text{Cur1} - \text{Cur2}$ .

The monitor display shows the potential difference and the drift monitor shows the 2 currents **Cur1** and **Cur2**, for example:

```
Cur1 -180.30 nA  Cur2 -109.05 nA
```

For details of the potentials and times, see “DIFFERENTIAL PARAMETERS” on page 91.

### The TEST1 Mode

**TEST1** is the dummy cell test that you use to test the complete electrochemical detector, see Figure 14 on page 46.

### The TEST2 Mode

**TEST2** is the amperometer/zero test that you use to test the complete amperometer, including the electronic parts that allow an offset of the cell current (zero level and background current), see Figure 13 on page 45.

### The TEST3 Mode

**TEST3** is the amperometer test that you use to test the amperometer noise. The cell is not connected electronically, and the electronic parts that allow an

**Display and Command Functions**

offset of the cell current (zero level and background current) are disabled, see Figure 12 on page 44.

**The TEST4 Mode**

**TEST4** is the analog output test that you use to test the noise contributed by the analog output, see Figure 11 on page 43.

**The REFERENCETEST Mode**

**REFERENCETEST** is a test for determining the actual potential of the reference electrode with respect to ground, under flow conditions. For reliable results at least 1 hour equilibration time is needed, see “Reference Electrode Test” on page 49.

**Cell on/off**

The **Cell on/off** key allows you to select the functions of the electrochemical cell. When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

<b>Cell on/off</b>	<b>CELL</b>
<b>Next ▼</b>	<b>AUTO CELL</b>
<b>Next ▼</b>	<b>AFTER</b>

**CELL**

Turns cell ON or OFF. Select **ON** or **OFF** using **Next ▼** or **Next ▼** and press **Enter**.

If the cell is turned OFF, the detector generates the message **CAUTION: cell is off**. If the cell is turned ON, but no cell is connected, the detector generates the message **CAUTION: cell not connected**.

When the cell is OFF, the detector calibrates its electronics automatically. The working and auxiliary electrodes are galvanically disconnected. When the cell is OFF, all not ready conditions are suppressed so that the detector is in a ready state.

**NOTE**

Always turn-ON the solvent pump before you turn-ON the cell. And, always turn-OFF the cell before you turn-OFF the solvent pump.

## Display and Command Functions

### **AUTO CELL**

Turns cell ON or OFF at a specified time, see **AFTER** below. Select **CELL ON** or **CELL OFF** using  or  and press .

### **AFTER**

Specifies time when cell will be turned ON or OFF (according to the **AUTO CELL** setting, see above).

Limits: 0 to 9999 min.

When you set **AFTER** to 0, the **AUTO CELL** function is inactive. If the **AFTER** time elapses and the detector is still in the **RUN** status, the cell will be turned-ON or OFF when the run has finished. You must set the **AFTER** time for every procedure in which you want to use the **AUTO CELL** function, because of an automatic reset to zero after each time has elapsed.

This mode is especially useful when the working electrodes require long set up times and high potentials need to be applied for long periods between several analyses.

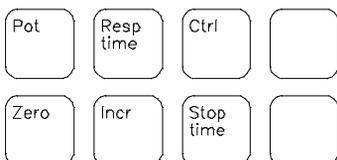
---

## Parameters

You access the parameters using the block of keys near the center of the keyboard.

**Figure 28**

### Parameter Keys



**Pot**

The **Pot** key allows you to select the potential functions of the electrochemical cell. When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

**Pot**

**POTENTIAL**

**Next ▼**

**UPPER LIMIT**

**Next ▼**

**LOWER LIMIT**

---

### NOTE

We strongly recommend that you use potentials only as high as necessary, because of possible irreversible absorption of reaction products on the working electrode when continuously using high potentials.

#### POTENTIAL

Sets the potential to be applied between the working electrode and the analyte.

Limits: +2.000 through -2.000 V in steps of 0.001 V or depending on limits set by **UPPER LIMIT** and **LOWER LIMIT**.

#### UPPER LIMIT

Sets a safe upper potential limit.

**Parameters**

Limits: +2.000 through -2.000 V in steps of 0.001 V. The **UPPER LIMIT** must be greater than the **LOWER LIMIT**.

**LOWER LIMIT**

Sets a safe lower potential limit.

Limits: +2.000 through -2.000 V in steps of 0.001 V.

The default settings for glassy carbon are +1.4 V and -0.4 V. These limits are not valid for pulse or sweep potentials. If you are using the increment feature, the potential increments will stop at the **UPPER LIMIT** or **LOWER LIMIT**.

Zero

The **Zero** key allows you to select the zero functions of the detector. When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

**Next ▼**

**ZEROCURRENT**

**Zero**

**ZERO BALANCE**

**Next ▼**

**ZEROLEVEL**

**Next ▼**

**ZERO CONTROL**

**ZERO BALANCE**

Limits: none, press **Enter** to do balance.

Balancing adjusts the actual analog signal to an output level which is set by **ZEROLEVEL**. Internally, current compensation recalibrates the amperometer to 0 by setting a new **ZERO CURRENT**. If any other automatic functions are currently active (pretreat or increment) the balance will be done after the **POSTTIME** or **PREPARETIME** has elapsed or after the baseline drift has reached a value lower than specified by the **DRIFTTRIGGER**. Typically you would set a **POSTTIME** if the function is stop-controlled, or a **PREPARETIME** if the function is prepare-controlled.

**ZEROLEVEL**

Limits: 1 through 99% in steps of 1%.

**ZEROLEVEL** is the present value with respect to the full scale definition of the **INTEGRATOR** and **AUXILIARY** outputs. For example, with 1 V full scale

## Parameters

a **ZEROLEVEL** of 25% will set the baseline to 250 mV after balancing with **ZERO BALANCE**.

### **ZERO CONTROL**

**ZERO CONTROL** controls automatic balancing (a manual balance can still be done regardless of the **ZERO CONTROL** setting).

Limits:

- OFF** No automatic **ZERO BALANCE** will be done.
- PREPARE** When the detector receives a prepare signal through the **REMOTE** or **GPIB** connectors the **PREPARETIME** will start. With this setting a balance will be done automatically when the **PREPARETIME** has elapsed.
- STOP** When the detector receives a stop signal through the **REMOTE** or **GPIB** connectors, through the keyboard or through **STOPTIME** elapsing, the **POSTTIME** will start. With this setting a balance will be done automatically when the **POSTTIME** elapsed.
- DRIFTTRIGGER** If the **DRIFTTRIGGER** is set to be greater than zero, the detector generates a not ready message until the drift drops below the set value. At this point a balance will be done automatically (note that the detector waits a certain time to make sure that the drift remains below the set **DRIFTTRIGGER** value).

### **ZEROCURRENT**

Limits: +500.0 through -500.0 nA in steps of 0.1 nA.

This parameter is set automatically by activating the **ZERO BALANCE** function. Using this parameter you can control or even correct internal current compensation, if you need special values at the analog outputs.

### **Resptime**

The **Resptime** key allows you to select the response time and peakwidth functions of the detector. Response time and peakwidth are the same parameter, but they are expressed in different chromatographic terms. By choosing the appropriate response time or peakwidth you can vary the number of data points which make up a peak and so influence peak shape and signal-to-noise ratio.

**Parameters**

When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

**Resptime**                      **RESPONSETIME**  
**Next ▼**                              **PEAKWIDTH**

**RESPONSETIME and PEAKWIDTH**

Set number of data points which make up a peak.

**Table 9****Limits for Peakwidth and Response Time**

Peakwidth [min]	Response time [s]
0.01	0.13
0.03	0.25
0.05	0.50
0.10	1.00
0.20	2.00
0.40	4.00
0.80	8.00

**NOTE**

Never set the **RESPONSETIME** or **PEAKWIDTH** lower than necessary—you will lose sensitivity.

**Incr**

The **Incr** key allows you to select the functions of the detector that control automatic changing of the applied potential for doing hydrodynamic voltamograms. When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

**Incr**                              **INCREMENT**  
**Next ▼**                              **REPETITIONS**  
**Next ▼**                              **DRIFTTRIGGER**

### Handling Equilibration Times with the Increment Function

The general problem of defining an appropriate wait time after a potential increment (caused by different equilibration times) can be solved by two different methods.

The **DRIFTTRIGGER** is based on current drift and defined in amps per minute. The drift is displayed in the drift monitor, press **Monitor** **Next** . The actual drift value is compared to your current per minute setting of the **DRIFTTRIGGER**. Before reaching the trigger condition, the detector generates a not ready signal, preventing an injection (assuming that the injector is able to receive such a signal). When the **DRIFTTRIGGER** is triggered the detector waits an additional 40 s to ensure that the drift remains below the set value. Your wait time is thus set automatically to an optimum value. This is especially useful when you are using a wide potential range which requires significantly different equilibration times.

With a certain knowledge of the equilibration time you can set the **POSTTIME** to put the detector into a not ready condition after a stop signal has been received. After the **POSTTIME** has elapsed the detector removes the not ready condition and the injector can start with the next injection.

#### INCREMENT

Sets the amount by which the applied potential is increased from analysis to analysis.

Limits: +2.000 V through -2.000 V in steps of 0.001 V. When **INCREMENT=0** the increment function is disabled. When the increment is enabled, the detector changes the potential in steps from the set potential through the upper limit or lower limit after receiving a stop signal through the REMOTE or GPIB connectors, through the keyboard or through the **STOPTIME** elapsing (see example below). If you are using a non-Agilent Technologies device which cannot supply a stop signal, start the detector through the REMOTE connector and use the **STOPTIME** to trigger the increment function.

The most effective way to use the **INCREMENT** function is with the **DRIFTTRIGGER** (if the injector accepts a ready/not ready signal through a remote connection) because the equilibration (wait) times necessary will vary depending on the applied potential.

#### REPETITIONS

Sets number of times an analysis (at a specific potential) is repeated after having received a stop signal.

**Parameters**

Limits: 1 through 99 in steps of 1.

The second number in the repetition display shows the actual number of analyses with the same potential values.

With the increment function the potential of the working electrode will normally be changed at a stop signal. In differential mode the potentials **DIFF POT1** and **DIFF POT2** are changed in the same way.

For example—with the following parameter settings:

```
■POTENTIAL = 0.400 ; Volt
```

```
■UPPER LIMIT = 1.200 ; Volt
```

```
■INCREMENT = 0.200 ; Volt
```

```
■REPETITIONS = 2 ; 2
```

The detector will analyze at the following potentials: 0.400, 0.400, 0.600, 0.600, 0.800, 0.800, 1.000, 1.000, 1.200, 1.200, 1.200 and so on.

**DRIFTTRIGGER**

Sets the drift monitor to create a not ready signal at the REMOTE an GPIB connector when the drift is greater than a specified value.

Limits: 0.1 through 500.0 nA/min in steps of 0.1 nA/min. When **DRIFTTRIGGER=0** the function is disabled.

**Stoptime**

The **Stoptime** key allows you to select the timing functions of the detector. When cursor is in left of display, press **Next ▼** or **Next ▼** to choose function. Functions are:

**Stoptime**

**STOPTIME**

**Next ▼**

**POSTTIME**

**Next ▼**

**PREPARETIME**

## Parameters

### STOPTIME

Sets the time limit for an analysis and creates a stop signal for the detector. Remember that you can also use the REMOTE or GPIB connectors to stop an analysis from another device, or you can use  .

Limits: 0 through 1440.00 min in steps of 0.01 min.

**STOPTIME=0** sets no limit—you must stop the analysis using   or through the REMOTE or GPIB connectors.

In **SWEEP** mode, the **STOPTIME** has no function.

With **STOPTIME** you can activate several programmable functions if a start signal has been generated through the REMOTE or GPIB connectors or through the keyboard ( ). These functions are **INCREMENT**, **PRETREAT** mode and the **ZERO BALANCE** function.

### POSTTIME

Sets limit for a time delay after an analysis. The detector will display **POSTRUN** and **notready**, and send a not ready signal through the REMOTE or GPIB connector, inhibiting your injector from injecting the next sample.

Limits: 0 through 1440.00 min in steps of 0.01 min.

**POSTTIME=0** sets no delay—the detector will display the **PRERUN** status and be ready for the next analysis, assuming that no automatic functions are active.

With **POSTTIME** you can give several programmable functions an appropriate time frame after the detector has received a stop signal by elapsed **STOPTIME**, through the REMOTE or GPIB connectors or through the keyboard ( ). These functions are the **PRETREAT** mode and the **ZERO BALANCE** function.

### PREPARETIME

Sets limit for a time delay before an analysis. The detector will display **PRERUN**, the remaining **PREPARETIME** and **notready**, and send a not ready signal through the REMOTE or GPIB connector, inhibiting your injector from injecting the next sample.

Limits: 0 through 1440.00 min in steps of 0.01 min.

**PREPARETIME=0** sets no delay

**Parameters**

With **PREPARETIME** you can give an appropriate time frame for the **PRETREAT** mode and the **ZERO BALANCE** after the detector has received a prepare signal through the REMOTE or GPIB connectors.

The  key allows you to access several sets of parameters that control output scaling, the different detection modes and the solvent thermostat (if installed). When cursor is in left of display, press  or  to choose parameter set and then press . Parameters sets are:

**SCALING PARAMETERS**

**PRETREAT PARAMETERS**

**SWEEP PARAMETERS**

**PULSE PARAMETERS**

**DIFFERENTIAL PARAMETERS**

**THERMOSTAT PARAMETERS**

**RESET FUNCTIONS**

**SCALING PARAMETERS**

The **SCALING PARAMETERS** allow you to set the scaling of the output at the INTEGRATOR and AUXILIARY connectors or of the detector preamplifier hardware. When **SCALING PARAMETERS** is displayed press , then press  or  to choose parameter. Parameters are:

**REC RANGE**

**INSTRUMENT FULLSCALE**

**POLARITY**

**REC RANGE**

Ranges are implemented in a 1:2:5 sequence. Depending on the setting of **INSTRUMENT FULLSCALE**, the **REC RANGE** has the same sensitivity on the AUXILIARY output as on the INTEGRATOR output, or has higher sensitivity in 12 steps. **ZERO BALANCE** also sets the AUXILIARY output to a preset **ZEROLEVEL**.

**Parameters****INSTRUMENT FULLSCALE**

The default range for thin-layer cells is 0.5  $\mu\text{A}$  fullscale. With **INSTRUMENT FULLSCALE** you are able to change the basic hardware definition of the pre-amplifier. With potential pulse or sweep applications, high capacitive currents typically require a 500  $\mu\text{A}$  setting. Besides these applications you should not need to change the **INSTRUMENT FULLSCALE** setting. We recommend to turn-OFF the cell when changing the **INSTRUMENT FULLSCALE** and wait a short time before turning-ON the cell, because the detector recalibrates to give a precise signal current display.

Limits: 0.05  $\mu\text{A}$ , 0.5  $\mu\text{A}$  or 500  $\mu\text{A}$ .

**POLARITY**

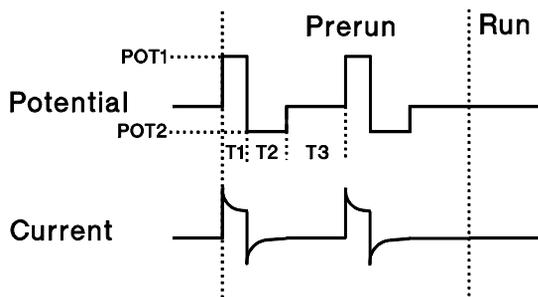
For negative peaks you are able to output complementary data or analog signals by setting the **POLARITY** to **OXIDATION** or **REDUCTION**. After switching the **POLARITY**, always balance the detector (**ZERO BALANCE**) to ensure proper operation.

Limits: **OXIDATION** or **REDUCTION**.

**PRETREAT PARAMETERS**

The **PRETREAT PARAMETERS** allow you to set the parameters of the detector's **PRETREAT** mode, see “The **PRETREAT** Mode” on page 69 and Figure 29.

**Figure 29**

**The Pretreatment Mode**

When **PRETREAT PARAMETERS** is displayed press , then press  or  to choose parameter. Parameters are:

**Parameters**

Enter	<b>PRETREAT CYCLES</b>
Next ▼	<b>PRETREAT POT1</b>
Next ▼	<b>PRETREAT POT2</b>
Next ▼	<b>PRETREAT TIME1</b>
Next ▼	<b>PRETREAT TIME2</b>
Next ▼	<b>PRETREAT TIME3</b>
Next ▼	<b>PRETREAT CONTROL</b>

**Using PRETREAT Mode**

Cell pretreatment is a technique that requires detailed information on how to use it. We will therefore give you some general recommendations before explaining each of the **PRETREATMENT PARAMETERS**.

Each type of working electrode material has its own optimum pulse shapes and time durations for pretreatment. Typical pretreatment procedures with glassy carbon are pure DC anodic or cathodic potential pretreatment or slow (seconds) square-wave pulses with high potentials. For noble metal electrodes we recommend 3 potential switching of several milliseconds.

After pretreatment the cell will need several seconds or even minutes to equilibrate. You can use the **POSTTIME** or **PREPARETIME** parameters (depending on your start/stop control) to define how long the detector remains in a not ready condition, preventing the injector from injecting a sample. A further powerful tool for efficient equilibration control is the **DRIFTTRIGGER**.

**NOTE**

Always keep in mind when using **POSTTIME** or **PREPARETIME** that the setting has to be much higher than the number of **PRETREAT CYCLES** multiplied by the sum of the **PRETREAT TIMES**.

$$\text{Total Time} = \text{CYCLES} (\text{TIME1} + \text{TIME2} + \text{TIME3})$$

Setting any **PRETREAT TIME** to 0 means that the corresponding change in potential will not be done.

Pretreatment can only be done using the PRETREAT mode.

## Parameters

### **PRETREAT CYCLES**

Sets number of times the detector goes through the pretreatment cycle of applying the two pretreatment potentials **PRETREAT POT1** and **PRETREAT POT2**.

Limits: 1 through 999 in steps of 1.

### **PRETREAT POT1**

Sets first potential that detector applies during a pretreatment cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **PRETREAT POT2**

Sets second potential that detector applies during a pretreatment cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **PRETREAT TIME1**

Sets time that detector applies first potential, **PRETREAT POT1**. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **PRETREAT TIME2**

Sets time that detector applies second potential, **PRETREAT POT2**. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **PRETREAT TIME3**

Sets time that detector applies the working potential, before starting next pretreatment cycle. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **PRETREAT CONTROL**

Sets type of control over pretreatment mode.

Limits:

**MANUAL** For manual activation of pretreatment, set **MODE** to **PRETREAT** and press . For repetitive

**Parameters**

pretreatments, press   repeatedly.

**PREPARE** For automatic pretreatment with the prepare signal through REMOTE or GPIB connectors, you must set the **PREPARETIME** to be longer than the duration of each cycle multiplied by the number of cycles, or use the **DRIFTTRIGGER**.

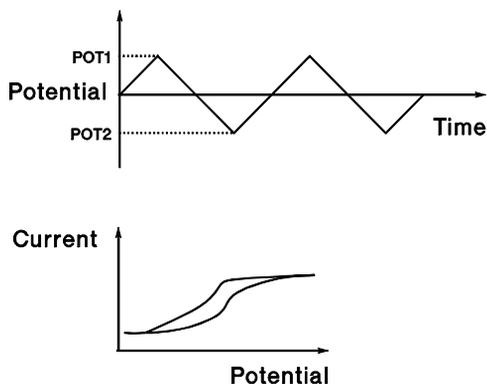
**STOP** For automatic pretreatment activated by a stop signal through REMOTE or GPIB connectors, elapsed **STOPTIME** or keyboard entry, you must set the **POSTTIME** to be longer than the duration of each cycle multiplied by the number of cycles, or use the **DRIFTTRIGGER**.

**SWEEP PARAMETERS**

The **SWEEP PARAMETERS** allow you to set the parameters of the detector's **SWEEP** mode, see "The SWEEP Mode" on page 70 and Figure 30.

Figure 30

**The Sweep Mode**



When **SWEEP PARAMETERS** is displayed press , then press  or  to choose parameter. Parameters are:

- SWEEP CYCLES**
- SWEEP POT1**
- SWEEP POT2**
- SWEEP RATE**

**Parameters****Using SWEEP Mode**

Before using the **SWEEP** mode, set the **INSTRUMENT FULLSCALE** to 500  $\mu\text{A}$ , because of high capacitive charging currents. Now set the desired **ZEROLEVEL** and balance the detector (**ZERO BALANCE**). The **REC RANGE** setting defines the current sensitivity of the **AUXILIARY** output. The **INTEGRATOR** output defines the potential range of  $-2\text{ V}$  through  $+2\text{ V}$  as follows:

**Table 10****Output at INTEGRATOR Connector**

Potential	INTEGRATOR Output
+2 V	+1 V
0 V	+0.5 V
-2 V	0 V

The **EXT CONTACT** connector provides pen-up and pen-down control for an X-Y plotter. Closing the contact moves the pen down.

You start the **SWEEP** mode by setting the **MODE** to **SWEEP** and then pressing  .

You can stop the **SWEEP** mode before it has finished by pressing  . Note that the **STOPTIME** parameter has no function in **SWEEP** mode.

You can calculate the duration of your sweep from the following formula.

$$T_{\text{sweep}} = \frac{1000}{R} [ |P1 - P| + (|P1 - P2|(2C - 1)) + |P - P2| ]$$

Where:

C is number of **SWEEP CYCLES**

P is working potential in [V]

P1 is **SWEEP POT1** in [V]

P2 is **SWEEP POT2** in [V]

R is **SWEEP RATE** in [ $\text{mVs}^{-1}$ ]

## Parameters

$T_{\text{sweep}}$  is **SWEEP** duration in [s]

### **SWEEP CYCLES**

Sets number of times the detector goes through the sweep cycle of applying the two sweep potentials **SWEEP POT1** and **SWEEP POT2**. The starting potential is the set working potential.

Limits: 1 through 999 in steps of 1.

### **SWEEP POT1**

Sets first potential that detector applies during a sweep cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **SWEEP POT2**

Sets second potential that detector applies during a sweep cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **SWEEP RATE**

Sets rate at which detector changes the applied potential during a sweep cycle.

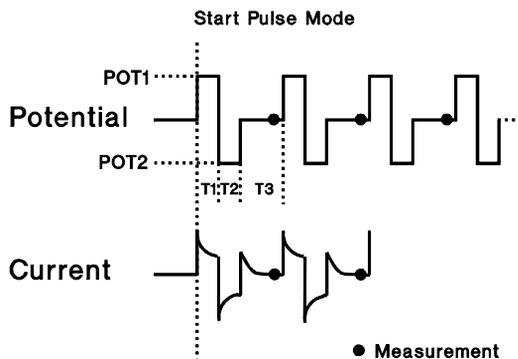
Limits: 1 through 1000  $\text{mVs}^{-1}$  in steps of 1  $\text{mVs}^{-1}$ .

### **PULSE PARAMETERS**

The **PULSE PARAMETERS** allow you to set the parameters of the detector's **PULSE** mode, see “The PULSE Mode” on page 71 and Figure 31.

Figure 31

## The Pulse Mode



When **PULSE PARAMETERS** is displayed press , then press  or  to choose parameter. Parameters are:

<input type="text" value="Enter"/>	<b>PULSE POT1</b>
<input type="text" value="Next ▼"/>	<b>PULSE POT2</b>
<input type="text" value="Next ▼"/>	<b>PULSE TIME1</b>
<input type="text" value="Next ▼"/>	<b>PULSE TIME2</b>
<input type="text" value="Next ▼"/>	<b>PULSE TIME3</b>

## Using PULSE Mode

Depending on the electrode materials and surface areas, you must use completely different methods to get good results. For example, you should not use pulses much lower than 500 ms with a glassy carbon electrode. In contrast, you can use pulses of just a few milliseconds with noble metal electrodes. You should set **PULSE TIME3** to be much longer (factor of 5 or more) than **PULSE TIME1** and **PULSE TIME2** to prevent the detector from measuring capacitance discharge currents instead of faradic currents.

You can set the time for a potential pulse to a minimum of 4.55 ms and upwards in multiples of 4.55 ms. The detector automatically rounds up any value you enter.

For pure anodic or cathodic pretreatment, set the times that are not used to 0. With this setting the corresponding potentials will not be applied. The **INSTRUMENT FULLSCALE** setting of 0.5  $\mu\text{A}$  might not be sufficient for some applications. In these cases set **INSTRUMENT FULLSCALE** to 500  $\mu\text{A}$  (remember to turn-OFF the cell for recalibration).

## Parameters

### **PULSE POT1**

Sets first potential that detector applies during a pulse cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **PULSE POT2**

Sets second potential that detector applies during a pulse cycle.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

### **PULSE TIME1**

Sets time that detector applies first potential, **PULSE POT1**. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **PULSE TIME2**

Sets time that detector applies second potential, **PULSE POT2**. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **PULSE TIME3**

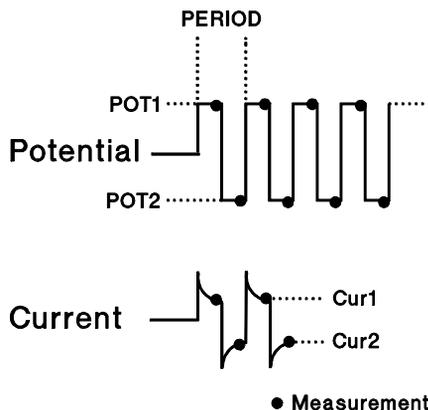
Sets time that detector applies the working potential, before starting next pulse cycle. The value you enter is rounded up by the detector to multiples of 4.55 ms.

Limits: 0 through 999 ms in steps of 1 ms.

### **DIFFERENTIAL PARAMETERS**

The **DIFFERENTIAL PARAMETERS** allow you to set the parameters of the detector's **DIFFERENTIAL** mode, see “The DIFFERENTIAL Mode” on page 72 and Figure 32.

Figure 32

**The Differential Mode**

When **DIFFERENTIAL PARAMETERS** is displayed press , then press  or  to choose parameter. Parameters are:

<input type="button" value="Enter"/>	<b>DIFF PERIOD</b>
<input type="button" value="Next ▼"/>	<b>PULSE POT1</b>
<input type="button" value="Next ▼"/>	<b>DIFF POT2</b>

**Using DIFFERENTIAL Mode**

You can use differential pulse techniques to solve several problems, such as poor selectivity, or high or drifting background currents.

As a selectivity tool you should bear in mind that differential pulse techniques provide a specific higher differential current at the potential at which the substance to be detected has its largest increase in sensitivity. This is typically the case at the half wave potential of a compound, which is the potential where the sensitivity reaches 50% of its maximum. The parameters for one of the substances to be detected are optimized when the two potentials are set between the beginning and end of the sensitivity transition found in a hydrodynamic voltammogram. Large potential differences reduce selectivity and sensitivity, but create huge differential currents.

Before starting a run we strongly recommend that you set the detector to do a **ZERO BALANCE**, because you do not see background currents in the monitor display due to subtraction of neighboring current values. When the detector does a balance in differential mode, it compensates using the average of the 2 currents: **Cur1** and **Cur2**. You can find out the amount of

**Parameters**

compensation by looking at the **ZERO CURRENT** which is set automatically by **ZERO BALANCE**.

The signal current shown in the monitor display (press ) is the differential part between to adjacent current samples. The differential monitor (press  ) shows the absolute current values, for example:

```
Cur1  72.11 nA  Cur2  32.30 nA
```

**DIFF PERIOD**

Sets duration of differential period. The detector applies the first potential (**DIFF POT1**) for half the time period and then the second potential (**DIFF POT2**) for the remaining half of the period. The value you enter is rounded up by the detector to multiples of 9.1 ms.

Limits: 9 through 1000 ms in steps of 1 ms.

**DIFF POT1**

Sets first potential that detector applies during the differential period.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

**DIFF POT2**

Sets second potential that detector applies during the differential period.

Limits: +2.000 V through -2.000 V in steps of 0.001 V.

**THERMOSTAT PARAMETERS**

The **THERMOSTAT PARAMETERS** allow you to set the parameters of the solvent thermostat. When **THERMOSTAT PARAMETERS** is displayed press , then press  or  to choose parameter.

Parameters are:

<input type="button" value="Enter"/>	<b>THERMOSTAT</b>
<input type="button" value="Next ▼"/>	<b>TEMPERATURE</b>

If solvent thermostat is not installed the detector will display:

```
■THERMOSTAT=OFF ;not installed
```

## Parameters

### THERMOSTAT

Turns thermostat ON or OFF. Select **ON** or **OFF** using  or  and press .

### TEMPERATURE

Sets temperature of solvent thermostat.

---

#### NOTE

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Take care when using settings above 40°C, dissolved gasses in the mobile phase might be released.

Limits: 20 through 60°C in steps of 1 degree.

The thermostat can heat only. Set the temperature a few degrees higher than the surroundings of the detector.

### RESET FUNCTIONS

The **RESET FUNCTIONS** allow you to reset parts of the detector selectively. When **RESET FUNCTIONS** is displayed press , then press  or  to choose function. Functions are:

<input type="button" value="Enter"/>	<b>RESET LEAKSENSOR</b>
<input type="button" value="Next ▼"/>	<b>RESET GPIB CONTROL</b>
<input type="button" value="Next ▼"/>	<b>RESET INSTRUMENT</b>
<input type="button" value="Next ▼"/>	<b>EC Detector 1049A (B 2947)</b>

#### RESET LEAKSENSOR

Resets the leak sensor. After a leak has occurred and you have corrected the fault, you must reset the leak sensor. To reset the leak sensor, select **RESET LEAKSENSOR** and press .

#### RESET GPIB CONTROL

This function is designed for when you turn-OFF line power to a controller connected through GPIB interface and a **WAIT** condition occurs. To reset the GPIB control, select **RESET GPIB CONTROL** and press .

**Parameters**

**RESET INSTRUMENT**

Resets all electronic parts and parameters of the detector to the default values. See Chapter 6 “Troubleshooting Your Detector” for details of when to use this function.

**EC Detector 1049A (B 2947)**

Displays firmware revision (**B 2947**) of your detector. Always quote this number, and the serial number of your detector, when you contact your Agilent Technologies engineer.



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## **Automating Your Detector**

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# Automating Your Detector

The Agilent 1049A electrochemical detector has many modes and functions which can be used in different combinations, to suit your own application needs. In this chapter we will describe several examples of practical applications. These examples will help you select the correct parameter settings for your particular application.

## Analyses Using ZERO BALANCE

*Problem:* You want to run a series of analyses in which the detector does a **ZERO BALANCE** before each analysis. The chromatogram is 20 min long and there should be about 5 min between each analysis.

### Example 1(a)

In this example you use an autosampler to send a prepare signal to the detector's REMOTE connector.

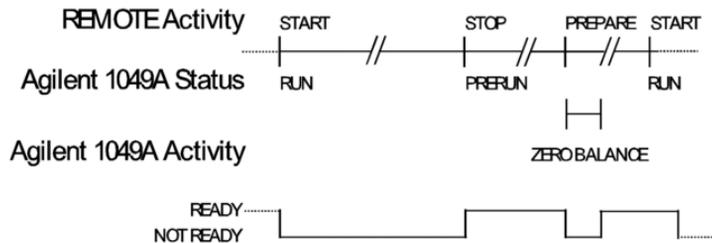
Set detector parameters as follows:

```
■ZERO CONTROL = PREPARE
```

```
■PREPARETIME = 0 ; off
```

Figure 33

### Detector Status in Example 1(a)



### Example 1(b)

In this example you use an autosampler to send start and stop signals to the detector's REMOTE connector.

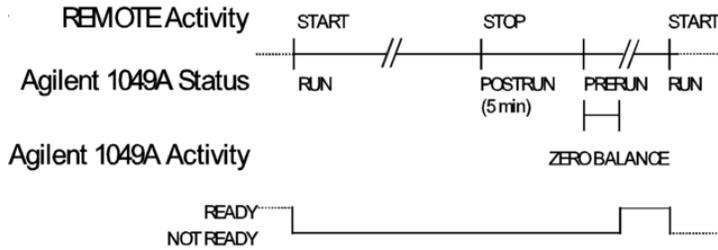
Set detector parameters as follows:

```
■ZERO CONTROL = STOP
```

```
■POSTTIME = 5.00 min
```

**Figure 34**

**Detector Status in Example 1(b)**



**Example 1(c)**

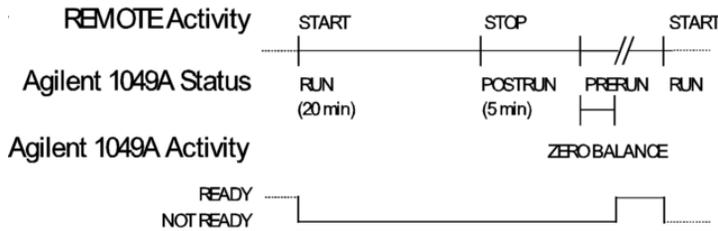
In this example you use an autosampler to send a start signal to the detector's REMOTE connector.

Set detector parameters as follows:

- **ZERO CONTROL = STOP**
- **STOPTIME = 20.00 min**
- **POSTTIME = 5.00 m**

**Figure 35**

**Detector Status in Example 1(c)**



---

## Analyses Using PRETREAT and ZERO BALANCE

*Problem:* You want to run a series of normal amperometric analyses whereby the working electrode is cleaned and reactivated by potential pulses between each analysis. In addition, the detector does a **ZERO BALANCE** before each analysis. The chromatogram is about 20 min long. Set the detector to **MODE=PRETREAT**.

The potential pulses are set as follows:

```
■PRETREAT CYCLES = 6
■PRETREAT POT1 = 1.400 ; V
■PRETREAT POT2 = -0.600 ; V
■PRETREAT TIME1 = 200 ; msec
■PRETREAT TIME2 = 300 ; msec
■PRETREAT TIME3 = 500 ; msec
```

The cleaning and reactivation pretreatment by the potential pulses will take:

$$T_{\text{pretreat}} = 6(200 + 300 + 500)\text{ms} = 0.1\text{min}$$

### Example 2(a)

In this example you use an autosampler to send a prepare signal to the detector's **REMOTE** connector.

Set detector parameters as follows:

```

■MODE = PRETREAT

■ZERO CONTROL = PREPARE

■PREPARETIME = 5.00 ; min

■STOPTIME = 0 ; off

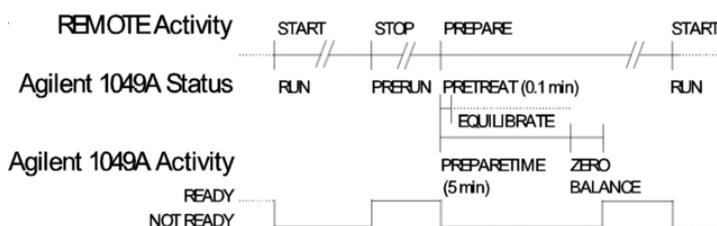
■POSTTIME = 0 ; off

```

We assume that the equilibration time for the cell after the potential pulses is less than or equal to 5 min (**PREPARETIME**).

Figure 36

### Detector status in Example 2(a)



### Example 2(b)

In this example you use an autosampler to send a prepare signal to the detector's **REMOTE** connector. You will use the **DRIFTTRIGGER** function to make sure the cell has equilibrated after pretreatment with the potential pulses.

Set detector parameters as follows:

```

■MODE = PRETREAT

■PRETREAT CONTROL = STOP

■PREPARETIME =      0 ; off

■ZERO CONTROL = PREPARE

■DRIFTTRIGGER =    1.0 ; nA/min

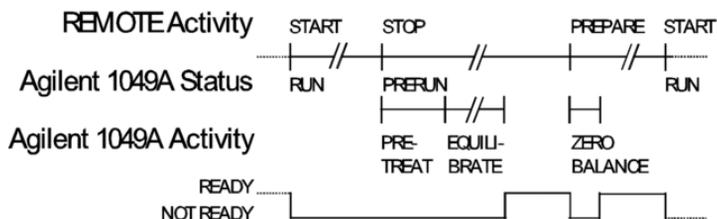
■STOPTIME =         0 ; off

■POSTTIME =         0 ; off

```

Figure 37

#### Detector status in Example 2(b)



#### Example 2(c)

In this example you use an autosampler to send start and stop signals to the detector's REMOTE connector. You will use the **DRIFTTRIGGER** function to make sure the cell has equilibrated after pretreatment with the potential pulses.

Set detector parameters as follows:

```

■MODE = PRETREAT

■PRETREAT CONTROL = STOP

■ZERO CONTROL = DRIFTTRIGGER

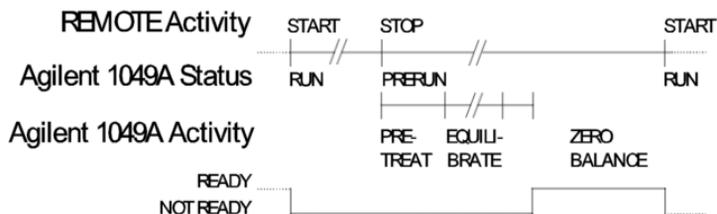
```

```

■DRIFTTRIGGER = 1.0 ; nA/min
■STOPTIME = 0 ; off
■POSTTIME = 0 ; off

```

Figure 38

**Detector status in Example 2(c)****Example 2(d)**

In this example you use an autosampler to send a start signal to the detector's **REMOTE** connector. You will use the **STOPTIME** function to stop the analysis and the **DRIFTTRIGGER** function to make sure the cell has equilibrated after pretreatment with the potential pulses.

Set detector parameters as follows:

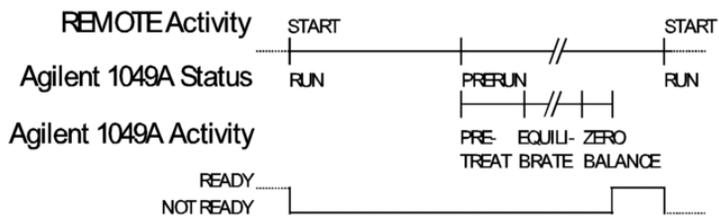
```

■MODE = PRETREAT
■PRETREAT CONTROL = STOP
■PREPARETIME = 0 ; off
■ZERO CONTROL = DRIFTTRIGGER
■DRIFTTRIGGER = 1.0 ; nA/min
■STOPTIME = 20.0 ; min
■POSTTIME = 0 ; off

```

Figure 39

Detector status in Example 2(d)



## Developing a Method Using INCREMENT

*Problem:* You have a new application and you want to determine the optimum detection potential for the analysis. You think that the optimum potential is between 0.4 and 0.7 V. You need to know what the value is to within 20 mV (you will do further experiments later to find the exact value). To be able to compare chromatograms you want the detector to do a balance before each analysis and as additional verification you want the analysis at each potential to be repeated twice.

### Example 3(a)

In this example you use an autosampler to send start and stop signals to the detector's REMOTE connector. You assume that the cell needs a maximum of 8 min to equilibrate after a change in potential.

Set detector parameters as follows:

```
■MODE = AMPEROMETRY
```

```
■INCREMENT = 0.020 ; Volt
```

```
■REPETITIONS = 2
```

```
■ZERO CONTROL = STOP
```

```
■POTENTIAL = 0.400 ; Volt
```

```
■STOPTIME = 0 ; off
```

```
■POSTTIME = 8.0 ; min
```

Set your injector to inject the sample according to:

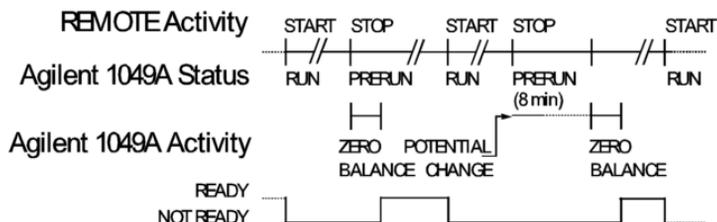
$$\text{No. of injections} = \text{Repetitions} \left( \frac{(P_{\text{end}} - P_{\text{start}})}{P_{\text{increment}}} + 1 \right)$$

In our example:

$$\text{No. of injections} = 2 \left( \frac{(0.7 - 0.4)}{0.02} + 1 \right) = 32$$

Figure 40

### Detector status in Example 3(a)



### Example 3(b)

In this example you use an autosampler to send start and stop signals to the detector's REMOTE connector. You will use the **DRIFTTRIGGER** function to make sure the cell has equilibrated after each change in potential.

Set detector parameters as follows:

■ **MODE = AMPEROMETRY**

■ **INCREMENT = 0.020 ; Volt**

■ **REPETITIONS = 2**

■ **ZERO CONTROL = DRIFTTRIGGER**

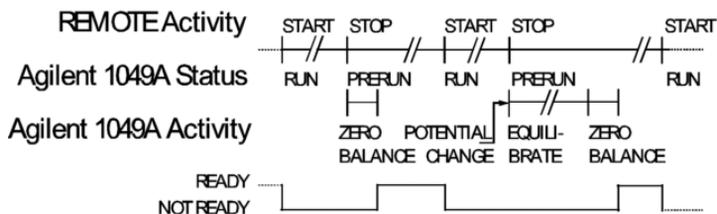
■ **POTENTIAL = 0.400 ; Volt**

■ **STOPTIME = 0 ; off**

■ **POSTTIME = 0 ; off**

Figure 41

**Detector status in Example 3(b)**



**Example 3(c)**

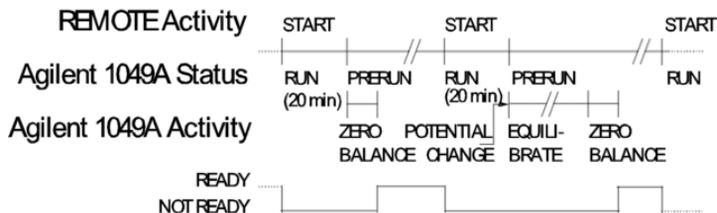
In this example you use an autosampler to send a start signal to the detector's **REMOTE** connector. You will use the **STOPTIME** function to stop the analysis and the **DRIFTTRIGGER** function to make sure the cell has equilibrated after each change in potential.

Set detector parameters as follows:

- **MODE = AMPEROMETRY**
- **INCREMENT = 0.020 ; Volt**
- **REPETITIONS = 2**
- **ZERO CONTROL = DRIFTTRIGGER**
- **POTENTIAL = 0.400 ; Volt**
- **STOPTIME = 20.0 ; min**
- **POSTTIME = 0 ; off**

Figure 42

**Detector status in Example 3(c)**



---

## Developing a Method for Differential Mode Using INCREMENT

*Problem:* You have an application that can be solved using the detector's differential mode and you want to determine the optimum detection potentials for the analysis. You assume that a potential difference of 0.05 V is adequate for the differential mode. You think that the optimum potential region is between 0.3 and 0.7 V. To get as near to the optimum value as possible during method development you use an increment of 0.025 V and repeat each analysis 3 times.

Set detector parameters as follows:

```
■MODE = DIFFERENTIAL
```

```
■DIFF POT1 = 0.300 ; Volt
```

```
■DIFF POT2 = 0.350 ; Volt
```

```
■INCREMENT = 0.025 ; Volt
```

```
■REPETITIONS = 3
```

**Developing a Method for Differential Mode Using INCREMENT**

With these parameter settings the following potential values for each analysis are used:

**Table 11****Potential Values Using Increment Function**

<b>Analysis</b>	<b>DIFF POT1</b>	<b>DIFF POT2</b>
1-3	0.300	0.350
4-6	0.325	0.375
7-9	0.350	0.400
10-12	0.375	0.425
...	...	...
49-51	0.700	0.750

The potential difference of 0.05 V between **DIFF POT1** and **DIFF POT2** is the same for each analysis.

As in Example 3 you must set your autosampler to do the correct number of injections.

$$\text{No. of injections} = 3 \left( \frac{(0.7 - 0.3)}{0.025} + 1 \right) = 51$$

In addition you can set an automatic **ZERO BALANCE** using **PREPARE**, **STOP** and **POSTTIME** or using the **DRIFTTRIGGER** function (as in Examples 1 through 3).

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**Troubleshooting Your  
Detector**

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# Troubleshooting Your Detector

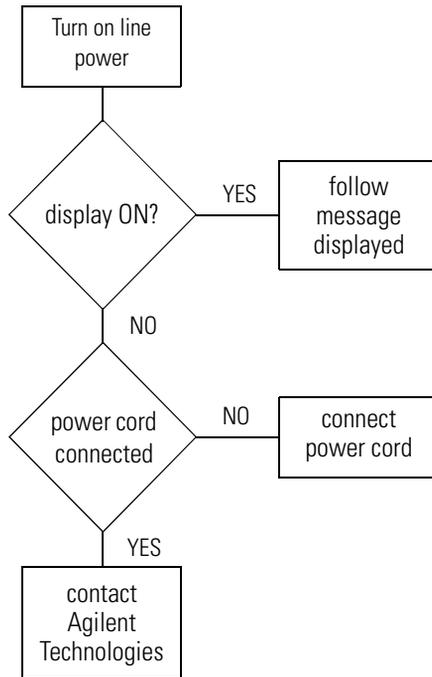
In this chapter we have organized the error conditions and malfunctions together, starting from the point where the condition can occur. This organization gets you to the source of a problem fast, without having to read through long lists of error messages.

Whenever you must make a repair or replace a part, we will reference the appropriate procedure in Chapter 7 “Maintaining Your Detector”.

## Troubleshooting During Start-up

When you turn on line power, the following errors can occur:

### Detector Down



## Troubleshooting During Operation

The messages described in this section can occur at any time during operation.

- The *Run* lamp on the front panel will be on during a run and will flash during **POSTTIME** or when data is being transferred through the GPIB interface (wait status of detector).
- The *Not ready* or *Error* lamp (or both) on the front panel will be on, depending on the message.
- You will see a short form of the message in the monitor display (press **Monitor**). **CAUTION** message will flash on and off.
- You will see **error**, **notready** or **caution** in the status display.
- You will see a list of the full messages by pressing **Status** and then **Next** ▼.

If a not ready condition occurs, the detector makes a connection between pins 1 and 7 of the REMOTE connector. The detector makes this connection for as long as the not ready condition exists. When the not ready condition is removed the connection is broken and the detector operates again.

A not ready condition can pause automated operation which will only continue when the not ready condition is removed.

If an error condition occurs, the detector makes a connection between pins 1 and 4 of the REMOTE connector. The detector makes this connection for as long as the error condition exists. When the error condition is removed, the connection is broken and the detector operates again. An error condition caused by a leak must be reset by **RESET LEAKSENSOR** to prevent you from overlooking the leak.

If an error still remains after you have checked or replaced the parts described, contact Agilent Technologies.

<b>leak</b>	<b>ERROR: leak, reset sensor</b>
Leak in detector.	Check for leaks. When you have corrected the fault, reset the leak sensor, see <b>"RESET FUNCTIONS"</b> on page 94.
Detector turns-OFF cell.	

Troubleshooting Your Detector  
**Troubleshooting During Operation**

<b>leak</b>	<b>ERROR: leak sensor failed</b>	Leak sensor failed. Check connection between J21 on ECP board (item 4 in <b>Figure 44</b> on page 129) and J95 on ECC board. If error persists, replace ECP board, see “Replacing the ECP Board” on page 131.
<b>temp</b>	<b>ERROR: out of temperature range</b>	Solvent thermostat failed. Check thermostat cable. Replace solvent thermostat, see “Replacing the Solvent Thermostat” on page 132.
<b>gpib</b>	<b>ERROR: detector overflow</b>	Detector's memory buffer is full with data. Read data from buffer through GPIB interface.
<b>test</b>	<b>CAUTION: test is running</b>	One of the detector tests is running. Test will stop when <b>MODE</b> changed.
<b>cell</b>	<b>CAUTION: cell is off</b>	Cell is OFF. Turn-ON cell if required..
<b>cell</b>	<b>CAUTION: cell not connected</b>	Cell is not connected. Connect cell, see “Connecting the Electrodes” on page 41.
<b>incr</b>	<b>CAUTION: increment is on</b>	Increment function is on. See “ <b>INCREMENT</b> ” on page 80.
<b>dyn</b>	<b>CAUTION: reduced dynamic range</b>	Scaling problem because <b>ZERO BALANCE</b> needs higher <b>ZERO CURRENT</b> setting than is possible. Balance detector, press <input type="button" value="Zero"/> <input type="button" value="Enter"/> . Find out why background current is too high. Change <b>INSTRUMENT FULLSCALE</b> .
<b>und</b>	<b>CAUTION: analog out underflow</b>	

**Troubleshooting During Operation**

	Output from INTEGRATOR too low (<0 V).	Balance detector, press <input type="button" value="Zero"/> <input type="button" value="Enter"/> .
<b>over</b>	<b>CAUTION: analog out overflow</b>	
	Output from INTEGRATOR too high (>1 V).	Balance detector, press <input type="button" value="Zero"/> <input type="button" value="Enter"/> .
<b>zero</b>	<b>NOTREADY: zero setting</b>	
	Detector is balancing.	Wait (about 5 s).
<b>full</b>	<b>NOTREADY: out of fullscale</b>	
	Preamplifier overflow.	Balance detector, press <input type="button" value="Zero"/> <input type="button" value="Enter"/> . Change <b>INSTRUMENT FULLSCALE</b> .
<b>temp</b>	<b>NOTREADY: temperature high</b>	
	Temperature in solvent thermostat is too high.	Wait until temperature stabilizes or change temperature setting, see <b>"THERMOSTAT PARAMETERS"</b> on page 93.
<b>temp</b>	<b>NOTREADY: temperature low</b>	
	Temperature in solvent thermostat is too low.	Wait until temperature stabilizes or change temperature setting, see <b>"THERMOSTAT PARAMETERS"</b> on page 93.
<b>pret</b>	<b>NOTREADY: pretreatment running</b>	
	Pretreatment mode is running.	See <b>"PRETREAT PARAMETERS"</b> on page 84.
<b>prep</b>	<b>NOTREADY: preparetime running</b>	
	Prepare time is running.	See <b>"PREPARETIME"</b> on page 82.
<b>drif</b>	<b>NOTREADY: drifttrigger is on</b>	
	Drift trigger function is enabled.	See <b>"DRIFTTRIGGER"</b> on page 81.

## **Troubleshooting the Chromatogram**

The quality of your chromatographic results depends on the performance of all modules in the chromatographic system (pump, injector, column, detector, etc.). If all the modules in your system perform according to the manufacturer's specification, an application problem might be preventing good chromatographic results. Use the following guide to help identify the source of the fault.

## **Retention times not reproducible**

Unstable flow.	Check flow system to improve flow stability.
Gas bubbles in pump.	Shake all bubbles from solvent inlet filters, degas thoroughly and prime pump. Priming pump with propanol will help to remove trapped air.
Leaks.	Disconnect detector and check pressure tightness of other modules.
Unstable column temperature.	Check stability of column temperature.
Impurities on column.	Flush column with solvent of high elution strength.
Column not conditioned:	
1. Conditioning time too short.	Condition column (for ion-pair chromatography, conditioning times can be long).
2. Water content not constant (in adsorption chromatography on silica).	Add defined amount of water to solvent or change to reversed phase chromatography.
3. New column loses polymeric material (chemically modified silica).	Flush column with solvent of high elution strength.
4. Chemical reaction between sample and stationary phase.	Use another type of column.

## **Areas not reproducible**

Unstable flow.	Check flow system to improve flow stability.
Gas bubbles in pump.	Shake all bubbles from solvent inlet filters, degas thoroughly and prime pump.
Leaks.	Disconnect detector and check pressure tightness of other modules.
Injection not reproducible.	Check performance of your injector.
Impure solvents cause baseline drift.	Use pure solvents. Make sure conditioning times before analysis are equal.

## **Baseline noise**

Flow rate too high	Reduce flow. Typical flow rates for thin-layer cells are 0.5 through 1.0 ml/min.
Unstable flow.	Check flow system to improve flow stability.
Non-miscible solvents.	Use suitable solvents.
Responsetime or peakwidth too low.	Set higher value according to chromatographic needs.

## **Baseline drift and wander**

Unstable column temperature.	Check stability of column temperature. Thermally insulate detector inlet capillary.
Gas bubbles in pump.	Shake all bubbles from solvent inlet filters, degas thoroughly and prime pump. Priming pump with propanol will help to remove trapped air.
Gas bubbles in cell.	Make sure that cell outlet points upwards.
Solvents not mixed completely.	Use suitable solvents or add a mechanical mixer.
Stains on working electrode.	Compare background current sweep of electrode with a freshly polished electrode and a new electrode. Check solvents for impurities. Avoid using manganese-containing substances, because of adsorption on electrode surfaces. Avoid using high potentials, check the upper and lower potential limits.  Polish the working electrode surface or replace the electrode.
Particulate matter and fingerprints on or between spacer and working electrode.	Clean working electrode, spacer and auxiliary electrode with a soft tissue soaked in methanol or acetone. Dry all parts of cell before re-assembly, keeping all parts free from dust.
Reference electrode (with internal electrolyte) drifting.	Check stability of electrode with reference test mode, see "Reference Electrode Test" on page 49. Refill electrolyte chamber with fresh potassium chloride (KCl) solution.

**Troubleshooting the Chromatogram**

Solid state reference electrode drifting.	Check chloride ion (Cl <sup>-</sup> ) concentration in mobile phase (should be about 1 to 2 mM). Use lithium chloride (LiCl) for organic buffer solutions. Check stability of electrode with reference test mode, see "Reference Electrode Test" on page 49.
High background current.	Check purity of water and of eluent compounds. Passivate your system, see Chapter 1 "Preparing Your Liquid Chromatograph". Check surface of working electrode. Check for proper eluent composition.
Bad thermal shielding.	Thermally shield detector inlet capillaries. Use solvent thermostat, see Chapter 2 "Installing Your Detector".
Poor grounding.	Connect all power lines of your system to the same line power socket. Avoid using long steel capillaries, which can act as antennas. Keep detector away from sources of radio interference.
High or unstable pressure drops in LC system.	Check all solvent inlet sieves, frits (pump, column, etc.) and valves.
Non-conditioned or new column.	Flush column with solvent of high elution strength.
Sample impurities that elute slowly.	Flush column with solvent of high elution strength.
Leaks.	Disconnect detector and check pressure tightness of other modules.

## **Tailing peaks**

Poor column	Check column performance. Check top of column for voids, repack if necessary.
Non-thermodynamic interaction between stationary phase and sample.	Use modifiers or change stationary phase.
Highly contaminated or passivated working electrode.	Check working electrode and polish or replace.
Dead volumes caused by poor connection of cell inlet capillary.	Disconnect the inlet capillary and check the fittings--the end of the capillary should be cut square (not slanted) and should protrude 2 mm out of the gripper.

## **Heading peaks**

Column overloaded.	Reduce sample concentration or injection volume.
--------------------	--

## **Double peaks of one substance**

Poor column.	Check column performance.
High sample volume injected in solvent with higher elution strength than mobile phase.	Use solvent for sample with lower elution strength than mobile phase.

## **Excessive baseline disturbance after injection**

High pressure drop after injection.	Check performance of your injector.
-------------------------------------	-------------------------------------

Troubleshooting Your Detector  
**Troubleshooting the Chromatogram**

---

## **Maintaining Your Detector**

---

# Maintaining Your Detector

In this chapter we describe the maintenance tasks you can do on your detector. Do only the maintenance specified in this handbook. Other maintenance or repairs must be done by Agilent Technologies trained personnel. Unauthorized maintenance can be dangerous. Damage caused by unauthorized maintenance is not covered by warranty.

---

**WARNING**

---

**High Voltages: Disconnect the line-power cord before removing any of the Agilent 1049A panels.**

---

**WARNUNG**

---

**Hochspannung: Bevor ein Gehäuseteil entfernt werden soll, muß das Netzkabel abgezogen werden.**

---

**CUIDADO**

---

**Alta tensión: Desconecte el instrumento de la línea antes de sacar el panel posterior del instrumento.**

---

**ATTENTION**

---

**Tensions élevées: Débranchez le cordon secteur avant d'ôter le panneau arrière.**

---

**ATTENZIONE**

---

**Alte tensioni: Scollegare il cavo di alimentazione prima di rimuovere il pannello posteriore.**

## Maintaining the Cell

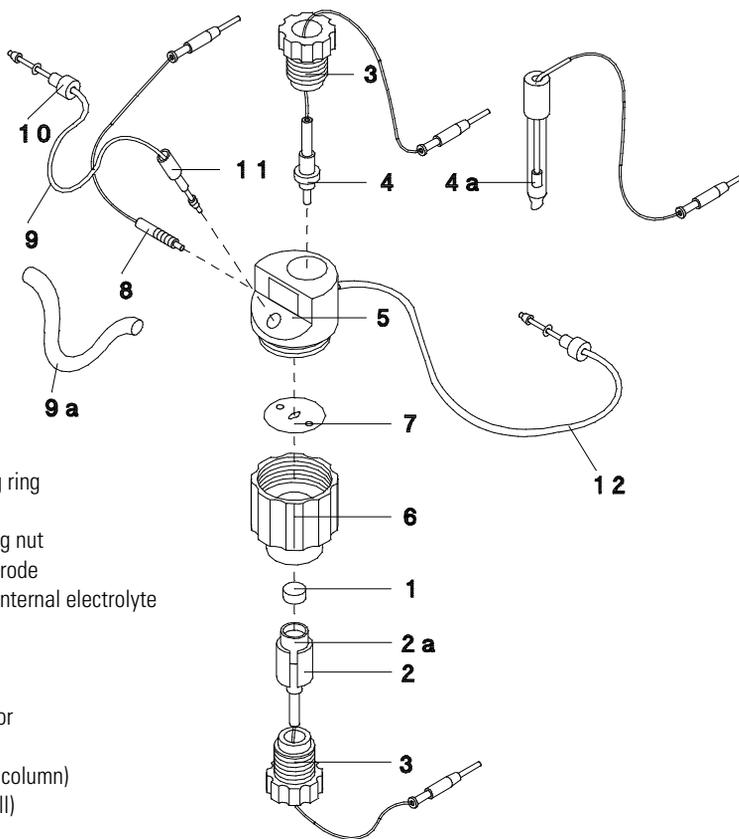
Before you do any maintenance on the cell, read the following on cell handling.

- Always unscrew and remove the working electrode assembly before unscrewing the two halves of the cell body.
- Always screw the two halves of the cell body together first, before fitting the working electrode assembly.
- Always make sure that the surfaces of the spacer and working electrode are dry and free from particulate matter before re-assembling the cell. Clean fingerprints from spacer and electrode surfaces with acetone or methanol.
- If the auxiliary electrode needs to be cleaned, wipe the surfaces carefully with a soft tissue soaked in acetone or methanol. Do not apply force—you may damage the electrode surface.
- Make sure that the working electrode has a mirror-like appearance before re-assembling the cell.
- If the detector has been stood idle for more than 1 day, we recommend that you disassemble the cell and clean all surfaces (remember to turn-OFF the cell).
- When you re-install the cell in the detector, make sure that you connect the inlet and outlet correctly (*red to red, blue to blue*).

There are several maintenance tasks you can do on the electrochemical cell. Figure 43 will help you identify the parts of the cell.

**Figure 43**

**Exploded Diagram of Electrochemical Cell**



1. Working electrode
2. Working electrode retaining ring
- 2a. Working electrode holder
3. Electrode assembly retaining nut
- 4a. Solid state reference electrode
- 4b. Reference electrode with internal electrolyte
5. Cell body (upper part)
6. Cell body (lower part)
7. Electrode spacer
8. Auxiliary electrode connector
9. Inlet capillary
10. Inlet capillary fitting (from column)
11. Inlet capillary fitting (to cell)
12. Outlet capillary

## Replacing a Working Electrode

The following working electrodes (item 1 in Figure 43) are available:

Part Number	Description
01049-64105	Glassy carbon working electrode
01049-28801	Platinum working electrode
01049-28802	Gold working electrode

To replace a working electrode:

## Maintaining the Cell

- 1 Unscrew nut (item 3 at bottom of Figure 43 on page 126 and remove electrode assembly.
- 2 Hold shaft of electrode assembly and pull down retaining ring (item 2 in Figure 43 on page 126).
- 3 Using tip of flat-bladed screwdriver, prise off old working electrode (item 1 in Figure 43 on page 126).
- 4 Insert new working electrode and push up retaining ring.
- 5 Insert electrode assembly into cell body and tighten nut.

### Replacing a Reference Electrode

The following reference electrodes are available:

Part Number	Description
01049-62901	Solid state reference electrode
01049-62902	Reference electrode with internal electrolyte
01049-92101	KCl solution for above electrode

To replace a reference electrode:

- 1 Unscrew nut (item 3 at top of Figure 43 on page 126) and remove reference electrode.
- 2 For reference electrode with internal electrolyte, check whether electrode is filled with electrolyte.
- 3 Insert new reference electrode in cell body and tighten nut.

### Replacing the Cell Spacer

The cell spacer (item 7 in Figure 43 on page 126) separates surface of working electrode from auxiliary electrode – the black-colored upper part of cell body (item 5 in Figure 43 on page 126). 3 spacers (part number 01049-24705) are supplied in the accessory kit.

- 1 To replace a cell spacer:
- 2 Remove working electrode assembly.
- 3 Unscrew lower part of cell body (item 6 in Figure 43 on page 126) from black auxiliary electrode (item 5 in Figure 43 on page 126).

## Maintaining the Cell

- 4 Remove old spacer.
- 5 Clean and dry the working and auxiliary electrodes.
- 6 Insert new spacer.
- 7 Reassemble two parts of cell body and tighten.
- 8 Insert working electrode assembly and tighten nut.

### Polishing Working Electrodes

A polishing kit is available (part number 01049-67001) which contains all the materials you need to polish working electrodes.

To polish a working electrode:

- 1 Remove working electrode from electrode assembly (as described above).
- 2 Place polishing tissue on bench.
- 3 Place 1 drop of polishing slurry on tissue.
- 4 Place working electrode face-down in slurry and polish by rubbing electrode circularly in slurry (rub carefully--do not use excessive force).
- 5 Clean slurry from electrode with acetone or methanol. When properly polished the electrode should give a mirror-like appearance.

---

#### NOTE

Use only the supplied polishing tissue to polish electrodes. Do not apply force when wiping any layers or contaminants from electrode surfaces. Always try to clean electrodes chemically before using mechanical polishing. Glassy carbon is resistant against most aggressive chemicals used in laboratories.

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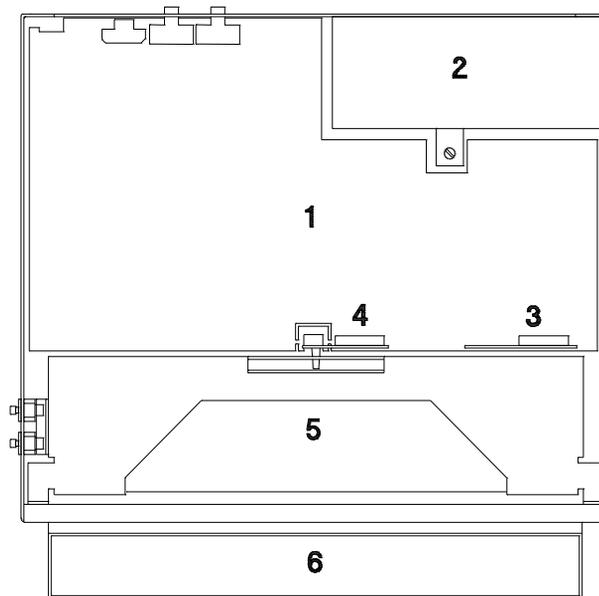
## Replacing Printed Circuit Boards

When identified as being defective, you can replace the FTC and ECP boards. Figure 44 is a top view of the detector, showing you the positions of the FTC board (item 3), the ECP board (item 4) and the ECC board (item 1).

**Figure 44**

1. ECC board
2. Power supply
3. FTC board
4. ECP board
5. Cell compartment
6. Keyboard

**Position of FTC, ECP and ECC Boards**



Before you replace a board, observe the following caution.

---

### **CAUTION**

The printed circuit boards contain many electronic parts that are sensitive to electronic discharge. Do not attempt to remove the boards, unless you use a portable grounding kit. **DO NOT TOUCH** any of the components on the boards. **DO NOT PLACE** printed circuit boards on any plastic or plastic-coated surface.

---

## Replacing Printed Circuit Boards

---

### Achtung

Die elektronischen Leiterplatten enthalten viele elektronische Bauteile, die sehr empfindlich gegenüber elektrostatischer Entladung sind. Entfernen Sie keine Leiterplatten ohne Verwendung einer antistatischen Ausrüstung (Portable Grounding Kit). Berühren Sie keine Bauteile auf den Leiterplatten. Legen Sie keine Leiterplatten auf Oberflächen mit Kunststoffbeschichtung.

---

---

### Precaución

Les circuitos impresos contienen muchas partes sensibles a las descargas electricas. No intente sacar los circuitos si no dispone del kit portátil de tierra. NO TOQUE ninguno de los componentes de las placas. NO COLOQUE ningun circuito impreso sobre material o superficies de plástico.

---

---

### Avertissement

Les circuits imprimés possèdent des éléments sensibles aux décharges électrostatiques. N'essayez pas de sortir les cartes sans utiliser l'accessoire portable de mise à la terre. NE TOUCHEZ PAS aucun des composants des cartes. NE POSEZ PAS les circuits imprimés sur des matières plastiques ou des surfaces recouverts de matières plastiques.

---

---

### Precauzione

Le schede a circuito stampato contengono molte parti elettriche. Non tentate di rimuovere le schede non siete dotati di un kit per la messa a terra dell'operatore. NON TOCCATE nessun componente sulle schede. NON APPOGGIATE le schede su superfici plastiche o plastificate.

---

## Replacing the FTC Board

You can order a new FTC board by quoting part number 01049-66503.

- 1 Disconnect the line power cord.
- 2 Remove front panel and top cover.
- 3 Disconnect electrodes from connectors on mounting panel.
- 4 Remove mounting panel.
- 5 Disconnect cable at connector J1 on FTC board.
- 6 Remove two screws that secure FTC board to inner panel.
- 7 Remove FTC board from ECC board.
- 8 Remove new FTC board from antistatic bag.
- 9 Insert connector of FTC board into connector J98 on ECC board.

## Replacing Printed Circuit Boards

- 10** Secure FTC board to inner panel with the two screws.
- 11** Connect cable from thermostat to connector J1 on FTC board.
- 12** Replace mounting panel.
- 13** Connect electrodes.
- 14** Replace front panel and top cover.

### Replacing the ECP Board

You can order a new ECP board by quoting part number 01049-66502.

- 1** Disconnect the line power cord.
- 2** Remove front panel and top cover.
- 3** Disconnect electrodes from connectors on mounting panel.
- 4** Remove mounting panel.
- 5** Remove screws that secure ECP board to inner panel.
- 6** Remove ECP board from ECC board.
- 7** Remove new ECP board from antistatic bag.
- 8** Insert connector of ECP board into connector J95 on ECC board.
- 9** Secure ECP board to inner panel with the two screws.
- 10** Replace mounting panel.
- 11** Connect electrodes.
- 12** Replace front panel and top cover.

## Replacing the Solvent Thermostat

You can order a new solvent thermostat by quoting the part number 01049-66901.

- 1** Disconnect the line power cord.
- 2** Remove front panel and top cover.
- 3** Disconnect capillaries from thermostat.
- 4** Disconnect cable at connector J1 on FTC board.
- 5** Lead cable back through holes in inner panel.
- 6** Remove thermostat from mounting.
- 7** Insert new thermostat into mounting.
- 8** Lead cable from thermostat through holes in inner panel.
- 9** Connect cable from thermostat to connector J1 on FTC board
- 10** Connect capillaries to thermostat.
- 11** Replace front panel and top cover.

---

## **Installing Accessories**

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# Installing Accessories

This appendix describes how to install accessories that are available for your detector. If you are replacing a defective part, see Chapter 7 “Maintaining Your Detector” for details of replacement procedures.

## **Installing Electrodes**

For details of how to install the different electrodes in the electrochemical cell, see Chapter 2 “Installing Your Detector”.

---

## Installing the Solvent Thermostat

The solvent thermostat is available as a separate accessory (quote order number 79845A) which you can install yourself into your detector.

### What You Will Need

Check the contents of the kit against the packing list below.

**Table 12**

---

#### Checklist of Accessories

---

Description	Part Number	Check
Solvent thermostat	01049-66901	...
FTC board	01049-66503	...
2 Screws (M3 × 3 mm)	0515-0886	...
Label	7121-0850	...

---

### Removing the Panels

Before you install the solvent thermostat you must:

- Remove the front panel.
- Remove the top cover.
- Remove the mounting panel.

---

**WARNING**

---

**High Voltages: Disconnect the line-power cord before removing any of the Agilent 1049A panels.**

---

**WARNUNG**

---

**Hochspannung: Bevor ein Gehäuseteil entfernt werden soll, muß das Netzkabel abgezogen werden.**

---

**CUIDADO**

---

**Alta tensión: Desconecte el instrumento de la línea antes de sacar el panel posterior del instrumento.**

---

---

**ATTENTION**

---

**Tensions élevées: Débranchez le cordon secteur avant d'ôter le panneau arrière.**

---

**ATTENZIONE**

---

**Alte tensioni: Scollegare il cavo di alimentazione prima di rimuovere il pannello posteriore.**

**Removing the Front Panel**

- 1** Loosen the 2 screws in the bottom-left and bottom-right corners of the front panel.
- 2** Pull the bottom edge of the front panel towards you and then pull it down away from the detector.
- 3** Using the 2 clips on the inside of the front panel, hang the front panel on the front edge of the detector's bottom panel.

**Removing the Top Cover**

- 1** Remove the 2 screws in the top-left and top-right corners of the rear panel.
- 2** Remove the 2 screws at the top-left and top-right sides of the detector.
- 3** Lift off the top cover.

**Removing the Mounting Panel**

- 1** Disconnect the electrodes from the connectors on the mounting panel.
- 2** Remove the cell from the cell clamp.
- 3** Loosen the 2 thumb screws in the bottom-left and bottom-right corners of the mounting panel.
- 4** Pull the bottom edge of the mounting panel towards you and then pull it down and away from the detector.

**Installing the FTC Board**

Before you install the FTC board, observe the following caution.

**Installing the Solvent Thermostat**

---

**CAUTION**

The printed circuit boards contain many electronic parts that are sensitive to electronic discharge. Do not attempt to remove the boards, unless you use a portable grounding kit. DO NOT TOUCH any of the components on the boards. DO NOT PLACE printed circuit boards on any plastic or plastic-coated surface.

---

---

**Achtung**

Die elektronischen Leiterplatten enthalten viele elektronische Bauteile, die sehr empfindlich gegenüber elektrostatischer Entladung sind. Entfernen Sie keine Leiterplatten ohne Verwendung einer antistatischen Ausrüstung (Portable Grounding Kit). Berühren Sie keine Bauteile auf den Leiterplatten. Legen Sie keine Leiterplatten auf Oberflächen mit Kunststoffbeschichtung.

---

---

**Avertissement**

Les circuits imprimés possèdent des éléments sensibles aux décharges électrostatiques. N'essayez pas de sortir les cartes sans utiliser l'accessoire portable de mise à la terre. NE TOUCHEZ PAS aucun des composants des cartes. NE POSEZ PAS les circuits imprimés sur des matières plastiques ou des surfaces recouverts de matières plastiques.

---

---

**Precauzione**

Le schede a circuito stampato contengono molte parti elettriche. Non tentate di rimuovere le schede non siete dotati di un kit per la messa a terra dell'operatore. NON TOCCATE nessun componente sulle schede. NON APPOGGIATE le schede su superfici plastiche o plastificate.

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---

**Precaución**

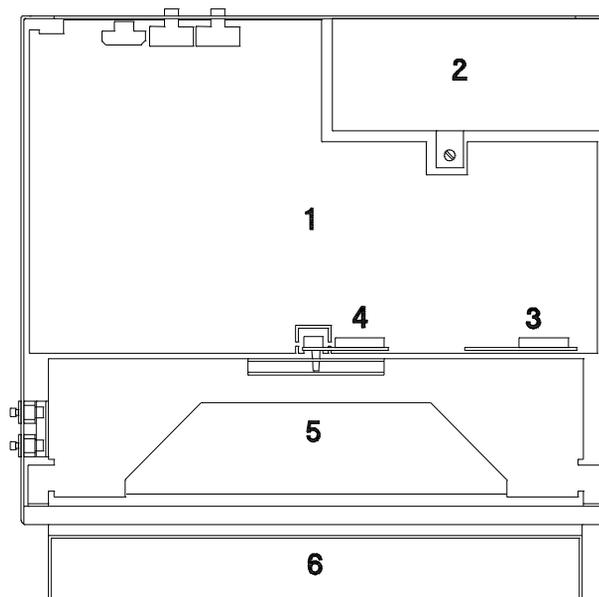
Les circuitos impresos contienen muchas partes sensibles a las descargas electricas. No intente sacar los circuitos si no dispone del kit portátil de tierra. NO TOQUE ninguno de los componentes de las placas. NO COLOQUE ningun circuito impreso sobre material o superficies de plástico.

---

Figure 45 is a top view of the detector, showing you where you must install the FTC board.

**Figure 45**

**Position of FTC Board (item 3) on ECC Board (item 1)**



- 1** Remove FTC board from antistatic bag.
- 2** Insert connector of FTC board into connector J98 on ECC board.
- 3** Secure FTC board to inner panel with the two screws.
- 4** Place solvent thermostat in front compartment of detector.
- 5** Lead cable from thermostat through holes in inner panel.
- 6** Connect cable from thermostat to connector on FTC board

### **Replacing the Cell**

- 1** Change the position of the cell clamp, according to the type of reference electrode you have and the position of the inlet and outlet connections, see Chapter 2 “Installing Your Detector”.
- 2** Replace the mounting panel in the detector and tighten the 2 thumb screws.
- 3** Connect the male fitting on the thermostat to the IN fitting on the inside of the side panel.
- 4** Insert thermostat into its clamp.

## Installing the Solvent Thermostat

- 5 Insert cell into cell clamp.
- 6 Connect the inlet tube (colored *red*) between the cell and the ZDV-fitting on the thermostat.
- 7 Connect the cell outlet tube (colored *blue*) between the cell and the OUT fitting on the inside of the side panel.
- 8 Connect the electrodes.

### Replacing the Panels

- 1 Put the top cover back into place.
- 2 Secure the side panels and top cover with the screws on each side and the screws on the rear panel.
- 3 Replace the front panel, securing it with the screws.

### Checking the Thermostat

- 1 Turn-ON line power to detector.
- 2 Press  , then press  several times to display **THERMOSTAT PARAMETERS** and press .

If you have installed the solvent thermostat correctly, the detector will display:

```
!THERMOSTAT = ON
```

If the detector displays:

```
!THERMOSTAT ;not installed
```

Check that the FTC board is correctly fitted to the ECC board and check that the cable from the thermostat is correctly fitted to the FTC board.

---

**Using the GPIB  
Interface**

---

# Using the GPIB Interface

The letters GPIB stand for General Purpose Interface Bus. It is Agilent Technologies' implementation of the IEEE-488-1978 interface standard (*Institute of Electrical and Electronics Engineers 488-1978 standard for a general-purpose interfacing bus, commonly termed the GPIB.*). The purpose of the GPIB is to provide for mechanical, electrical, timing, and data compatibilities between all devices adhering to the standard (*You should be aware that some devices claiming "IEEE-488 compatible" really are not fully compatible.*). In essence, interfacing computers to other devices has been simplified by the GPIB. Instead of worrying about *how* to hook up your devices so that they can communicate, you merely have to consider *what* they are going to communicate.

---

## **GPIB Address**

All instruments that use GPIB communication can be accessed using an GPIB address.

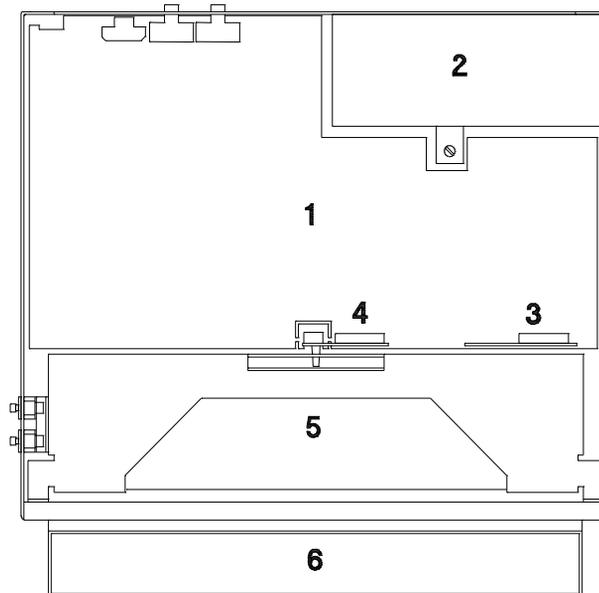
*The GPIB address of your Agilent 1049A is preset at the factory to 11.*

You can change the GPIB address by changing the settings of the DIP switch (S82 on ECC board). To change the GPIB address:

- 1** Turn-OFF line power to the detector and disconnect power cord.
- 2** Remove the 2 screws at each side of the rear panel.
- 3** Remove the 2 screws at both the left and right side of the detector.
- 4** Lift off the top cover.
- 5** Find the DIP switch S82—it is located on the ECC board (item 8 in Figure 46) near the front left of the detector.

**Figure 46**

### **Location of DIP Switch**



### **GPIB Address**

- 6** Set the switches to the GPIB address of your choice. The switch numbers 1 through 8 have values of 1, 2, 4, 8, 16, 32, 64 and 128. To set an address of 11, open switch numbers 1 (value 1), 2 (value 2) and 4 (value 8). A switch is open when the rocker is down on the side marked *open*.
- 7** Put the top cover back into place.
- 8** Secure the side panels and top cover with the 2 screws on each side and the 2 screws on the rear panel.
- 9** Connect power cord and turn-ON line power.

---

## Communication Units

The GPIB communication of the Agilent 1049A Electrochemical Detector is based on the GPIB transformer firmware that is already used in the HP 1040 Diode Array Detector and HP 1090 Liquid Chromatographs. Its main features are the different Communication Units (called CU in the following text) to provide immediate access to different datapaths. The instrument accepts messages via In-CUs and provides messages via Out-CUs. The individual CUs can be selected with the GPIB Secondary Command.

Here some examples to give you an idea how to talk to the instrument via GPIB .

### Read OutCU-0

**controller:** UNT, UNL, TALK <nn>, SCG 0

**instrument:** DATA,..... last DATA-byte with EOI

The OutCU-0 always sends 7 bytes which describe the state of the user-CUs .

<b>1st byte</b>	SUMMARY
<b>2nd byte</b>	REPLY (OutCU-1)
<b>3rd byte</b>	CHROM (OutCU-2)
<b>4th byte</b>	not used (OutCU-3)
<b>5th byte</b>	RAWDATA (OutCU-4)
<b>6th byte</b>	EVENTS (OutCU-5)
<b>7th byte</b>	INSTR (InCU-1)

The individual bits of each byte have following meaning:

- bit 0 set to 1 : input not ready
- bit 1 set to 1 : output ready
- bit 2 set to 1 : transfer started
- bit 3 set to 1 : error
- bit 4 set to 1 : not used
- bit 5 set to 1 : not used

## Communication Units

bit 6 set to 1 : service request

bit 7 set to 1 : not used

---

### NOTE

The SUMMARY-byte contains the bitwise logical OR of all following bytes except the bit 0 (1 = input not ready) of the OutCUs. With OutCUs the bit 0 (input-notready) is always set to 1 .

---

### Write Instruction Text to InCU-1 (Instruction-CU)

**controller:** UNT, UNL, LISTEN 11, SCG 1, ...<text>.. DAB/EOI  
(Databyte)

### Read reply

**controller:** UNT, UNL, TALK 11, SCG 1

**instrument:** ...<reply text>... DAB/EOI

### Read Chromatogram CU

**controller:** UNT, UNL, TALK 11, SCG 2

**instrument:** ...<reply text>... DAB/EOI

---

### NOTE

With the SCG-command you address the individual CUs. All messages are terminated with the EOI together with the last DATA byte.

---

**warmstart** A warmstart of the detector by SCG31 will send the message **EC02 crlf 1049A cleared** and initialize the

detector without loosing the current parameter settings.

GPIB	Status Mask	InCU-0
Trafo	CU-Status	OutCU-0
Instruction Processor	Instruction Reply	InCU-1 OutCU-1
Measurement	Chromatogram	OutCU-2
Result	not used	OutCU-3
Generator	Rawdata	OutCU-4
	Events	OutCU-5

### **Short Description of CUs**

**Mask-CU and Status-CU ( InCU-0, OutCU-0 ):** These CUs are for communication control only.

**Instruction-CU (InCU-1):** All messages sent to that CU are sent to the Instruction Processor.

**Reply-CU (OutCU-1):** All replies generated by a foregoing instruction can be read from that CU.

**Chromatogram-CU (OutCU-2):** Provides chromatographic signal for the online monitor plot.

**Not used (OutCU-3):**

**Rawdata-CU (OutCU-4):** Output for the runbuffered Rawdata from RUN.

**Event-CU (OutCU-5):** Events will be available here.

## Instruction and Reply Messages (InCU-1, OutCU-1)

### General Rules

*Instruction* and *reply* messages must follow these rules:

- 1 Both message types contain only printable ASCII characters.
- 2 The instruction-reply sequence is a closed loop datapath.
- 3 There is only one reply for one instruction.
- 4 The next instruction may not be sent until the reply of the previous instruction has been received.
- 5 Concerning the direction of information-flow there are two types of *instructions*:

Download from operator/computer to instrument, e.g.

parameter value assignment: POTENTIAL = 1.200

command instruction: STOP

Upload from instrument to operator/computer, e.g.

Request specific parameter line: POTENTIAL

Request status report: STATUS

- 6 The *reply* consists of an ASCII header and a text part.

Header:

C2 C1 C0 N2 N1 N0 crlf.

C2..0 capital letters to build a 3-level tree for categorizing messages.

N2..0 digit to allow enumeration of messages of the same category.

cr lf end of header

Text:

Printable ASCII characters terminated or separated with cr lf.

- 7 With an upload-instruction that requests more than one line of information there will exist two types of replies:

**Instruction and Reply Messages (InCU-1, OutCU-1)**

Fixed length, see instruction PARAMETER.

Variable length, see instruction STATUS.

**Reply Format**

Header format: C2 C1 C0 N2 N1 N0 cr lf.

reply categories								
C2	R indicates a reply message							
C1	A instruction accepted				E instruction error			
C0	L	T	D	C	L	T	D	C

- L** Reply contains one *line* of a simple parameter that shows also the value of this parameter.
- T** Reply contains one line out of a *table* structure.
- D** Reply contains more than one line (e.g. *different* status report lines).
- C** Reply contains only a text reply to a simple *command* like "STOP" (reply: "stopped") or a text reply if parameters are not properly set like "check potential limits".

N2 N1 N0 definition depends on the message category.

**Instruction error REX**

(X = L / T / D / C):

- 002** parameter out of range
- 003** another keyword expected
- 004** equal expected
- 005** parameter missing
- 006** invalid format
- 007** parameter overflow
- 008** keyword not identified
- 010** not allowed in RUN
- 018** not implemented

**Instruction and Reply Messages (InCU-1, OutCU-1)**

<b>040</b>	press STOP before START
<b>041</b>	read rawdata before START
<b>042</b>	press START before STOP
<b>043</b>	detector notready
<b>044</b>	postrun not finished
<b>045</b>	check potential limits

**Instruction Accepted RAL**

N2..0 contains internal code of parameter keyword:

<b>001</b>	REC RANGE
<b>002</b>	ZEROCURRENT
<b>003</b>	RESPONSETIME
<b>004</b>	ZEROLEVEL
<b>005</b>	POTENTIAL
<b>006</b>	INCREMENT
<b>007</b>	REPETITIONS
<b>008</b>	INSTRUMENT FULLSCALE
<b>009</b>	DRIFTTRIGGER
<b>010</b>	THERMOSTAT
<b>011</b>	TEMPERATURE
<b>012</b>	MODE
<b>013</b>	ZERO CONTROL
<b>014</b>	CELL
<b>015</b>	POLARITY
<b>016</b>	PEAKWIDTH
<b>017</b>	PRETREAT CYCLES
<b>018</b>	PRETREAT POT1
<b>019</b>	PRETRAEAT POT2
<b>020</b>	PRETREAT TIME1

**Instruction and Reply Messages (InCU-1, OutCU-1)**

<b>021</b>	PRETREAT TIME2
<b>022</b>	PRETREAT TIME3
<b>023</b>	PRETREAT CONTROL
<b>024</b>	SWEEP CYCLES
<b>025</b>	SWEEP POT1
<b>026</b>	SWEEP POT2
<b>027</b>	SWEEP RATE
<b>028</b>	PULSE POT1
<b>029</b>	PULSE POT2
<b>030</b>	PULSE TIME1
<b>031</b>	PULSE TIME2
<b>032</b>	PULSE TIME3
<b>033</b>	DIFF PERIOD
<b>034</b>	DIFF POT1
<b>035</b>	DIFF POT2
<b>036</b>	AUTO CELL
<b>037</b>	AFTER
<b>038</b>	UPPERLIMIT
<b>039</b>	LOWERLIMIT
<b>060</b>	MONITOR
<b>061</b>	DRIFT
<b>062</b>	STOPTIME
<b>064</b>	POSTTIME
<b>066</b>	PREPARETIME
<b>070</b>	MAXRECORDS

**Instruction Accepted RAT**

Contains line number of the tableline returned in the textpart of the reply.

**Instruction Accepted RAD**

N2..0 contains internal code of command parameter (this reply contains more than one text line!).

<b>001</b>	PARAMETER (or CONT)
<b>002 - 006</b>	CONT
<b>050</b>	STATUS

**Instruction Accepted RAC**

N2..0 contains internal code of command parameter.

<b>000</b>	IDENTIFY
<b>040</b>	START
<b>041</b>	STOP
<b>042</b>	PREPARE
<b>044</b>	SYSREADY
<b>045</b>	SYSNOTREADY
<b>047</b>	ZERO BALANCE
<b>049</b>	RESET LEAKSENSOR
<b>051</b>	RESET GPIB CONTROL
<b>053</b>	KEY LOCK
<b>054</b>	KEY UNLOCK
<b>055</b>	DATA ON
<b>056</b>	DATA OFF
	RESET INSTRUMENT

**Descriptions of Some Special GPIB Commands**

**Instruction: MAXRECORDS = 4 ... 32767**

This parameter specifies the max. number of rawdata-records that may be generated during a run . This parameter is set by the workstation to avoid disc overflow.

## Instruction and Reply Messages (InCU-1, OutCU-1)

If the actual number of records is greater than MAXRECORDS - 3 the event 'EA05 cr lf storage overflow' is generated.

### **Instruction: IDENTIFY**

Reply text: "Agilent 1049A (B 2947)" cr lf.

With this command you can identify the Agilent 1049A Electrochemical Detector. The expression in brackets is the revision and date code of the instrument firmware.

### **Instruction: SYSREADY**

Instrument action: Used to control the remote control signal "READY" at the REMOTE connector. This command resets an independent internal not ready condition that is OR'ed with the hardware related conditions and sent to the REMOTE connector.

### **Instruction: SYSNOTREADY**

Instrument action: Used to control the remote control signal "NOTREADY" at the REMOTE connector. The not ready condition is OR'ed with the hardware related conditions and sent to the REMOTE connector.

### **Instruction: RESET GPIB CONTROL**

Instrument action: WAIT handling will be switched off until new GPIB read or write will happen.

Attention: If the CU-status via CU-0 or OUT-CU (include reply CU is read, then GPIB-controlling automatically will be switched on!)

---

**NOTE**

---

Do not read the reply or any CU after this instruction. If you read or write to the detector, GPIB control will be turned-ON and the GPIB control will not be reset.

### **Instruction: KEY LOCK**

Instrument action : Parameter entry will be locked.

---

**NOTE**

---

Only RESET command group of interface will be accessible by keyboard, RESET GPIB CONTROL switched keyboard control on.

**Instruction: KEY UNLOCK**

Instrument action: see KEY LOCK.

**Instruction: DATA OFF**

Instrument action: for pure parameter control without need of handling data CUs. A DATA OFF command should be sent when only parameter control is done.

---

**NOTE**

---

The DATA OFF instruction switches off the complete raw data acquisition (Out CU4 support). This command should be set in PRERUN or while initializing the system, if only parameter control is done by GPIB.

**Instruction: DATA ON**

Instrument action: see DATA OFF.

**Instruction: RESET INSTRUMENT**

---

**NOTE**

---

This instruction causes a complete firmware coldstart! All parameters return to default values.

No Reply will be generated.

**Instruction: STATUS**

Reply header: RAD050 cr lf

RUN	tttt.tt	Agilent 1049A ready	cr lf
RUN	tttt.tt	error	cr lf
RUN	tttt.tt	caution	cr lf
RUN	tttt.tt	notready	cr lf
RUN	tttt.tt	error notready	cr lf
RUN	tttt.tt	error caution	cr lf
PRERUN	tttt.tt		cr lf
PRERUN			cr lf
POSTRUN	tttt.tt		cr lf
WAIT			cr lf

**Instruction and Reply Messages (InCU-1, OutCU-1)**

In state RUN tttt.tt is the current runtime (running forward).

In state PRERUN tttt.tt is optional. If tttt.tt is displayed the preparetime is running (backward) and the current preparetime is displayed.

In state POSTRUN tttt.tt is the current posttime (running backward).

In state WAIT no time is displayed (state WAIT will finished if all data from GPIB is read via a controller).

More lines will be generated if any ERROR, NOTREADY or CAUTION condition is true.

```

ERROR:  leak, reset sensor          cr lf
ERROR:  leaksensor failed           cr lf
ERROR:  out of temperature range    cr lf
CAUTION: test is running            cr lf
CAUTION: cell is off                cr lf
CAUTION: cell not connected         cr lf
CAUTION: increment is on            cr lf
CAUTION: reduced dynamic range      cr lf
CAUTION: analog out underflow       cr lf
CAUTION: analog out overflow        cr lf
NOTREADY: zero setting              cr lf
NOTREADY: out of fullscale          cr lf
NOTREADY: temperature high          cr lf
NOTREADY: temperature low           cr lf
NOTREADY: pretreatment running      cr lf
NOTREADY: preparetime running       cr lf
NOTREADY: drifttrigger is on        cr lf
; *END-OF-LIST

```

**NOTE**

A leak error requires a reset of the error flag by sending the command RESET LEAKSENSOR.

**Instructions: PARAMETER and CONT**

Reply header: RAD001 cr lf

```
REC RANGE = 500.0 ; nA          cr lf
ZEROCURRENT = 23.67 ; nA       cr lf
RESPONSETIME = 2.0 ; sec      cr lf
ZERO LEVEL = 10 ; % of output cr lf
POTENTIAL = 0.800 ; Volt      cr lf
INCREMENT = 0.000 ; Volt      cr lf
REPETITIONS = 1                cr lf
INSTRUMENT FULLSCALE = 0.5 uA cr lf
CONT                             cr lf
```

---

**NOTE**

The length of this multiline reply is fixed with maximum 9 lines.

A maximum 8 parameters are transferred per message, each message will be closed in the last line with **CONT** or **;\*END-OF-LIST**. **CONT** means that the parameter table is longer than the message. **;\*END-OF-LIST** means that the parameter table was completely read.

The next packages can read with the **CONT** command.

Unlimited repetition of **CONT** commands will perform a roll function in the parameter table.

---

**IMPORTANT**

Each package of maximum 8 parameters will have its own reply header for identification:

Reply header: RAD00[n] cr lf

where n = number of package

---

## ECD rawdata (OutCU-4)

The following sections describe the records generated by the detector.

ECD rawdata block consists of

- One or more ECD data records (description follows ), and;
- Exactly one ECD stop record at the end of the block.

Within the ECD data records there exist 5 different types of records, each recognizable by the fourth byte of the record. These types and their respective ID character in the fourth byte are as follows:

ID	Record name	Remark
P, p	Chromatogram plus	Chromatogram with signal parameters
M, m	Chromatogram minus	Chromatogram without signal parameters
I	Info	List of data records
G	Global	List of info records
Z	Stop record	Indicates the end

For the internal structure of an ECD data record it is not relevant whether the ID character is in upper or lower case. It has, however, some overall meaning for the analysis data.

Lower case IDs indicate system overload in the following sense:

Each record with a lower case ID indicates a foregoing gap in the analysis data. That is, the detector wanted to generate a data record, but there was no empty buffer available as a result of slow data transfer to the mass storage. This overload may result in a time gap within the chromatogram.

The generation of *info* and *global* records, however, is never affected by system overload.

All ECD data records have a 10-byte header with a fixed meaning, regardless of the record type:

Byte #	Meaning	Internal representation
1-3	ECD file identification ("#ED")	3 ASCII
4	ID ("P" or "M" or "I" or "G")	ASCII
5-8	Time of generation of record	Time
9-10	Index of data record	INTEGER (2)

---

**NOTE**

The index of data records is counted from 1!

Time is given in units of milliseconds in a 4-byte 2's-compl. Integer.

---

### Chromatogram ("P", "M" records)

Here we have two types of records: One with (i.e. "P" or "p", called "plus") and the other without (i.e. "M" or "m", called "minus") the actual signal acquisition parameters. Each time these parameters are changed during analysis (or in sweep-mode a fix potential : sweep pot1 or sweep pot2 is reached), a new "plus" record is generated. These parameters are valid for all subsequent "minus" records until the next "plus" record is stored. A "plus" record always contains the complete set of parameters, regardless of which or how many single parameters have changed.

The rough structure of both records may be described as follows:

P record byte #	Meaning	M record byte #
1-10	ECD data record header	1-10
11-18	Chromatogram header	11-18
19-52	Signal parameters	Non-existent
53-256	Chromatogram data	19-256

### Structure of ECD Data Record Header

Byte #	Meaning	Internal representation
1-3	ECD file identification (“#ED”)	3 ASCII
4	ID	ASCII
5-8	Time of generation of record	Time
9-10	Index of data record	INTEGER (2)

The field *time of generation of record* contains in this context the time of the *last* sample within the record in millisecc.

### Structure of Chromatogram Header

Byte #	Meaning	Internal representation
11-12	Index of previous P record	INTEGER (2)
13-14	Number of samples in this record	INTEGER (2)
15-16	Current Potential (numerator)	INTEGER (2)
17-18	Current Potential (denominator)	INTEGER (2)

- 1 The first "P" record has a zero in the field "index of previous P record".
- 2 In a "M" record the field "index of previous P record" can never be zero.

**Structure of Signal Parameter**

<b>Byte #</b>	<b>Meaning</b>	<b>Internal representation</b>
19-22	Time increment between two adjacent samples	Time
23-24	Reason for parameter change ("MA" manual)	2 ASCII
25-26	POLARITY 0 = OXIDATION = normal mode 1 = REDUCTION = inverted peak mode	INTEGER (2)
27-28	ZEROCURRENT = scaling-offset (numerator)	INTEGER (2)
29-30	ZEROCURRENT (denominator) (= fix: 10)	INTEGER (2)
31-34	INSTRUMENT FULLSCALE (numerator) = resolution (as Signed_32) 500 or 50 or 100 (Ref-Test)	INTEGER (4)
35-38	INSTRUMENT FULLSCALE (denominator) = Max_S24 = 2E23-1 (as Signed_32)	INTEGER (4)
39-42	Rate of Potential-Increments in [V/msec] (numerator)	INTEGER (4)
43-46	Rate (denominator)	INTEGER (4)
47-48	Length of Unit String (norm. 4)	INTEGER (2)
49-52	Unit as String "nA", "uA", "mV"	4 ASCII

**Chromatogram Data Part**

Comprises an array of current values. The physical dimension is defined by **INSTRUMENT FULLSCALE**. Values are coded in a 3-byte 2's complement INTEGER. The data path implementation is shown below:

Cell current - ZEROCURRENT			
Case <b>INSTRUMENT FULLSCALE</b> of			
0.05 µA:	-50 nA	0 nA	+50nA
0.5 µA:	-500 nA	0 nA	+500nA
500 µA:	-500 µA	0 nA	+500 µA
Rawdata values/Samples (24 bit)			
	80 00 00H	0	7F FF FF
Analog out definition			
	underflow	0 V x% (Zerolevel)	+1 V

It is possible to calculate the cell current using the following equations.

If POLARITY (bytes 25-26) = 0 = OXIDATION, then:

$$\text{Cell current [Unit]} = \text{ZEROCURRENT} + \text{INSTRUMENT FULLSCALE} \times \text{Rawdata}$$

Otherwise, if POLARITY (bytes 25-26) = 1 = REDUCTION, then:

$$\text{Cell current [Unit]} = \text{ZEROCURRENT} - \text{INSTRUMENT FULLSCALE} \times \text{Rawdata}$$

Where:

**ECD rawdata (OutCU-4)**

Units as string (bytes 49-52) = unit

String length is 4 (bytes 47-48)

Strings are **nA** , **uA** , **mV**

**ZEROCURRENT**/10 (bytes 27-30)

**ZEROCURRENT** varies from -500 to +500

**INSTRUMENT FULLSCALE** (bytes 31-38)

**INSTRUMENT FULLSCALE** values are 50, 500, 1000

---

**NOTE**

The default POLARITY=OXIDATION equals to standard chromatograms as usual with absorbance or fluorescence detection; this is the case for 80% of applications.

For negative peak settings of POLARITY=REDUCTION negates the signal for standard data handling. But in terms of physical units a new definition is required by changing the sign of the expression to minus.

---

**NOTE**

Each **ZERO BALANCE** sets Rawdata/Samples near to 0; only with **GPIB** or **MONITOR** also negative data can be monitored; in spite of that balancing should be done with baseline values higher than about 5% far from 0 because of hardware-definition concerning dynamic range of analog hardware.

A general rule for proper balancing is to interpret the caution message **analog underflow** to do a ZEROBALANCE.

---

**Chromatogram Data Part and Sweep Handling**

A sweep is a controlled potential ramp with a start or working potential and 2 potentials where the sign of the ramp is changed periodically. While doing the ramp, standard data acquisition will work. So more than one current unit will be related to one potential. The resulting X-Y plots are so-called hydrodynamic voltamograms.

According to the standard chromatogram definition the records will have the standard protocol, which means the first record after START will be P-type, all subsequent ones will be M-types.

---

**NOTE**

---

The only exception with sweeps is the generation of one P-type record when changing the direction of the ramp.

The end of the sweep is finished by a Z-type record.

By the specifiers:

current potential (bytes 15-18)

rate of potential increments (bytes 39-46)

time increment (bytes 19-22)

and the P-type information, all potential values and related current values are defined as shown with the following equation.

**VALUE = meaning, byte #, internal rep.**

TI [ms] = time increment, 19-22, INTEGER(4)

CP [V] = current potential = 15-16/17-18 =  
INTEGER(2)/INTEGER(2)

RA [V/ms] = Rate of Potential = 39-42/43-46 =  
INTEGER(4)/INTEGER(4)

It is possible to calculate the potential of every data-point. The potential of the first data-point is CP. The potential of the n<sup>th</sup> data-point is:

$$CP + [(n-1)(TI)(RA)]$$

### **Info and Global ("I", "G" records)**

For the sake of fast (almost direct) access to the ECD rawdata a special retrieval system is maintained during analysis and inserted as "info" and "global" records within the measurement data records. Thereby the "info" records are a list of contents of all ECD data records in the file, whereas the "global" records are a list of contents of all "info" records in the file.

#### **Info ("I" record)**

This record primarily consists of an array of 6-byte elements (ID and time of this record). The number of elements is at most 40. The last of these elements represents the "info" record itself. Thus, by chaining together in RAM these arrays of all "info" records, you can obtain a gapless directory of all ECD data records. The data record index can then be used to directly access the mass storage file.

Structure of "info" record:

Byte #	Meaning	Internal representation
1-3	ECD file identification ("#ED")	3 ASCII
4	ID ("I")	ASCII
5-8	Time of generation of this record	Time
9-10	Data record index	INTEGER (2)
11-12	Index of previous "info" record	INTEGER (2)
13-14	Number of elements in this record	INTEGER (2)
15-256	Array of elements	element

---

**NOTE**

---

The first "Info" record of the file has a zero in the field "index of previous info record".

Structure of element (byte counting relative to element):

Relative byte #	Meaning	Internal representation
1	ID of data record	ASCII
2	Not used	
3-6	Time of data record	Time

**Global ("G" record)**

This record is a list of "info" records together with their essential contents. The number of info entries is at most 40. Therefore the span of one "global" record can be up to 1600 data records.

Structure of "global" record:

Byte #	Meaning	Internal representation
1-3	ECD file identification ("#ED")	3 ASCII
4	ID ("G")	ASCII
5-8	Time of generation of this record	Time
9-10	Data record index	INTEGER (2)
11-12	Index of previous "global" record	INTEGER (2)
13-14	Number of info entries	INTEGER (2)
15-256	Array of info entries	info entry

---

**NOTE**

---

The first "global" record has a zero in the field "index of previous global record".

Structure of info entry (byte counting relative to info entry)::

Relative byte #	Meaning	Internal representation
1-2	Index of "info" record	INTEGER (2)
3-6	Time of last chromatogram record within the span of the "info" record	Time

- 1 If there was no chromatogram record generated within the scope of the particular "info" record, the appropriate time in the info entry is set to zero.
- 2 All chromatogram records are sorted by increasing time.

### **Stop Record ("Z" record)**

It indicates the end of ECD rawdata part and contains information about the firmware, also a timing information.

Structure:

Using the GPIB Interface  
**ECD rawdata (OutCU-4)**

<b>Byte #</b>	<b>Meaning</b>	<b>Internal representation</b>
1-3	ECD file identification (“#ED”)	3 ASCII
4	ID (“Z”)	ASCII
5-10	Firmware revision	6 ASCII
11-14	Time of last chr sample	Time

---

## Chromatogram Plot Data Structure (OutCU-2)

This data structure has a constant record length of 40 byte and contains 12 samples. The frequency of incoming record is every 600 msec. The time increment between two adjacent samples is constant 50 ms independent of the responsetime.

Byte #	Meaning
1-4	Number of lost samples
5-7	1 <sup>st</sup> sample
.	.
.	.
.	.
38-40	12 <sup>th</sup> sample

## Events (OutCU-5)

The event messages generated by the Agilent 1049A Electrochemical Detector have following common format:

4-CHAR-header

cr lf

text

Power-On Events:

Header	Text	Reason
EC01 cr lf	<b>1049A power on</b>	After power on
EC02 cr lf	<b>1049A cleared</b>	After warmstart with SCG31
EC03 cr lf	<b>parameter lost</b>	After INIT or power-on
EC04 cr lf	not used	not used

Error Events:

Header	Text
ER01 cr lf	<b>leak detected</b>
ER02 cr lf	<b>leak sensor failed</b>
ER03 cr lf	<b>out of temperature range</b>

Analysis State Transition Events:

**Events (OutCU-5)**

<b>Header</b>	<b>Text</b>
EA01 cr lf <b>RUN</b>	Detector has entered RUN-state after a START-command
EA02 cr lf <b>WAIT</b>	Detector has entered WAIT after a STOP, but Rawdata records are still to be read from the internal buffer.
EA03 cr lf <b>PRERUN</b>	Detector has entered PRERUN-state after elapsed posttime and after a STOP-command and after all Rawdata records are read from the external computer.
EA04 cr lf <b>POSTRUN</b>	Detector has entered POSTRUN-state after WAIT while the posttime-clock is running
EA05 cr lf <b>detector overflow</b>	The internal rawdata buffer is full because the external computer does not read rawdata records. Data are lost.
EA06 cr lf <b>storage overflow</b>	There is not enough space on disk (see MAXRECORDS).

---

## Examples

### Example of a BASIC Program

The following is an example of a communication program written in BASIC for the HP-85B Personal Computer and Agilent 1049A Electrochemical Detector.

```
100 ! *****
110 !  "CHEMFLY GPIB"
120 !  *****
130 !
140 ! Global Var
150 !
160 DIM Z$(520) ! Buffer
170 DIM I$(50) ! In -String
180 DIM O$(50) ! Out-String
190 !
200 A=11 ! GPIB address
210 N=1 ! CU read or write
220 ! N=0: Status-CU
230 ! N=1: Instruction/Reply-CU
240 ! N=2: Chromatogram-Plot-CU
250 ! N=3: not used
260 ! N=4: Rawdata-RUN-CU
270 ! N=5: Event-CU
280 !
290 ! Subroutines
300 !
310 ! 2000 Instruction
320 ! ---- Input,Send & Reply
330 ! 2500 Read CU N in Z$
340 ! ----
350 ! 3000 Instruction
360 ! ---- Send & Reply
370 ! 3500 Reply
380 ! ----
390 !
400 !
990 ! *****
1000 ! MAIN-PROGRAM
1010 ! *****
1020 !
1030 CLEAR
1040 !
```

## Using the GPIB Interface

### Examples

```
1050 I$="DATA OFF"
1060 GOSUB 3000
1070 !
1100 DISP ""
1110 GOSUB 2000
1120 GOTO 1100
1130 !
1140 STOP
1150 !
1160 !
1990 ! *****
2000 ! Instruction Input,Send
2010 ! *****
2020 !
2030 LINPUT "enter instruction :
      ",I$
2040 GOSUB 3000 ! Send instr.
2050 RETURN
2060 !
2070 !
2490 ! *****
2500 ! Read CU N in Z$
2510 ! *****
2520 !
2530 IOBUFFER Z$
2540 SEND 7 ; UNT UNL MLA TALK A
      SCG N
2550 RESUME 7
2560 TRANSFER 7 TO Z$ FHS ; EOI
2570 RETURN
2580 !
2590 !
2990 ! *****
3000 ! Instruction Send & Reply
3010 ! *****
3020 !
3030 IOBUFFER Z$
3040 Z$=I$
3050 SEND 7 ; UNT UNL MTA LISTEN
      A SCG 1
3060 RESUME 7
3070 CONTROL 7,16 ; 128 ! EOI
3080 TRANSFER Z$ TO 7 FHS
3090 !
3100 !
3490 ! *****
3500 ! Reply: Read & Display
3510 ! *****
3520 !
3530 N=0 ! Read CU 0
2540 GOSUB 2500
```

## Using the GPIB Interface

### Examples

```
3550 IF BIT(NUM(Z$(2,2)),1) THEN
      GOTO 3580
3560 GOTO 3530
3570 !
3580 N=1 ! Read CU 1
3590 GOSUB 2500
3600 !
3610 DISP Z$(1,6)
3620 !
3630 L=LEN(Z$)
3640 I=9
3650 S=9
3660 IF I>L THEN 3760
3670 IF Z$(I,I)=CHR$(13) THEN GO
      TO 3710
3680 I=I+1
3690 GOTO 3660
3700 !
3710 DISP Z$(S,I-1]
3720 I=I+2
3730 S=I
3740 GOTO 3660
3750 !
3760 RETURN
3770 !
3780 !
8990 ! *****
9000 ! END OF "CHEMFLY GPIB"
9010 ! *****
```

### Example of a Pascal Program

The following is an example of a communication program written in Pascal for the LC ChemStation (Pascal series) and Agilent 1049A Electrochemical Detector.

```
$SYSPROG$
$UCSD$

PROGRAM CHEMFLY (INPUT,OUTPUT,LISTING);

IMPORT IODECLARATIONS, IOCOMASM,
      GENERAL_0, GENERAL_1, GENERAL_2,
      GPIB_0, GPIB_1, GPIB_2, GPIB_3;

VAR
  MY_CHAR      : CHAR;
  MY_ADR      : INTEGER;
  MY_GPIBSTRING : STRING[255];
```

## Using the GPIB Interface

### Examples

```
CONST
    STATUSCU      = 0;    { used CUs }
    INSTRUCTIONCU = 1;

    SUMMARY      = 1;    { Byte of Status-CU }
    REPLY        = 2;
    INSTR        = 7;

    MY_GPIB      = 7;    { address of GPIB-interface-card }
    MY_DEVICE    = 11;   { GPIB-address of Agilent 1049A }

    RETRIES      = 100;  { communication retries to get ready status }

{*****}

FUNCTION SENDREADY : BOOLEAN;

VAR
    MY_WORD      :      0 .. 255;
    I            :      INTEGER;
    ENDING       :      BOOLEAN;
    RETRY        :      INTEGER;

BEGIN
    ENDING := FALSE;
    RETRY  := 0;
    REPEAT
        UNTALK (MY_GPIB);
        UNLISTEN (MY_GPIB);
        TALK (MY_GPIB,MY_DEVICE);
        SECONDARY (MY_GPIB,STATUSCU);
        LISTEN (MY_GPIB,MY_ADDRESS(MY_GPIB));
        I := 1;
        REPEAT
            READCHAR (MY_GPIB,MY_CHAR);
            MY_WORD := ORD(MY_CHAR);
            IF (I = INSTR) AND NOT (BIT_SET(MY_WORD,0)) THEN ENDING :=
TRUE;
                I := I + 1;
            UNTIL END_SET(MY_GPIB);
            RETRY := RETRY + 1;
        UNTIL ENDING OR (RETRY=RETRIES);
        SENDREADY := ENDING;
    END;

{*****}

FUNCTION READREADY : BOOLEAN;

VAR
    MY_WORD      :      0 .. 255;
```

## Using the GPIB Interface

### Examples

```
I           :           INTEGER;
ENDING      :           BOOLEAN;
RETRY       :           INTEGER;

BEGIN
  ENDING := FALSE;
  RETRY  := 0;
  REPEAT
    UNTALK (MY_GPIB);
    UNLISTEN (MY_GPIB);
    TALK (MY_GPIB,MY_DEVICE);
    SECONDARY (MY_GPIB,STATUSCU);
    LISTEN (MY_GPIB,MY_ADDRESS(MY_GPIB));
    I := 1;
    REPEAT
      READCHAR (MY_GPIB,MY_CHAR);
      MY_WORD := ORD(MY_CHAR);
      IF (I = REPLY) AND (BIT_SET(MY_WORD,1)) THEN ENDING := TRUE;
      I := I + 1;
    UNTIL END_SET(MY_GPIB);
    RETRY := RETRY + 1;
  UNTIL ENDING OR (RETRY=RETRIES);
  READREADY := ENDING;
END;

{*****}

PROCEDURE READGPIB;

VAR
  DUMMY           :           INTEGER;

BEGIN
  WRITE ('String from Device', MY_ADR, ' : ');
  UNTALK (MY_GPIB);
  UNLISTEN (MY_GPIB);
  TALK (MY_GPIB,MY_DEVICE);
  SECONDARY (MY_GPIB,INSTRUCTIONCU);
  LISTEN (MY_GPIB,MY_ADDRESS(MY_GPIB));
  MY_GPIBSTRING := '';
  DUMMY := 1;
  REPEAT
    READCHAR (MY_GPIB,MY_CHAR);
    STRWRITE (MY_GPIBSTRING,DUMMY,DUMMY,MY_CHAR);
  UNTIL END_SET(MY_GPIB);
  MY_GPIBSTRING := STRLTRIM (STRRTRIM (MY_GPIBSTRING));
END;

{*****}

PROCEDURE SENDGPIB;
```

## Using the GPIB Interface

### Examples

```
VAR
    I           :           1 .. 255;

BEGIN
    WRITE ('String to Device', MY_ADR, ' : ');
    READLN (MY_GPIBSTRING);
    MY_GPIBSTRING := STRLTRIM (STRRTRIM (MY_GPIBSTRING));

    UNTALK (MY_GPIB);
    UNLISTEN (MY_GPIB);
    LISTEN (MY_GPIB,MY_DEVICE);
    SECONDARY (MY_GPIB,INSTRUCTIONCU);
    TALK (MY_GPIB,MY_ADDRESS(MY_GPIB));
    FOR I := 1 TO STRLEN (MY_GPIBSTRING) - 1 DO
        WRITECHAR (MY_GPIB,MY_GPIBSTRING[I]);
    SET_GPIB(MY_GPIB,EOI_LINE);
    WRITECHAR (MY_GPIB,MY_GPIBSTRING[STRLEN(MY_GPIBSTRING)]);
END;

{*****}

BEGIN
    MY_ADR := MY_GPIB * 100 + MY_DEVICE;

    CLEAR (MY_ADR);

    IF SENDREADY THEN SENDGPIB;

    IF READREADY THEN READGPIB;

    WRITELN (MY_GPIBSTRING);

    CLEAR (MY_ADR);

END.
```





## Warranty Statement

### All Chemical Analysis Products

Agilent Technologies (Agilent) warrants its chemical analysis products against defects in materials and workmanship. For details of the warranty period in your country, call Agilent. During the warranty period, Agilent will, at its option, repair or replace products which prove to be defective. Products that are installed by Agilent are warranted from the installation date, all others from the ship date.

If buyer schedules or delays installation more than 30 days after delivery, then warranty period starts on 31<sup>st</sup> day from date of shipment (60 and 61 days, respectively for products shipped internationally).

Agilent warrants that its software and firmware designed by Agilent for use with a CPU will execute its programming instructions when properly installed on that CPU. Agilent does not warrant that the operation of the CPU, or software, or firmware will be uninterrupted or error-free.

### Limitation of Warranty

Onsite warranty services are provided at the initial installation point. Installation and onsite warranty services are available only in Agilent service travel areas, and only in the country of initial purchase unless buyer pays Agilent international prices for the product and services. Warranties requiring return to Agilent are not limited to the country of purchase.

For installation and warranty services outside of Agilent's service travel area, Agilent will provide a quotation for the applicable additional services.

If products eligible for installation and onsite warranty services are moved from the initial installation point, the warranty will remain in effect only if the customer purchases additional inspection or installation services, at the new site.

The foregoing warranty shall not apply to defects resulting from:

- 1 improper or inadequate maintenance, adjustment, calibration, or operation by buyer,
- 2 buyer-supplied software, hardware, interfacing or consumables,
- 3 unauthorized modification or misuse,

## Warranty Statement

- 4 operation outside of the environmental and electrical specifications for the product,
- 5 improper site preparation and maintenance, or
- 6 customer induced contamination or leaks.

THE WARRANTY SET FORTH IS EXCLUSIVE AND NO OTHER WARRANTY, WHETHER WRITTEN OR ORAL, IS EXPRESSED OR IMPLIED. AGILENT SPECIFICALLY DISCLAIMS THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

## Limitation of Remedies and Liability

THE REMEDIES PROVIDED HEREIN ARE BUYER'S SOLE AND EXCLUSIVE REMEDIES. IN NO EVENT SHALL AGILENT BE LIABLE FOR DIRECT, INDIRECT, SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES (INCLUDING LOSS OF PROFITS) WHETHER BASED ON CONTRACT, TORT OR ANY OTHER LEGAL THEORY.

## Responsibilities of the Customer

The customer shall provide:

- 1 access to the products during the specified periods of coverage to perform maintenance,
- 2 adequate working space around the products for servicing by Agilent personnel,
- 3 access to and use of all information and facilities determined necessary by Agilent to service and/or maintain the products (insofar as these items may contain proprietary or classified information, the customer shall assume full responsibility for safeguarding and protection from wrongful use),
- 4 routine operator maintenance and cleaning as specified in the Agilent operating and service manuals, and
- 5 consumables such as paper, disks, magnetic tapes, ribbons, inks, pens, gases, solvents, columns, syringes, lamps, septa, needles, filters, frits, fuses, seals, detector flow cell windows, and so on.

## Responsibilities of Agilent Technologies

Agilent Technologies will provide warranty services as described in the following table.

**Table 13**

<b>Warranty Services</b>		
<b>Services During Warranty *</b>	<b>Warranty Period **</b>	<b>Type</b>
Agilent CE instruments, Agilent 1100 Series LC modules, Agilent 8453 UV-visible spectrophotometers	1 Year	Onsite
CE, LC, UV-visible supplies and accessories	90 Days	Onsite
Columns and consumables ***	90 Days	Return to Agilent
Gas discharge and tungsten lamps	30 Days	Return to Agilent
Repairs performed onsite by Agilent ****	90 Days	Onsite

\* This warranty may be modified in accordance with the law of your country. Please consult your local Agilent office for the period of the warranty, for shipping instructions and for the applicable wording of the local warranty.

\*\* Warranty services are included as specified for chemical-analysis products and options purchased concurrently provided customer is located within a Agilent-defined travel area. Agilent warranty service provides for 8 a.m. to 5 p.m. onsite coverage Monday through Friday, exclusive of Agilent holidays.

\*\*\* Columns and consumables are warranted to be free from defects for a period of 90 days after shipment and will be replaced on a return-to-Agilent basis if unused.

\*\*\*\* Agilent repair warranty is limited to only the item repaired or replaced.

# Safety Information

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Agilent Technologies assumes no liability for the customer's failure to comply with these requirements.

## General

This is a Safety Class I instrument (provided with terminal for protective earthing) and has been manufactured and tested according to international safety standards.

## Operation

Before applying power, comply with the installation section. Additionally the following must be observed.

Do not remove instrument covers when operating. Before the instrument is switched on, all protective earth terminals, extension cords, auto-transformers, and devices connected to it must be connected to a protective earth via a ground socket. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. Whenever it is likely that the protection has been impaired, the instrument must be made inoperative and be secured against any intended operation.

Make sure that only fuses with the required rated current and of the specified type (normal blow, time delay, and so on) are used for replacement. The use of repaired fuses and the short-circuiting of fuseholders must be avoided.

Some adjustments described in the manual, are made with power supplied to the instrument, and protective covers removed. Energy available at many points may, if contacted, result in personal injury.

Any adjustment, maintenance, and repair of the opened instrument under voltage should be avoided as much as possible. When inevitable, this should be carried out by a skilled person who is aware of the hazard involved. Do not attempt internal service or adjustment unless another person, capable of

## Safety Information

rendering first aid and resuscitation, is present. Do not replace components with power cable connected.

Do not operate the instrument in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a definite safety hazard.

Do not install substitute parts or make any unauthorized modification to the instrument.

Capacitors inside the instrument may still be charged, even though the instrument has been disconnected from its source of supply. Dangerous voltages, capable of causing serious personal injury, are present in this instrument. Use extreme caution when handling, testing and adjusting.

## Safety Symbols

Table 14

**Safety Symbols used on Instruments and in Manuals**

Symbol	Description
	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect the apparatus against damage.
	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	Eye damage may result from directly viewing light produced by deuterium lamps used in detectors and spectrophotometers. Always turn off the deuterium lamp before opening the lamp door on the instrument.

---

### WARNING

**A warning alerts you to situations that could cause physical injury or damage to the equipment. Do not proceed beyond a warning until you have fully understood and met the indicated conditions.**

---

### CAUTION

A caution alerts you to situations that could cause a possible loss of data. Do not proceed beyond a caution until you have fully understood and met the indicated conditions.

## Solvent Information

Observe the following recommendations on the use of solvents.

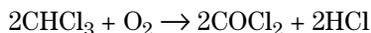
### Flow Cells

Avoid the use of alkaline solutions (pH > 9.5) which can attack quartz and thus impair the optical properties of flow cells.

### Solvents

Always filter solvents, small particles can permanently block capillaries. Avoid the use of the following steel-corrosive solvents:

- Solutions of alkali halides and their respective acids (for example, lithium iodide, potassium chloride, and so on).
- High concentrations of inorganic acids like nitric acid, sulfuric acid especially at higher temperatures (replace, if your analysis method allows, by phosphoric acid or phosphate buffer which are less corrosive against stainless steel).
- Halogenated solvents or mixtures which form radicals and/or acids, for example:



This reaction, in which stainless steel probably acts as a catalyst, occurs quickly with dried chloroform if the drying process removes the stabilizing alcohol.

- Analysis-grade ethers, which can contain peroxides (for example, THF, dioxane, di-isopropylether) such ethers should be filtered through dry aluminium oxide which adsorbs the peroxides.
- Solutions of organic acids (acetic acid, formic acid, and so on) in organic solvents. For example, a 1-% solution of acetic acid in methanol will attack steel.
- Solutions containing strong complexing agents (for example, EDTA, ethylene diamine tetra-acetic acid).
- Mixtures of carbon tetrachloride with 2-propanol or THF.

## **Radio Interference**

### **Manufacturer's Declaration**

This is to certify that this equipment is in accordance with the Radio Interference Requirements of Directive FTZ 1046/1984. The German Bundespost was notified that this equipment was put into circulation, the right to check the series for compliance with the requirements was granted.

### **Test and Measurement**

If test and measurement equipment is operated with equipment unscreened cables and/or used for measurements on open set-ups, the user has to assure that under operating conditions the radio interference limits are still met within the premises.

## Sound Emission

### Manufacturer's Declaration

This statement is provided to comply with the requirements of the German Sound Emission Directive of 18 January 1991.

This product has a sound pressure emission (at the operator position) < 70 dB.

- Sound Pressure  $L_p < 70$  dB (A)
- At Operator Position
- Normal Operation
- According to ISO 7779:1988/EN 27779/1991 (Type Test)

## **Agilent Technologies on Internet**

For the latest information on products and services visit our worldwide web site on the Internet at:

<http://www.agilent.com/chem>





**Agilent Technologies**

## **In This Book**

This handbook is for the Agilent 1049A electrochemical detector. It describes installation, operation, maintenance and troubleshooting of your detector and is designed to be used as both a beginner's self-teaching guide and a day-to-day reference guide.



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