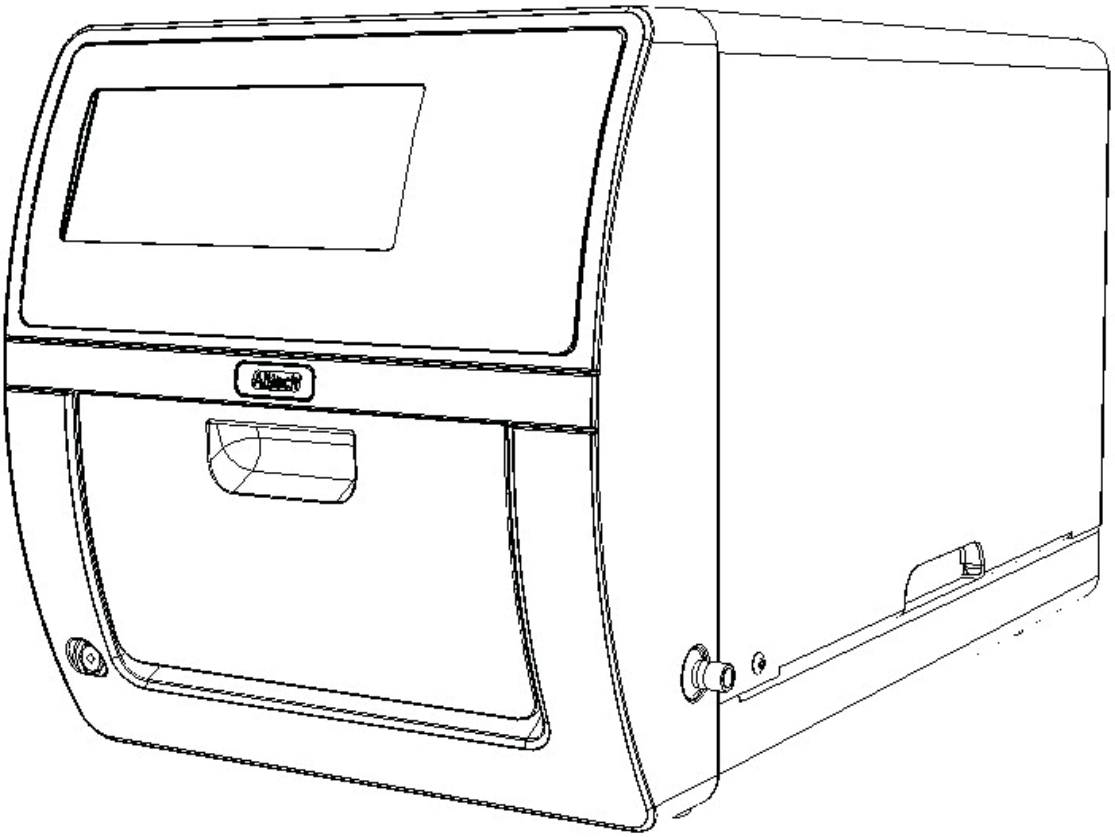
Alltech® Model 3300 ELSD
Operation Manual



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Safety Symbols used in this Manual:

⚠ DANGER

The Danger symbol indicates a hazardous situation that, if not avoided, will result in death or serious injury.

⚠ WARNING

The Warning symbol indicates a hazardous situation that, if not avoided, could result in death or serious injury.

⚠ CAUTION

The Caution symbol indicates a hazardous situation that, if not avoided, could result in minor or moderate injury.

NOTICE

The Notice symbol is used to highlight information that will optimize the use and reliability of the system.

Important Safety Guidelines for the Model 3300 ELSD

⚠ DANGER

Please read the following cautions and warning statements carefully before using the Model 3300 ELSD:

- *The equipment must be used as specified by the manufacturer otherwise overall safety will be impaired.*
- *All service must be completed by qualified personnel only.*
- *Only use the power supply cord recommended by the manufacturer.*
- *Fire hazard, only use the same type and rated CERTIFIED fuse (—▭— 250V 5A F).*
- *Shock hazard, disconnect power before replacing the fuse.*
- *Remove power cord from the power module to disconnect electrical power from the unit.*
- *Check the MSDS forms and dispense and dispose of all reagents in accordance with local and national regulations.*
- *Be sure to provide proper ventilation for all solvent vapors.*
- *Avoid open flames and sparks when using flammable solvents.*
- *If the unit is damaged and does not function properly, stop the unit safely and contact the manufacturer immediately.*
- *Class 3B LASER radiation inside can cause severe eye damage. Do not open or defeat interlocks. Avoid exposure to the beam.*

Table of Contents

1. INTRODUCTION

1.1 ABOUT THE MODEL 3300 ELSD

The Model 3300 ELSD is designed for use with High Performance Liquid Chromatography (HPLC) systems to analyze any sample compound that has sufficiently lower volatility than the mobile phase. Some of its possible application areas include carbohydrates, pharmaceuticals, lipids, triglycerides, underivitized fatty and amino acids, polymers, surfactants, nutraceuticals, and combinatorial libraries.

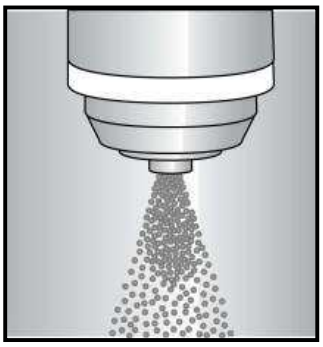
Evaporative light scattering detection eliminates common problems associated with other HPLC detectors. Refractive Index (RI) detectors can be complicated by solvent front interferences, baseline instability, and gradient incompatibility. RI detectors can also have a less sensitive response than ELSD. Low-wavelength UV can suffer baseline drift with steep gradients and also requires that the analyte contains a chromophore. ELSD does not have these limitations. It can achieve stable baselines with multisolvent gradients for improved resolution and faster separations. Also, since ELSD response does not depend on the sample's optical characteristics, the sample does not require a chromophore or fluorophore for detection.

The Model 3300 ELSD features the most advanced evaporative light scattering detection technology available. It provides sensitivity in the low nanogram range. An intuitive, Windows®-based software interface provides a nested menu of options for detector operation, including Method Wizard for simplified method development, multiple languages, and built-in Diagnostic tests for troubleshooting. The Model 3300 ELSD also features compact, stackable dimensions, allowing it to fit easily into limited laboratory space.

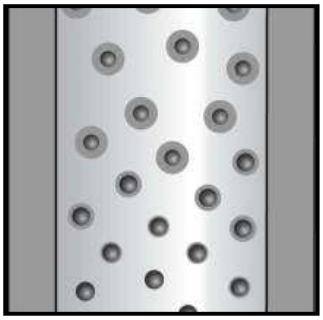
1.2 PRINCIPLE OF OPERATION

The unique detection principle of evaporative light scattering detection involves a three-step process: nebulization of the column effluent to form an aerosol, solvent evaporation within a heated drift tube, and detection of the non-volatile solute particles in the light scattering cell.

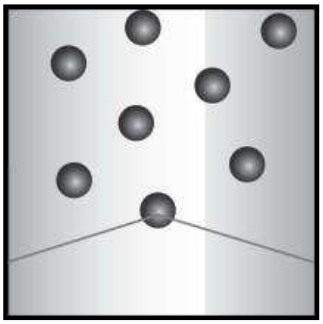
NEBULIZATION



EVAPORATION



DETECTION



NEBULIZATION

The column effluent from an HPLC separation enters the nebulizer, where it is mixed with a steady stream of nebulizing gas (usually nitrogen) to form an aerosol. The aerosol consists of a uniform distribution of droplets whose size is dependent on the gas flow rate used for the analysis. The lower the gas flow rate used, the larger the resulting droplets will be. Larger droplets scatter more light and increase the sensitivity of the analysis, but they are also more difficult to evaporate in the drift tube. There will be an optimum gas flow rate for each method which will produce the highest signal-to-noise ratio.

Lower mobile phase flow rates will require lower gas flow rates for optimal nebulization. Substitution of a 2.1mm i.d. column for your standard 4.6mm i.d. column will allow you to greatly reduce the mobile phase flow rate while also increasing the sensitivity of the analysis.

EVAPORATION

Evaporation of the volatile components in the aerosol occurs in a heated, stainless steel drift tube. The optimum drift tube temperature setting for an application will depend on mobile phase composition, mobile phase flow rate, and on sample volatility. Highly organic mobile phases require lower drift tube temperatures for evaporation than highly aqueous mobile phases. Lower mobile phase flow rates require lower drift tube temperatures than higher mobile phase flow rates. Semi-volatile analytes require the use of much lower drift tube temperatures to obtain optimum sensitivity. The optimum temperature should be determined by observing the signal-to-noise ratio with respect to temperature.

Within the Model 3300 ELSD drift tube, a PTFE-coated, stainless steel impactor plate is located three inches from the nebulizer. The plate is perpendicular to the aerosol path so as the aerosol contacts the plate, larger droplets exit through the drain tube on the side panel. The remaining droplets pass around the impactor and travel through the drift tube to the optical cell for detection. The removal of the larger droplets allows operation of the Model 3300 ELSD at low temperatures, making it ideal for the analysis of semi-volatile compounds.

Non-volatile impurities in the mobile phase or nebulizing gas will produce noise. Using the highest quality gas, solvents, and volatile buffers (preferably filtered) will greatly reduce baseline noise. Noise will also increase if the mobile phase has not been completely evaporated. Detector settings must be carefully selected to ensure adequate mobile phase evaporation.

DETECTION

The non-volatile sample particles emerge from the drift tube in the mobile phase vapor and enter the light scattering cell. In the optical cell, sample particles scatter light emitted by a laser light source. The scattered light is detected by a silicon photodiode, generating a signal proportional to sample mass. This signal is sent to the analog output for data collection. The advanced design of the Model 3300 ELSD optical components[1] provides excellent sensitivity for HPLC analysis.

[1] US Patent #6229605; other patents pending

2. INSTALLATION

2.1 WHAT YOU WILL NEED

In addition to the Model 3300 ELSD detector and its accessories, the following will be needed for installation of a complete chromatographic system:

Exhaust System:

- A fume hood or other ventilation device located close to the detector to remove the detector exhaust from the laboratory.

NOTICE

Make sure the exhaust system provides adequate but not excessive suction. Excessive suction can cause a noisy baseline.

Gas Supply:

- A supply of clean, dry nebulization gas, preferably nitrogen, regulated from 65 to 80psig. 99.9% purity or better is recommended. The gas source can be a high-pressure gas cylinder, high-pressure liquid tank, or a nitrogen generator.

HPLC System Components:

- An HPLC pump, isocratic or gradient, capable of low-pulsation solvent delivery at a flow rate ranging from at least 0.1 to 1.5mL/min against pressures of at least 3,000psig. Lower flow rate capabilities may be necessary for smaller bore columns.
- An autosampler or manual injection valve.
- A column capable of separating the compounds of interest.
- A guard column or cartridge compatible with the separation column is recommended to prolong separation column lifetime.
- A column heater, if needed.
- A data system or integrator, capable of accepting analog voltage data. 0-10mV or 0-1000mV systems can be used.

Other:

- HPLC-grade mobile phase solvents.

NOTICE

Only volatile buffers may be used in the mobile phase. Refer to Section 7.4, Volatile Mobile Phase Modifiers, for a list of suitable buffers.

- Solvent reservoirs, tubing, inlet filters, paper, etc. required for pump and data system operation. Consult the appropriate manuals for requirements.

2.2 UNPACKING

The Model 3300 ELSD detector and its accessories are shipped in the same container. Unpack components carefully, making sure all items in the list below have been included and are in good condition. Save the container and packing material for future use.

The Model 3300 ELSD shipping container should contain the following:

- Model 3300 ELSD
- Model 3300 ELSD Operating Manual
- Model 3300 ELSD Performance Documents
- Model 3300 ELSD Driver for Agilent ChemStation CD*
- Power Cord
- Signal Cable
- PEEK Tubing: 1/16" o.d. x .005" i.d., 10’
- SofGrip™ Fittings, 10/pk
- Gas Tubing, 10’
- Gas Fittings: 1/8” Brass Nut and Ferrule
- Drain Tubing, 5’ (Reservoir not included)
- Drain Tubing Clamp
- Exhaust Adapter
- Exhaust Tubing, 20’
- 14-Pin Connector
- Open-End Wrench, 3/8" x 7/16"
- Open-End Wrench, 1/4" x 5/16"
- Hex Ball Driver, 3/32" x 5 1/4”
- Hex Ball Driver, 7/64" x 4”
- Drift Tube Cleaning Brush
- Fuse, 5 Amp

*The Model 3300 ELSD Driver for Agilent ChemStation CD can be used with Agilent ChemStation to control and collect data from the 3300 ELSD. Agilent ChemStation must be purchased separately.

The Model 3300 ELSD has been carefully shipped to ensure that it is received in proper condition. Any damage to the container or its contents should be reported immediately to your local BUCHI Customer Service office which can be found on the BUCHI website at: www.buchi.com. Please refer to **Section 7.5**, Warranty, Returns, and Repairs, for more information.
Refer to **Section 7.2**, Replacement Parts, for part numbers if replacement parts are needed.

2.3 CONTROLS AND FEATURES

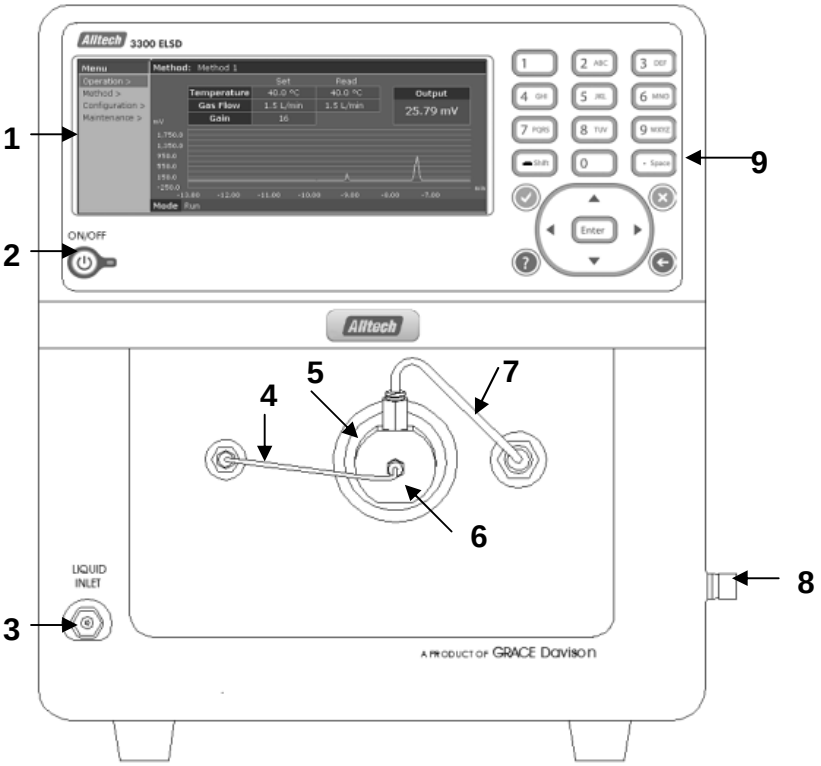


Figure 2.1: Front Panel of 3300 ELSD; Door Removed

2.3.1 FRONT PANEL (FIGURE 2.1)

1. **LCD (Liquid Crystal Display):** The LCD displays the software interface screens.

2. **Soft Power Button:** The Model 3300 ELSD can be powered on/off from the front panel of the unit using the soft power button. NOTE: The power button on the back panel must be set to the On position in order for the front panel button to be active.

3. **Liquid Inlet:** The column effluent tubing connects to the LIQUID INLET with a 1/16" male fingertight fitting.

4. **Nebulizer Liquid Tubing:** The stainless steel tubing carries the column effluent to the liquid inlet of the nebulizer.

5. **Impactor Cartridge:** The cartridge can be removed in order to access the drift tube for cleaning. Refer to **Section 5.3** for details.

6. **Nebulizer:** The column effluent and nitrogen gas are combined within the nebulizer to form an aerosol. The nebulizer is easily removed for cleaning by using a counterclockwise twisting motion.

7. **Nebulizer Gas Tubing:** The gas tubing carries nitrogen gas to the nebulizer gas inlet.

8. **Drain Port:** The DRAIN port is located on the right side panel. The drain tubing and clamp included with the unit are connected to the DRAIN port and then directed to a waste reservoir (not included) located at bench level if the unit is stacked or floor level. NOTE: The drain tubing must not be submerged in the liquid inside the reservoir during operation.

9. **Keypad:** The keypad is used to interact with the software interface screens.

10. **Removable Door (not shown):** The front door can be opened or removed to access the nebulizer and drift tube for cleaning. To remove the door, gently pull the door towards you.

2.3.2 BACK PANEL (FIGURE 2.2)

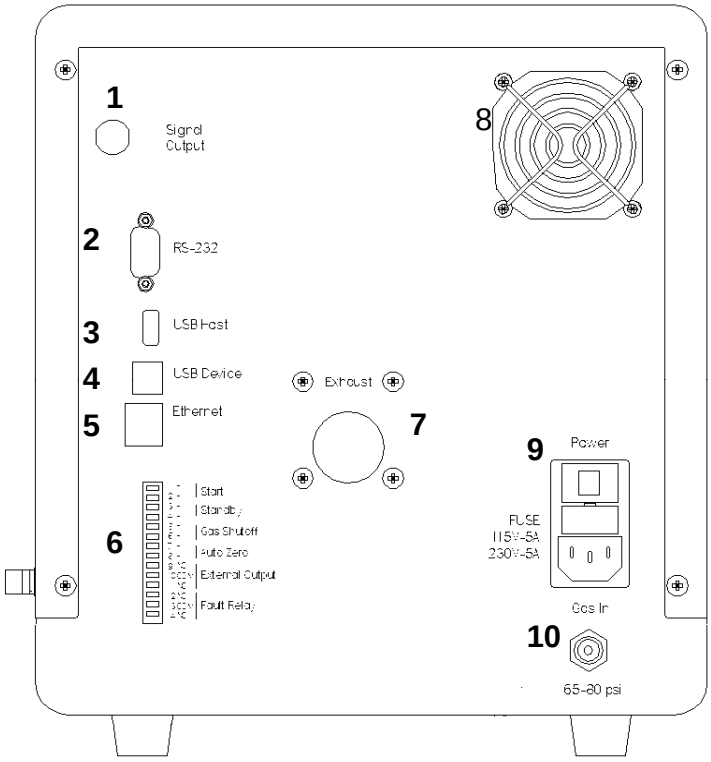


Figure 2.2: Model 3300 ELSD Back Panel

1. **Signal Output:** The signal cable included with the unit is connected to the SIGNAL OUTPUT port on the back panel of the unit and is used to send the analog signal to the data collection device.

2. **RS-232:** This port is currently reserved for service tech functions only.

3. **USB Host:** A pen drive (not included) can be inserted into this port for file importing/exporting and software upgrades. Also for possible future pc control capabilities.

4. **USB Device:** This port is currently reserved for service tech functions only.

5. **Ethernet:** This port can be used with the 3300 ELSD Driver for Agilent ChemStation. This port is also used for service tech functions.

6. **14-Pin Connector:** Outputs TTL/contact closure signals or accepts signals from peripheral equipment.

7. **Exhaust Outlet:** Nebulizer gas, mobile phase vapor, and solute mist or particles produced during an analysis will exit the detector through the EXHAUST outlet. The exhaust outlet accepts the exhaust adapter and exhaust tubing included with the unit, which must be then directed to a fume hood.

8. **Fan:** Provides cooling airflow through the instrument. Do not block.

9. **Power Module:** The power module contains a socket for the incoming power cord. It also contains the main power switch, which is used to turn the system power On/Off, and the line fuse. The module is autoselecting for 85-265V and contains a 5 Amp fuse.

10. **Gas Inlet:** The GAS INLET accepts the nebulizer gas supply tubing. The inlet features a quick-connect port, which requires no additional fittings. Gas pressure should be regulated from 65 – 80psig.

2.4 MAKING ELECTRICAL AND FLUID CONNECTIONS

1. **Unpacking the Unit:** Remove the ELSD from its shipping container and position it on a level surface near the column outlet of your HPLC system and the fume hood. Make sure there is free flow of air to the bottom of the ELSD and to the cooling fan at the back panel of the ELSD. Allow the detector to warm to ambient temperature if necessary. Save the shipping container for future use.

2. **Exhaust Adapter:** Screw in the threaded exhaust adapter provided in the accessory kit to the EXHAUST port on the back panel of the unit.

3. **Fill the Internal Drain Reservoir:** Place a beaker under the DRAIN port on the right side panel. Using a water bottle or pipette, add water into the EXHAUST port on the back panel of the unit until excess liquid is seen exiting the DRAIN port. Filling the internal drain reservoir prevents gas from escaping the drift tube.

4. **Power Connection:** Plug the power cord provided with the unit into the power module on the back panel of the detector. The module is autoselecting for 85-265V.

⚠CAUTION	**Only use the power supply cord recommended by the manufacturer.**

5. **14-Pin Connector:** Insert the 14-pin connector into the port on the back panel. Make the appropriate wiring connections to the 14-pin connector depending on which of the following functions are needed:

   Start: Pin 1: Ground (-) Pin 2: Signal (+)
   Pins 1 and 2 on the Model 3300 ELSD can accept a TTL/contact closure signal to start a method run on the detector.

   Standby: Pin 3: Ground (-) Pin 4: Signal (+)
   Pins 3 and 4 on the Model 3300 ELSD can accept a TTL/contact closure signal to put the detector into Standby mode.

   Gas Shutoff: Pin 5: Ground (-) Pin 6: Signal (+)
   Pins 5 and 6 on the Model 3300 ELSD can accept a TTL/contact closure signal to turn off gas flow at the end of a run. This signal is typically sent from an autosampler or a data collection system. Consult the appropriate manuals for wiring information.

   Autozero: Pin 7: Ground (-) Pin 8: Signal (+)
   Pins 7 and 8 on the Model 3300 ELSD can accept a TTL/contact closure signal from a start signal cable to autozero the detector. This signal is typically sent from an autosampler or a manual injection valve with a position-sensing switch. Consult the appropriate manuals for wiring information.

   External Output: Pin 9: Normally Open (NO) (+)
   Pin 10: Common (-)
   Pin 11: Normally Closed (NC) (+)

   Pins 9, 10, and 11 on the Model 3300 ELSD can output a TTL/contact closure signal to other instruments. Consult the appropriate manuals for wiring details.

   Fault Relay: Pin 12: Normally Open (NO) (+)
   Pin 13: Common (-)
   Pin 14: Normally Closed (NC) (+)

   Pins 12, 13, and 14 on the Model 3300 ELSD can output a TTL/contact closure signal to stop pump flow when an error occurs on the detector. Consult the appropriate manuals for wiring details.

6. **Gas Connection:** Connect the nitrogen gas supply tubing to the GAS INLET on the back panel. The nitrogen gas supply should be regulated from 65 – 80psig. A stable gas flow and pressure are necessary for reproducible results. The gas must be free of contaminants, such as oil, water, particulates, or any other non-volatile substances. A 0.1um gas filter is built into the instrument.

7. **Liquid Connection:** Connect the column effluent line to the LIQUID INLET with a 1/16" male fitting. The i.d. and length of the tubing between the column and the detector should be kept as small as possible to avoid band broadening. 0.005" i.d. tubing is recommended for best results. Tubing and fittings are included in the ELSD 3300 accessory kit.

8. **Drain Setup:** Attach the drain tubing and clamp included with the detector to the DRAIN OUTLET on the right side panel. Extend the tubing to a drain waste container (not included) either at bench level if the detector is stacked or at floor level. Make sure the container is sealed to prevent solvent fumes from escaping. Monitor the liquid level in the container during operation and decant excess liquid when the level approaches the top of the container.

⚠CAUTION	**Do NOT allow the tubing to become submerged in the liquid inside the container. Remember that the waste container will contain solvents from your mobile phase and should be disposed of properly.**

9. **Signal Output:** Connect the signal cable provided with the unit to the SIGNAL OUTPUT port on the back panel of the detector and connect to your data collection device.

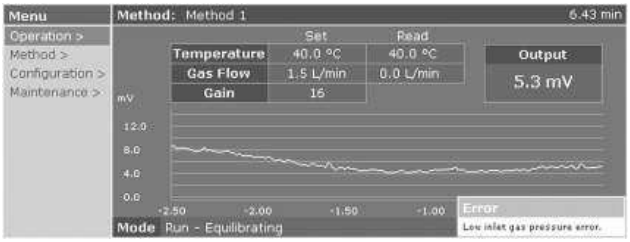
10. **Exhaust Tubing:** Connect the exhaust tubing included with the unit to the EXHAUST outlet on the rear panel of the unit. Extend the tubing to the hood/ventilation system. There should be no low spots in the tubing where condensate can collect.

NOTICE
Make sure the exhaust system provides adequate but not excessive suction. Excessive suction can cause a noisy baseline.

3. NAVIGATING THE SOFTWARE INTERFACE

The Model 3300 software interface features a collapsible menu located in the upper left corner of the LCD screen. The following sections describe the software menu options in detail. The Help Button, "?" can be pressed in many software screens in order to access Help Screens which offer assistance with using the software functions.

3.1 MAIN SCREEN



The Operation screen is the main screen displayed during use of the instrument. This screen provides the following information for the currently loaded method:

- **Method Name:** Currently loaded method name.
- **Temperature:** Set point and read value of the drift tube temperature in °C. The temperature range is from 25.0 to 120.0°C. NOTE: There are two heated zones in the drift tube, a lower zone and an upper zone. The value displayed on the main screen is the average value for the two zones.
- **Gas Flow:** Set point and read value of the nebulizer gas flow rate in L/min. The gas flow range is from 0.0 to 4.0L/min.
- **Gain:** Current gain setting. Possible gain values are 1, 2, 4, 8, and 16. A gain setting of 1 produces an unamplified signal, and each increase in gain setting produces twofold signal amplification over the previous setting.
- **Signal Output:** The signal output in mV is displayed when the instrument is in Run or Cleaning modes. The output is not displayed in Standby or Heating modes. If the output value exceeds 2.5V, the signal output will read 'High'. If output drops below -500mV, the signal output will read 'Low'.
- **Method Run Time:** The elapsed time for the method run is displayed in the upper right corner if a method run has been started. The time value will remain until the method run has been stopped.
- **Chart:** The chromatogram trace of up to 60 minutes will be active for Run and Cleaning modes.
- **Mode:** The current mode of the detector (Standby, Run, Heating, or Cleaning).
- **Equilibrating Indicator:** If the detector is in Run mode and the heaters and gas flow have not reached their set points, the screen will display "Equilibrating" next to the Mode status until the equilibration requirements are met. The equilibration requirements are: lower drift tube, upper drift tube, and optics block must be within 1.5C of their set points; gas flow must be within 0.3L/min of its set point; the temperatures and gas flow must meet the requirements for 60 seconds before the message disappears.
- **Timed Mode Change Reminder (not shown):** If a timed mode change has been scheduled, a reminder for the event will be displayed next to the Mode status.
- **Errors:** Any errors that occur on the instrument will be displayed in the lower right corner of the screen.

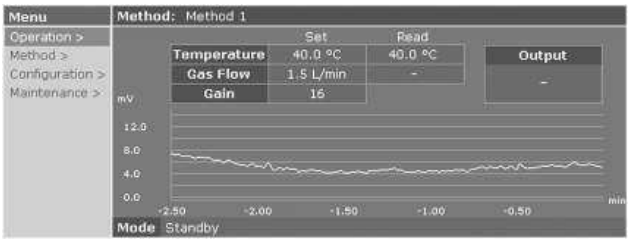
The software Menu is located in the upper left corner of the screen. There are four main Menu headers: Operation, Method, Configuration, and Maintenance. The following sections describe the software functions within these headers.

3.2 OPERATION

3.2.1 MODE

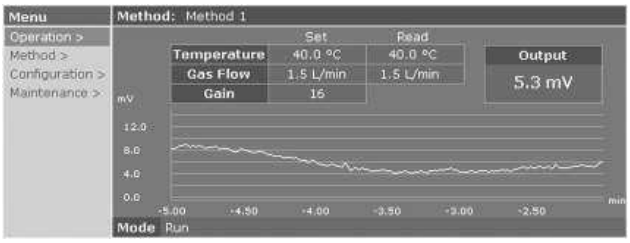
The Model 3300 ELSD has four operational modes: Standby, Run, Heating, and Cleaning.

STANDBY MODE



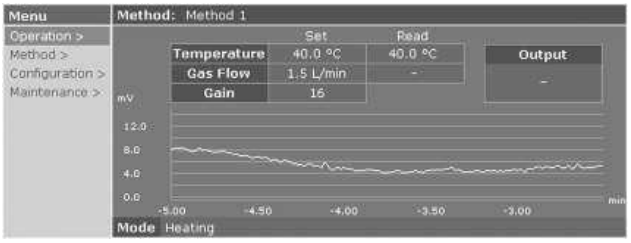
Select Operation/Mode/Standby and press Enter to put the detector into Standby mode. In Standby mode, the heaters, gas flow, and laser are off. The signal output is not displayed, and the chromatogram trace is not active. The detector enters Standby mode after power-up with the last saved method conditions.

RUN MODE



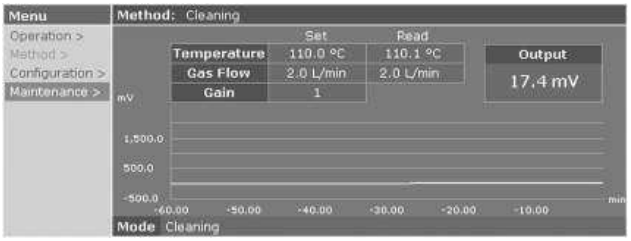
Select Operation/Mode/Run and press Enter to put the detector into Run mode. In Run mode, the heaters, gas flow, and laser are on. The signal output is displayed and the chromatogram trace is active. Run mode is used for sample analysis.

HEATING MODE



Select Operation/Mode/Heating and press Enter to put the detector into Heating mode. In Heating mode, the heaters are on, but the gas flow and laser are off. The signal output is not displayed and the chromatogram trace is not active. Heating mode keeps the detector in a ready state, so the user won't have to wait for temperature equilibration when switching to Run mode for sample analysis.

CLEANING MODE



Select Operation/Mode/Cleaning and press Enter to put the detector into Cleaning mode. In Cleaning mode, the heaters, gas flow, and laser are on. Temperature is set to 110°C, gas flow is set to 2.0L/min, and Gain is set to 1. The detector should be run with 100% water or other suitable solvent at 1.0mL/min for at least one hour to clean the detector. Cleaning mode can be used for routine cleaning of the detector to prevent any buildup of sample materials inside the drift tube and optics. A dirty drift tube and optics can cause excess baseline noise.

3.2.2 START/STOP RUN

Select Operation/Start Run and press Enter to manually start a method run. The method elapsed time will then appear in the upper right corner of the screen above the output, and the timed events programmed in the method will start. This function should be used only if timed events will be used with the method. Select Operation/Stop Run and press Enter to manually stop the method run. Refer to **Section 3.3.1** for more information on programming timed method events. Alternately, method timed events can be started by connecting the proper wiring to the Start input pins on the back panel of the unit. Refer to **Section 2.4** for details.

3.2.3 LOCK/UNLOCK DETECTOR

The Lock Detector option can be used to lock the method settings on the detector to prevent unwanted method changes during operation.

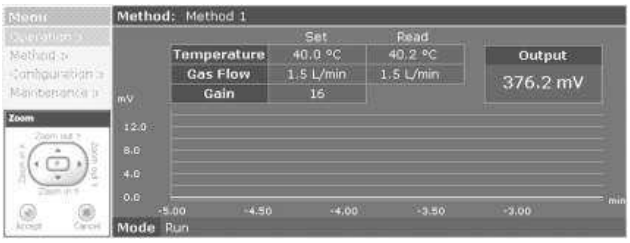
Select Operation/Lock Detector and press Enter to reach the Lock Detector screen. Enter and then re-enter the PIN using the keypad. Press Accept to lock the detector, or press Cancel to cancel locking the detector.

To unlock the detector, select Operation/Unlock Detector and press Enter to bring up the Unlock Detector screen. Enter the PIN created when the detector was locked to unlock the detector. The detector can also be unlocked by recycling the power on the unit.

3.2.4 CHART

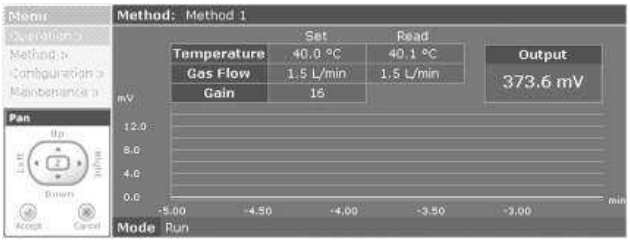
The Chart displays a scalable chromatogram trace of up to 60 minutes.

ZOOM



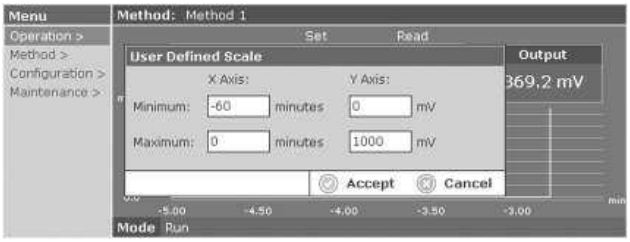
The Zoom function can be used to zoom in/out on the chromatogram trace. Select Operation/Chart/Zoom and press Enter, and the zoom tool will appear in the lower left corner of the screen. Use the up arrow to Zoom out the Y-axis; use the down arrow to Zoom in the Y-axis; use the left arrow to zoom in the X-axis; or use the right arrow to zoom out the x-axis. Once the desired chart view has been achieved, press Accept to save the new chart view, or press Cancel to return to the original chart view. The x-axis range is –60 to 0 minutes. The y-axis range is –500mV to 2.5V. To reach Pan directly from Zoom, press Enter.

PAN



The Pan function can be used to pan left, right, up, or down on the chromatogram trace. Select Operation/Chart/Pan and press Enter and the Pan tool will appear in the lower left corner of the screen. Use the up arrow to Pan up the Chart; use the down arrow to Pan down the Chart; use the left arrow to Pan left on the Chart; or use the right arrow to Pan right on the Chart. Once the desired chart view has been achieved, press Accept to save the new chart view, or press Cancel to return to the original chart view. The x-axis range is –60 to 0 minutes. The y-axis range is –500mV to 2.5V. To go directly to Zoom from Pan, press Enter.

USER DEFINED SCALE



The User Defined Scale screen can be used to enter user defined x-axis and y-axis values for the chart scaling. Select Operation/Chart/User Defined Scale and press Enter to reach the User Defined Scale screen. Enter the minimum and maximum x-axis and y-axis values in the window that appears. The x-axis range is –60 to 0 minutes. The y-axis range is –500mV to 2.5V. Once the new chart values have been entered, press Accept to save the new chart settings or press Cancel to return to the original settings.

ENABLE/DISABLE Y-AXIS AUTOSCALE

The Enable Y-Axis Autoscale function causes the chart to automatically scale to the highest peak and ensures that the chromatogram trace will always be on-scale. Select Operation/Chart/Enable Y-Axis Autoscale and press Enter to enable this feature. Select Operation/Chart/Disable Y-Axis Autoscale and press Enter to disable this feature.

RESET SCALE

The Reset Scale function will zoom out the chart view to its maximum x-axis and y-axis values: -60 to 0 min; -500mV to 2.5V. Select Operation/Chart/Reset Scale and press Enter to reset the chart axes.

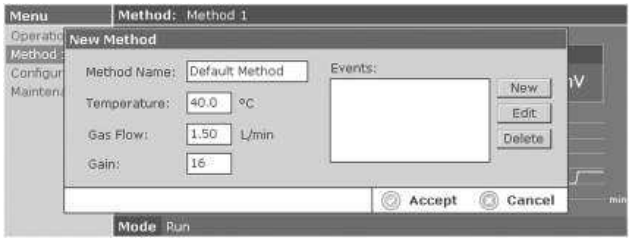
3.2.5 AUTOZERO

The Autozero function can be used to zero the output on the detector. To autozero, select Operation/Autozero and press Enter using the up/down arrows on the keypad. The autozero voltage will depend on the full-scale voltage setting on the detector. For a 1V full-scale voltage setting, the default Autozero value is 5mV. For a 10mV full-scale voltage setting, the default Autozero value is 0mV. If the Custom Autozero Output has been enabled, the detector will be autozeroed to the value selected, from 0 to 100mV. Refer to **Section 3.3.2** for more details on setting the Custom Autozero Output value.

3.3 METHOD

Methods are created to set the temperature, gas flow, gain, and event settings on the detector.

3.3.1 NEW



Select Method/New and press Enter to bring up the New Method window. Enter the Method Name, Temperature, Gas Flow, Gain, and Timed Events using the keypad. Use the up/down arrows to tab through the method parameters.

Method Name: The Method Name may include the following characters: A - Z, a - z, 1 - 9, <space>, or . (period). Rapidly press a button on the keypad to cycle between letter and number options. For upper case, press Shift and then enter the letter. The method name may contain up to 80 characters; only 25 will be displayed in the main operation screen due to space limitations.

Temperature: Enter a temperature value between 25 -120°C using the keypad.

Gas Flow: Enter a value between 0.0 – 4.0L/min using the keypad.

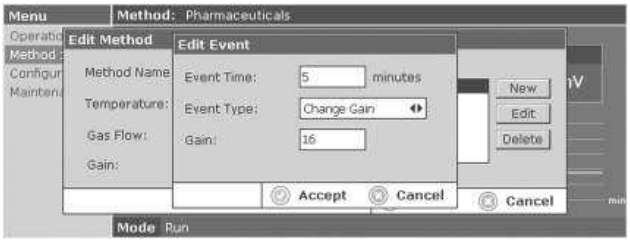
Gain: Enter a value of 1, 2, 4, 8, or 16 using the keypad. Each increase in gain setting will produce twofold signal amplification over the previous setting.

Events: Events can be added to the method in order to program timed gain changes or timed external output events. Timed gain changes can be used so sample compounds of widely varying concentration can be on-scale in the same chromatogram. Timed external output events can be used to trigger events on other instruments.

New Event

To add a new timed event, use the up/down arrows to highlight the New button and press Enter. The New Event window will then appear. Enter the Event Time, from 0 – 60 minutes. Enter the Event Type (Change Gain or Set Control Relay). If Change Gain has been selected, then enter the Gain value: 1, 2, 4, 8, or 16. If Set Control Relay has been selected, select On or Off for the External Contact. Once the new event has been created, press Accept to save the new event, or press Cancel to cancel the new event.

Edit Event

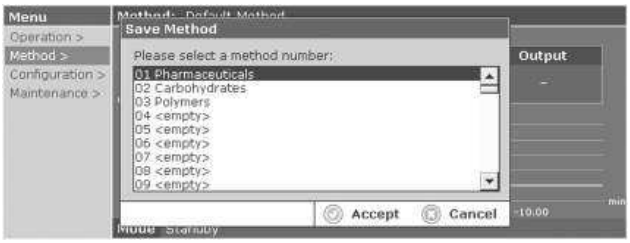


To Edit an existing event, use the up/down arrows to highlight the event to be edited from the table. Then use the right arrow to select Edit and press Enter. The Edit Event table will then appear, and any changes to the Event Time, Event Type, Gain, or External Contact can be made. Press Accept to save the event changes, or press Cancel to cancel the changes.

Delete Event

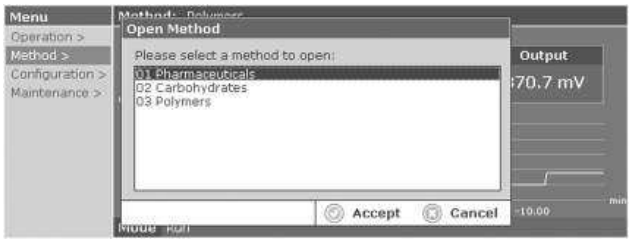
To Delete an event, use the up/down arrows to highlight the event to be deleted from the table. Then use the right arrow and the down arrow to select Delete, and press Enter. The event will then be deleted.

Once all method parameters have been entered, press Accept to save the new method. A Save Method window will then appear requesting a method number to be selected.



Use the up/down arrows or the keypad to select a method number for the new method. Press Accept to save the new method, or press Cancel to cancel the new method.

3.3.2 OPEN

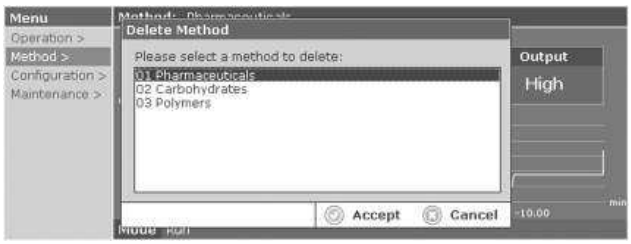


Select Method/Open and press Enter to open an existing method. The Open Method window will then appear. Use the up/down arrows or keypad to select a method from the list. Press Accept and the method will then load on the main screen, or press Cancel to cancel loading the method.

3.3.3 EDIT

To Edit an existing method, select Method/Edit and press Enter. The Edit Method window will then appear. Use the up/down arrows to tab through the method parameters and enter a new Method Name, Temperature, Gas Flow, Gain, and/or Events using the keypad. Press Accept to save the new method settings, or press Cancel to return to the original method settings.

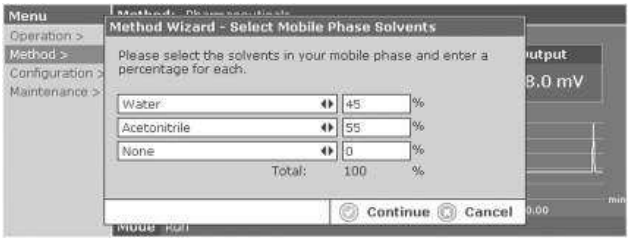
3.3.4 DELETE



To Delete a method, select Method/Delete and press Enter. The Delete Method window will then appear. Select a method to delete from the list using the up/down arrows or by entering/cycling a number. Press Accept to delete the method, or press Cancel to cancel deleting the method.

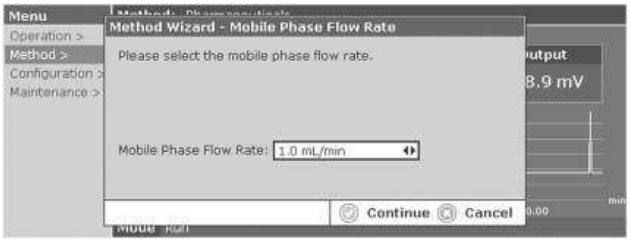
3.3.5 WIZARD

The Method Wizard can help provide initial detector settings based on the mobile phase solvent composition, mobile phase flow rate, and sample concentration. Select Method/Wizard and press Enter, and the first Method Wizard screen will appear:



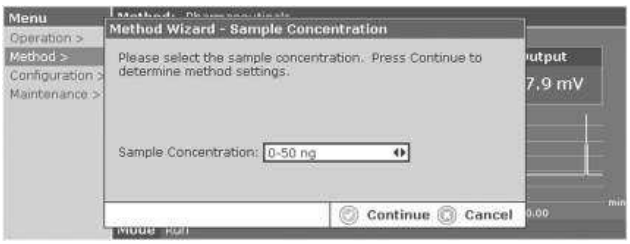
Select Mobile Phase Solvents

Use the left/right arrow keys to select from the list of solvents. Use the up/down arrows to tab down the table. Enter a percentage for each solvent. The total percentage must add up to 100%. Once the solvents/percentages have been entered, press Continue to continue on to the next screen in the Method Wizard, or press Cancel to cancel using the Method Wizard.



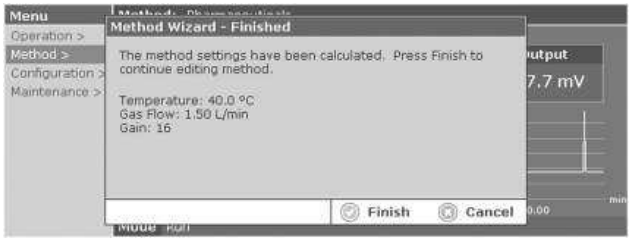
Mobile Phase Flow Rate

Select the Mobile Phase flow rate using the left/right arrows. Possible choices are 0.2, 0.5, 1.0, 2.0, and 3.0mL/min. Press Continue to continue on to the next Method Wizard screen, or press Cancel to cancel the Method Wizard.



Sample Concentration

Use the left/right arrows to select the sample concentration from the list. Press Continue to reach the next Method Wizard screen, or press Cancel to cancel using the Method Wizard.

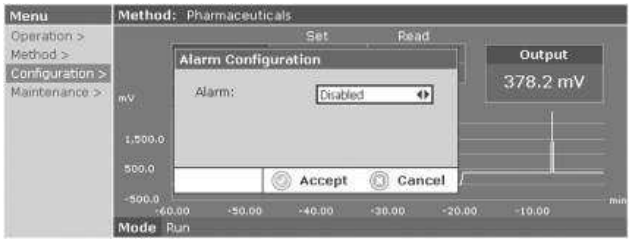


Finished

The final screen in the Method Wizard provides the recommended Temperature, Gas Flow, and Gain settings based on the mobile phase solvent composition, mobile phase flow rate, and sample concentration. Press Finish to continue creating the method, and the New Method window will appear, or press Cancel to cancel the Method Wizard.

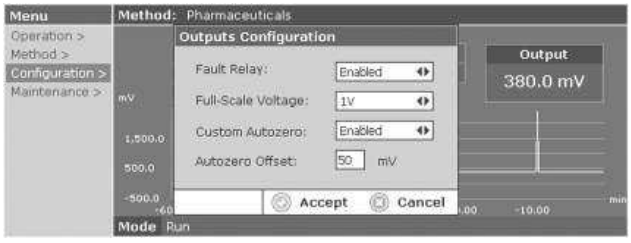
3.4 CONFIGURATION

3.4.1 ALARM



The Alarm function can be used to sound an alarm when errors occur on the detector. Select Configuration/Alarm and press Enter to bring up the Alarm Configuration window. Use the left/right arrows to select Enabled or Disabled. If Enabled, the alarm will sound when errors occur on the unit and will continue to sound until the errors are remedied. If Disabled, the alarm will not sound when errors occur on the unit. Press Accept to save the alarm configuration, or press Cancel to return to the original alarm configuration.

3.4.2 OUTPUTS



Select Configuration/Outputs and press Enter to configure the outputs on the unit.

Fault Relay

Use the left/right arrow keys to select Enabled or Disabled for the Fault Relay setting. If Enabled, the detector will output a signal that can be connected to a pump to stop the pump if errors occur on the unit. The proper wiring must be connected for the Fault Relay to work. If Disabled, errors will not trigger an output to a pump for pump shutdown. Refer to **Section 2.4** for wiring details.

Full-Scale Voltage

Use the left/right arrows to select 10mV or 1V for the full-scale voltage, depending on your data collection system.

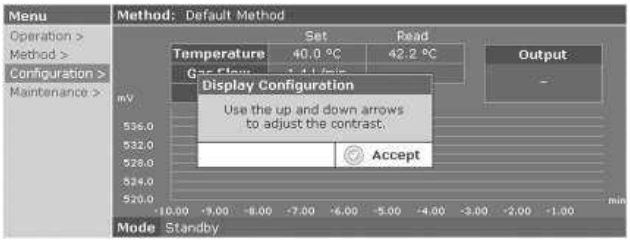
Custom Autozero

Use the left/right arrows to select Enabled or Disabled for the Custom Autozero. If Enabled, the user can select an Autozero Offset, from 0 – 100mV. If Disabled, the Autozero will be based on the full-scale voltage setting on the detector: 5mV for 1V FS; 0mV for 10mV FS.

Autozero Offset

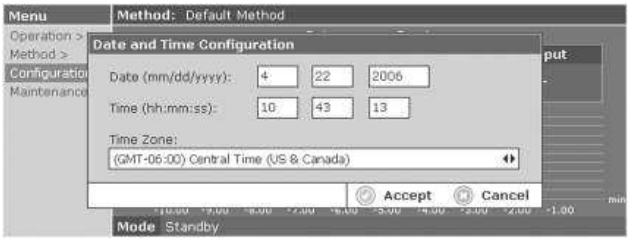
If the Custom Autozero has been enabled, enter a value from 0 – 100mV for the Autozero offset. This is the signal output that will be displayed when the detector is autozeroed. If the Custom Autozero has been disabled, then the Autozero Offset will be grayed out. Press Accept to save the changes to the Outputs Configuration, or press Cancel to cancel the changes.

3.4.3 DISPLAY



Select Configuration/Display and press Enter to bring up the Display Configuration screen. Use the up/down arrows to adjust the Display Contrast. The up arrow lightens the display, and the down arrow darkens the display. Press Accept to save the new Display settings.

3.4.4 DATE AND TIME



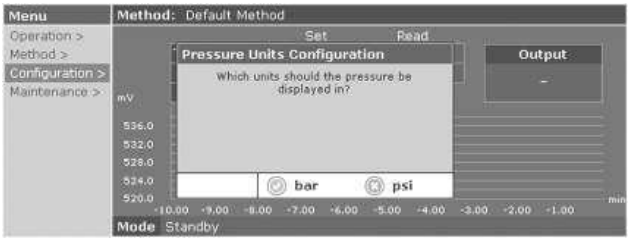
Select Configuration/Date and Time and press Enter to bring up the Date and Time Configuration screen. Enter the Date (month, day, and year) and the Time (hour, minute, and second) using the keypad. Use the up/down arrows to tab across the entry blanks. Select the Time Zone from the list using the left/right arrows. Press Accept to save the changes, or press Cancel to cancel the changes.

3.4.5 LANGUAGE

The Model 3300 ELSD can be configured for the following languages: English, Spanish, French, German, Italian, Korean, Chinese (Simplified), and Japanese. The unit must have the correct software versions installed in order to be compatible with certain languages; contact BUCHI for details. Select Configuration/Language and press Enter to bring up the Language Configuration screen. Use the up/down arrows to select a language from the list. Press Accept to save the new language configuration, or press Cancel to cancel the new language configuration.

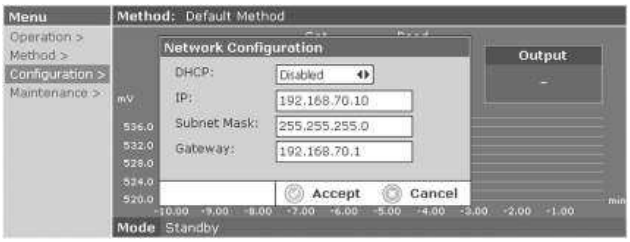
The unit must be restarted before the language change can take effect. Select Yes to restart the device and change the language, or select No and the new language will take effect the next time the unit is rebooted.

3.4.6 PRESSURE UNITS



Select Configuration/Pressure Units and press Enter to configure the pressure units on the detector. Select either Bar or Psi.

3.4.7 NETWORK

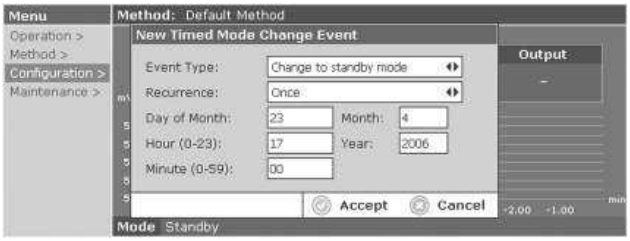


Select Configuration/Network and press Enter to bring up the Network Configuration window. Select DHCP Enabled or Disabled. If Disabled is selected, enter the IP address, Subnet Mask, and Gateway settings. If Enabled is selected, the IP address, Subnet Mask, and Gateway will be grayed out. Press Accept to save the new settings, or press Cancel to cancel the new settings.

3.4.8 TIMED MODE CHANGES

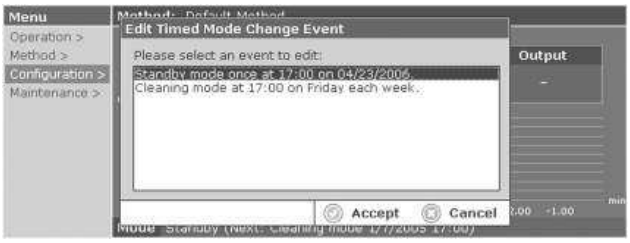
Timed Mode Change Events can be used to put the detector into another operation mode using a timer. This can be convenient to schedule routine cleaning, heating, etc. events. A reminder for the next Timed Mode Change will be displayed on the main screen next to the Mode.

NEW TIMED MODE CHANGE



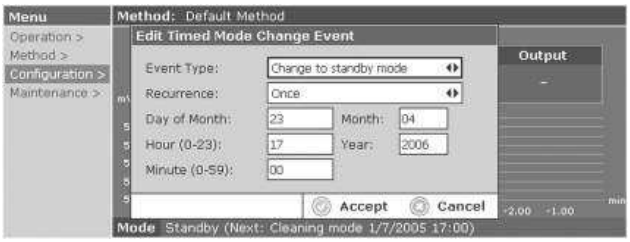
Select Configuration/Timed Mode Changes/New and press Enter to bring up the New Timed Mode Change Event window. Select an Event Type from the list using the left/right arrows keys: Change to Standby mode, Change to Heating mode, Change to Run mode, or Change to Cleaning mode. Select the Recurrence using the left/right arrow keys: Once, Weekly, or Monthly. For a Recurrence of Once, enter the Day of Month, Month, Hour, Year, and Minute. Use the up/down arrows to tab through the entry blanks. For Weekly Recurrence, enter the Day of Week, Hour, and Minute. For Monthly Recurrence, enter the Day of Month, Hour, and Minute. Press Accept to save the new event settings, or press Cancel to cancel the new event settings.

EDIT TIMED MODE CHANGE



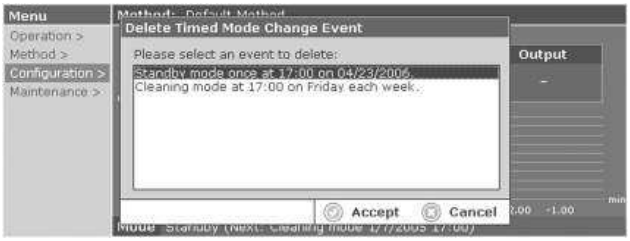
To Edit a Timed Mode Change Event, select Configuration/Timed Mode Changes/Edit and press Enter.

Use the up/down arrows to select an event from the table. Press Accept to select the event to be edited, or press Cancel to cancel the editing.



If Accept has been selected, the Edit Timed Mode Change Event window will appear. Make the desired changes to the Timed Mode Change Event. Press Accept to save the changes, or press Cancel to cancel the changes.

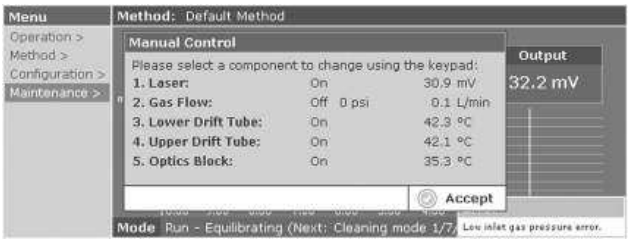
DELETE TIMED MODE CHANGE



Select Maintenance/Timed Mode Changes/Delete and press Enter to delete a timed mode change event. Use the up/down arrows to highlight the event to be deleted from the table. Once the event has been highlighted, press Accept to delete the selected event; or press Cancel to cancel deleting the event.

3.5 MAINTENANCE

3.5.1 MANUAL CONTROL



Select Maintenance/Manual Control and press Enter to reach the Manual Control screen.

Laser: On/Off
Press 1 to toggle the Laser On or Off. The current signal output value is also displayed in mV.

Gas Flow: On/Off
Press 2 to toggle the Gas Flow On or Off. The current gas flow rate and nebulizer gas pressure are also displayed.

Lower Drift Tube: On/Off
Press 3 to toggle the Lower Drift Tube heater On or Off. The current Lower Drift Tube Temperature value is also displayed.

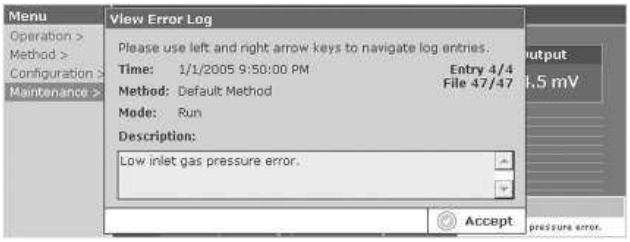
Upper Drift Tube: On/Off
Press 4 to toggle the Upper Drift Tube heater On or Off. The current Upper Drift Tube Temperature is also displayed.

Optics Block: On/Off
Press 5 to toggle the Optics Block heater On or Off. The current Optics Block temperature is also displayed. The Optics Block heats to 90% of the drift tube temperature set point.

Press Accept to return to the main screen, and the detector will return to its original settings.

3.5.2 LOGS

VIEW ERROR LOG



Select Maintenance/Logs/View Error Log and press Enter to access the Error Logs on the unit. Each error log entry contains the Time, Method, Mode, and Description of the error.

3.5.3 TESTS

Diagnostic tests can be run to help troubleshoot the unit. The available tests are:

- Nebulizer Gas Pressure Test
- Gas Flow Test
- Optics Test
- Optics Heating Test

Select Maintenance/Tests/Nebulizer Gas Pressure Test, Gas Flow Test, Optics Test, or Optics Heating Test and press Enter to run a test. Refer to **Section 6.2** for details on these tests.

3.5.4 FILES

Files can be imported or exported from the unit using a pen drive inserted into the USB Host port on the back panel of the unit. NOTE: The pen drive must be 128MB or less; pen drives with higher memory are NOT compatible with the 3300 ELSD.

EXPORT

To Export files from the detector, insert a pen drive into the USB Host port on the back panel of the unit. Select Maintenance/Files/Export/Configuration, Methods, or Error Log and press Enter. The selected files will be exported to the pen drive, and the pen drive containing the files can then be removed.

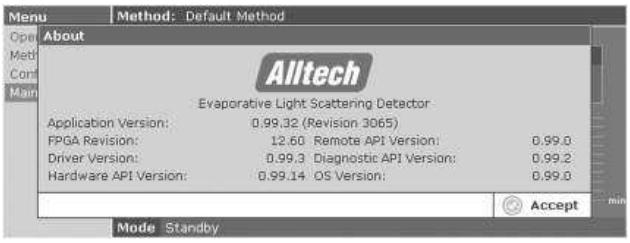
IMPORT

To Import files to the detector, insert a pen drive containing the files into the USB Host port on the back panel of the unit. Select Maintenance/Files/Import/Configuration or Methods and press Enter. The selected files will be imported from the pen drive, and the pen drive can then be removed.

3.5.5 SERVICE TECH

Service Tech functions are reserved for qualified personnel only.

3.5.6 ABOUT



Select Maintenance/About and press Enter to display the current software versions on the unit. Press Accept to return to the main screen.

4. ROUTINE OPERATION

4.1 SAFETY

Please use the following guidelines to help ensure safe operation of the Model 3300 ELSD:

⚠ DANGER

1. **Be sure to provide proper ventilation for all solvent vapors.**
2. **Use a fume hood or other ventilation device to prevent the inhalation of any solvent fumes expelled through the exhaust tube.**
3. **Avoid open flames and sparks when using flammable solvents.**
4. **Always power off before removing the cover.**
5. **Laser Radiation – Avoid exposure to beam – Class IIIB laser product.**

CLASS 1 LASER PRODUCT

Contains a class 3B 30mW-650nm laser. Designed to comply with 21CFR 1040.10, 1040.11, and IEC60825 with amendments as of date of manufacture.

No user serviceable components inside. Refer servicing to qualified personnel.

4.2 OPERATING NOTES

1. The internal drain reservoir must be filled with liquid (water) prior to using the detector. The reservoir can be filled through the EXHAUST port on the back panel. Once water is visibly draining from the DRAIN port on the right side panel, the internal drain reservoir has been filled.
2. Monitor the liquid level in the drain waste container and remove excess liquid when necessary. Never let the end of the drain tubing become submerged in the liquid inside the container.
3. The mobile phase should not be flowing when the drift tube is not at proper vaporization temperature or when the nebulizer gas is turned off.
4. Only volatile buffers are allowed in the mobile phase. Non-volatile buffer particles will be viewed as sample by the detector, causing unwanted baseline noise. Refer to **Section 7.4**, Volatile Mobile Phase Modifiers, for a list of suitable buffers.

4.3 SELECTING INITIAL OPERATING CONDITIONS

Use the recommendations provided by the Method Wizard for initial method settings. The Method Wizard can be reached by selecting Method/Wizard in the software interface. Refer to **Section 3.3.5** for details.

Semi-Volatile Compounds

The recommendations provided by the Method Wizard are based on non-volatile sample compounds. If the sample compound is semi-volatile, it may require a lower drift tube temperature than the Method Wizard recommends. Some experimentation may be necessary.

Gradient Separations

Choose operating conditions based on the least volatile portion of the mobile phase when performing gradient separations.

Unlisted Solvents

For any solvents not listed in the Method Wizard, please refer to the solvent's boiling point and vapor pressure in a reference book such as the Merck Index or Handbook of Chemistry and Physics. Use the temperature and gas flow rate for the solvent that most closely matches the boiling point and vapor pressure of the solvent of interest.

4.4 STARTUP SEQUENCE

1. Set up the unit as described in **Section 2.4**, Making Electrical and Fluid Connections.
2. Turn on the nebulizer gas supply. Set the regulator between 65 and 80psig.
3. Power up the Model 3300 ELSD.
4. When the Operation screen appears, set up the desired method and configuration as indicated in **Sections 3.3 and 3.4**.
5. Put the detector into Run mode (Operation/Mode/Run).
6. Allow the detector to equilibrate. The Equilibrating indicator will appear next to the Mode in the lower left corner until the Lower Drift Tube heater, Upper Drift Tube heater, Optics Block heater, and Gas Flow reach their set points.
7. Once the detector has equilibrated, record a gas-only baseline for 10-15 minutes. Observe the signal output displayed on the front panel and on the chromatogram. You should get a stable, low-noise baseline. The noise should be in the millivolt range, within 2mV.
8. If the baseline is unstable and/or drifting, the unit may need longer equilibration time.
9. If the noise is higher than expected, you may want to perform the optics test to determine if there is a possible laser or electronics problem. Refer to **Section 6.2.3** for details on the optics test.

10. Flush the column with mobile phase before connecting it to the detector. The length of purging will depend on how long the column was in storage and what type of sample and mobile phase were used. It is very important that the column is free from silica “fines” or other contaminants before connecting to the ELSD. The Model 3300 ELSD will detect the contaminants with great sensitivity.
11. After flushing the column, connect it to the LIQUID INLET on the left side of the front panel on the detector. The tubing between the column and the nebulizer should be a short as possible. 0.005” i.d. inlet tubing is recommended.
12. Turn the pump on to the desired flow rate. Do not exceed the recommended maximum solvent rates.
13. Check connections for leaks and tighten the fittings, if necessary.
14. Observe the signal output on the display and the recorded baseline. After an initial large rise when the pump is turned on, the signal level should drop close to the “gas only” level after several minutes. If it remains high after sufficient equilibration time, the mobile phase may be contaminated (column fines, buffers, etc.), or the temperature or gas flow settings may be too low for optimal evaporation of the mobile phase. Take corrective measures and allow the system to re-equilibrate for a few minutes.
15. The detector is ready for sample analysis. The first time the Model 3300 ELSD is used, you may want to reproduce the Model 3300 ELSD QC procedure listed in the performance documents sent with the unit.

4.5 SHUTDOWN SEQUENCE

1. Turn off the mobile phase flow.
2. Allow gas only to flow for approximately five minutes to clear any remaining droplets.
3. Put the detector in Standby mode (Operation/Mode/Standby).
4. Turn off the gas supply at the source, if required.
5. Power off the ELSD using either the soft power button on the front panel, or the main power switch on the rear panel.

NOTICE

If the ELSD will not be used for several days, disconnect the column from the nebulizer inlet and plug the inlet. Flush the column before reconnection.

Instrument power may be left on when the instrument is not in use. The laser may be turned by putting the detector into Standby or Heating mode.

4.6 OPTIMIZATION PROCEDURE

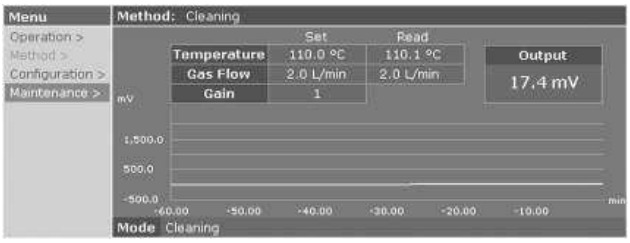
1. Set the drift tube temperature, gas flow, and gain to the initial settings recommended in **Section 4.3**.
2. Start mobile phase flow and allow the system to equilibrate.
3. Increase the drift tube temperature by 1°C increments if necessary until adequate evaporation of the mobile phase is achieved (indicated by a stable baseline).
4. Inject the sample and obtain peak areas for each of the components. Choose a sample concentration that shows a peak that is on-scale with baseline noise.
5. Change the gas flow rate in 0.2L/min increments from the recommended set point and observe the change in peak area with each change.
6. The optimal gas flow rate will produce the largest peaks with the lowest amount of baseline noise. Plot the signal-to-noise ratio vs. peak area to help in identifying the optimal gas flow rate.

5. MAINTENANCE

5.1 CLEANING MODE

Cleaning mode can be used for routine cleaning of the detector to prevent any buildup of sample materials inside the drift tube and optics. A dirty drift tube and optics can cause excess baseline noise.

To put the detector into Cleaning mode, select Operation/Mode/Cleaning and press Enter. In Cleaning mode, the heaters, gas flow, and laser are on. Temperature is set to 110°C, Gas Flow is set to 2.0L/min, and Gain is set to 1. The detector should be run with 100% water or other suitable solvent at 1.0mL/min for at least one hour to clean the detector. The detector will remain in Cleaning mode until the detector is switched to another mode.



5.2 NEBULIZER CLEANING PROCEDURE

The nebulizer can become blocked over time with sample and mobile phase materials. A dirty or blocked nebulizer can cause increased baseline noise and decreased sensitivity. The following procedure can be used to clean the nebulizer.

Materials Needed:

- *Open-end wrench, 1/4" x 5/16"*
- *HPLC-grade water*
- *HPLC-grade 50:50 methanol:water solution*
- *Sonication bath*

1. Power off the unit from the back panel and disconnect the power cord.
2. Remove the front door by gently pulling it towards you from the handle and set aside.
3. Disconnect the stainless steel liquid inlet line from the nebulizer using the 1/4” wrench.
4. Disconnect the quick-connect gas tubing from the back of the front panel. Push in, then pull out to remove the tubing.
5. Remove the nebulizer from the drift tube by turning it firmly counterclockwise.
6. Remove the orange wear band from the nebulizer and set aside.
7. Disconnect the gas inlet (with gas tubing attached) from the nebulizer using the 5/16” wrench and set aside.
8. Place the nebulizer in a beaker filled with 50:50 methanol:water solution.
9. Sonicate the nebulizer in an ultrasonic bath for 10 minutes.

NOTICE

Do NOT sonicate the wear band or nebulizer gas inlet.

10. If the nebulizer is still completely blocked, connect a high-pressure air line to the nebulizer inlet to help remove the blockage.
11. If the nebulizer is permanently blocked or cannot be cleaned, the nebulizer should be replaced.
12. Replace the nebulizer wear band and nebulizer gas inlet.
13. Replace the nebulizer back into the unit by aligning the grooves and turning clockwise until the nebulizer locks firmly into place.
14. Replace the liquid and gas inlet lines to the nebulizer.
15. Replace the front door.

5.3 DRIFT TUBE CLEANING PROCEDURE

The drift tube can become dirty over time from sample and mobile phase materials. A dirty drift tube can cause increased baseline noise and decreased sensitivity. The following procedure can be used to clean the drift tube.

Materials Needed:

- *Open-end wrench, 1/4" x 5/16"*
- *Hex ball driver, 3/32"*
- *Drift tube cleaning brush*
- *HPLC-grade cleaning solvent (water or other suitable solvent)*

NOTICE

Use only the drift tube cleaning brush included with the unit. This brush has been specially selected so it will not travel too far down the drift tube, which could splash the optics and result in additional noise. Do not attempt to force the brush further into the drift tube than it was designed to go.

1. Power off the unit from the power switch on the back panel and unplug the power cord from the unit.
2. Allow the detector to cool for at least 30 minutes.
3. Remove the nebulizer if it has not already been removed. Refer to **Section 5.2**, Steps 2 – 5 for instructions.
4. Use the hex ball driver to remove the two screws on the removable cartridge on the front panel of the unit. Remove the cartridge and set aside.
5. Using water or other suitable solvents, wet the drift tube cleaning brush provided in the accessory kit and carefully insert the brush into the drift tube from the front panel. NOTE: Use only the brush provided with the unit.
6. Carefully clean the sides of the drift tube with the wire brush, loosening any particles that adhere to the drift tube.
7. Once the drift tube has been cleaned, reinsert the impactor cartridge and tighten the screws. Make sure the drain hole is located on the bottom of the tube as it is inserted.

NOTICE

The drain hole located on the bottom of the impactor cartridge must be aligned with the drain hole inside the unit when the cartridge is reinserted. Otherwise, flooding could occur inside the unit.

8. Replace the nebulizer and reattach the liquid and gas tubing. Refer to **Section 5.2**, Steps 13 – 15.

5.4 OPTICS CLEANING PROCEDURE

The optics can become dirty over time from sample and mobile phase materials. Dirty optics can cause increased baseline noise and decreased sensitivity. The following procedure can be used to clean the optics.

⚠ DANGER **Use of controls, adjustments, or performance of procedures other than those specified herein may result in hazardous exposure to laser light.**

⚠ DANGER **Class 3B laser radiation when optics are open and interlocks defeated. Avoid exposure to beam.**

Materials needed:

- *Phillips screwdriver*
- *Hex ball driver, 3/32"*
- *HPLC-grade cleaning solvent*
- *Cleaning swabs/wipes*

1. Power off the unit from the back panel and disconnect the power cord.
2. Allow the unit to cool for at least 30 minutes.
3. Using a Phillips screwdriver, remove the cover screws, 1 on each side and 4 on the back panel.
4. Remove the ELSD cover by carefully sliding it toward the back of the instrument.

⚠ CAUTION **The optics block and drift tube may still be hot.**

5. Disconnect the sweep gas tubing from the light trap. The connection is a quick connect fitting; push in and then pull towards you to remove. Refer to **Figure 5.1** for details.
6. Using the hex ball driver, remove the 4 hex head screws connecting the light trap to the optics block and carefully remove the light trap. Refer to **Figure 5.1** for details. To clean, inspect the inside of the light trap for residue. Swab the inside of the light trap as necessary with a suitable solvent to clean any residue. The inside of the light trap should be black.
7. With the light trap removed, the optics block can be accessed for cleaning. Inspect the inside of the optics block for residue. Swab the inside of the optics block with a suitable solvent to clean any residue. The inside of the optics block should be black.

8. Once the optics have been cleaned, reattach the light trap and replace the 4 hex head screws. Make sure the light trap has the proper orientation so the magnetic interlock makes the proper connection. Refer to **Figure 5.1** for details. Carefully reconnect the light trap sweep gas tubing.
9. Replace the instrument cover and tighten the cover screws.
10. Reconnect the power cable.
11. Resume normal operation of the unit. If the problem persists, continue troubleshooting with the manual or contact your local BUCHI Customer Service office which can be found on the BUCHI website at: www.buchi.com.

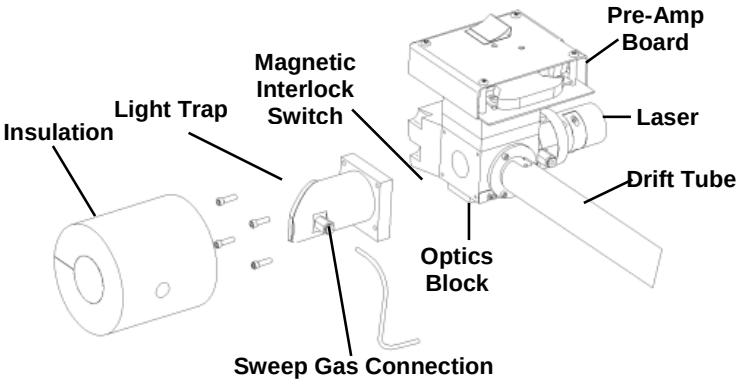


Figure 5.1: Optics Cleaning Procedure

5.5 FUSE REPLACEMENT

⚠ DANGER **Only suitably qualified personnel should carry out these adjustments. Shock hazard: disconnect the power cord before replacing the fuse.**

⚠ CAUTION **Fire hazard: only use the same type and rated CERTIFIED fuse (—▭— 250V 5A F).**

1. Power off the detector from the back panel and unplug the power cord.
2. Gently squeeze the side tabs of the fuse drawer and pull the fuse drawer straight out. Refer to **Figure 5.2** for details.
3. Remove the blown fuse and replace with the new fuse. Be sure to use the appropriate fuse: 5 Amp 250V. Make sure the replacement fuse is placed into the fuse slot on the right side of the fuse drawer.
4. Replace the fuse drawer. Make sure the fuse inserts into the fuse slot on the right side of the fuse block. The correct slot has a metal contact inside the fuse block.

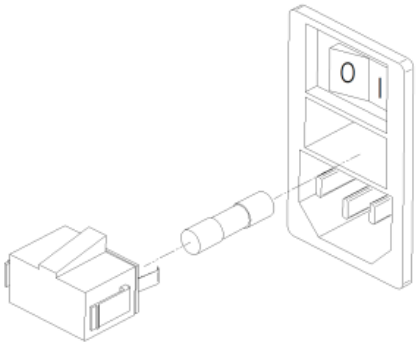


Figure 5.2: Model 3300 ELSD Fuse Block

6. DIAGNOSTICS AND TROUBLESHOOTING

6.1 ERRORS

The main screen will list any errors currently occurring on the instrument in the lower right corner. Operation error messages will remain until the error condition is remedied. Operation errors relate to temperature and gas flow accuracy during operation. Initialization errors will appear on bootup for 15 seconds and then disappear. Initialization errors relate to calibration issues (i.e. if the unit is uncalibrated or if the yearly calibration is out of date).

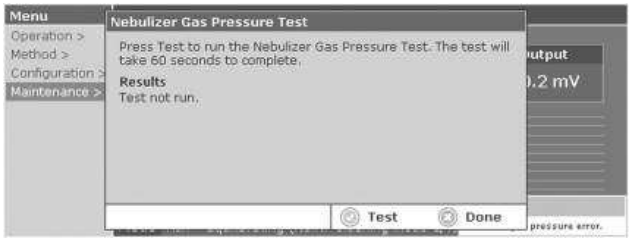
ERRORS

ERROR	WHEN OCCURS	POSSIBLE CAUSES	SOLUTIONS
Drift tube temperature error.	Drift tube temperature reading is more than 10°C (+/-) off its set point.	Heater, thermal fuse, or electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Optics block temperature error.	Optics block temperature reading is more than 10°C (+/-) off its set point.	Heater, thermal fuse, or electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Gas flow error.	Gas flow reading is more than 0.3L/min (+/-) off its set point.	Gas source may be low or empty. Regulator pressure setting is too low. Nebulizer may be blocked. Gas flow valve, meter, or electronics error.	Check gas source and replace if necessary. Adjust regulator pressure to 65-80psig. Clean nebulizer. Refer to **Section 5.2** for details. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Inlet gas pressure is low.	Inlet gas pressure is lower than 40psig.	Regulator pressure setting is too low. Gas source may be low or empty. Gas pressure sensor or electronics error.	Adjust regulator pressure to 65-80psig. Replace gas source if necessary. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Calibration (laser, gas, and/or heaters) is out of date.	Unit requires its yearly recalibration.		Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Any errors not included in this list.			Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

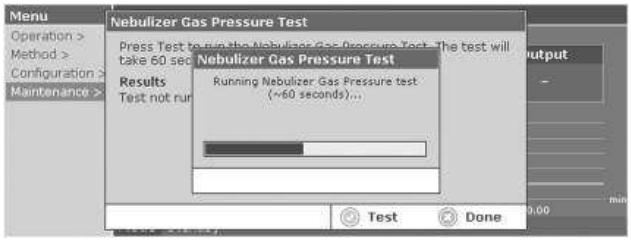
6.2 PERFORMING DIAGNOSTIC TESTS

6.2.1 NEBULIZER GAS PRESSURE TEST

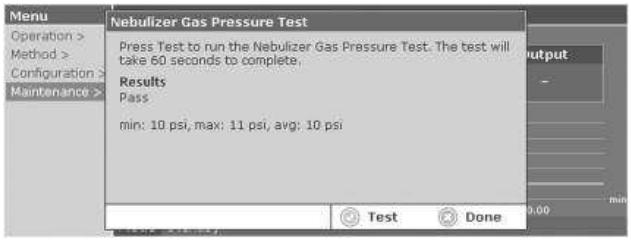
1. Make sure the inlet gas pressure is set between 65 and 80psig before starting the test.
2. Turn off mobile phase flow. Wait several minutes for the detector to stabilize.
3. In the software, select Maintenance/Tests/Nebulizer Gas Pressure Test and press Enter. The Nebulizer Gas Pressure Test screen will then appear:



4. Press Test and the Nebulizer Gas Pressure Test will begin. The test takes 60 seconds to complete. A timer bar will appear showing how much time is left for the test:



5. The following steps occur during the Nebulizer Gas Pressure Test:
   - *Gas flow is set to 2.0L/min and allowed to stabilize.*
   - *Minimum, maximum, and average nebulizer gas pressure values are determined.*
   - *Test results are displayed. The gas flow is returned to its pre-testing set point.*



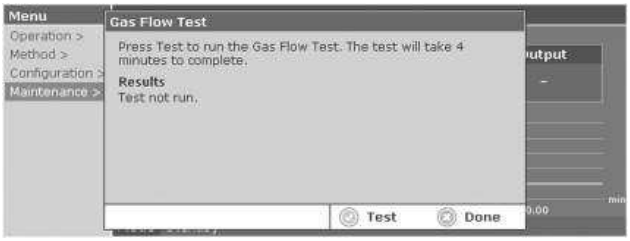
6. Passing requirements for the Nebulizer Gas Pressure Test are:
   - *Nebulizer gas pressure must be between 3 – 30psig.*
   - *Pressure variation must be less than 3psig during the testing period.*
7. Once the test is completed, press Done to return to the main screen, or press Test to repeat the test.
8. If the test has failed, refer to the following table, Nebulizer Gas Pressure Test Results, for possible fail messages and their solutions.

NEBULIZER GAS PRESSURE TEST RESULTS

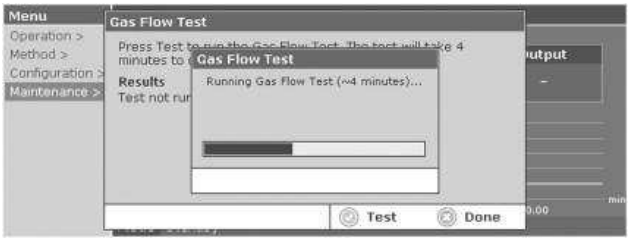
RESULT	CAUSE	SOLUTION
Fail: Gas pressure is too high.	Blocked nebulizer.	Clean the nebulizer. Refer to **Section 5.2** for details.
	Nebulizer gas pressure sensor error and/or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Fail: Gas pressure is too low.	Gas source may be low or empty.	Check gas source and replace if necessary.
	Regulator pressure may be too low.	Adjust regulator pressure to 65-80psig.
	Gas leaks may be present before the nebulizer.	Check gas connections for leaks and tighten fittings if necessary.
	Possible nebulizer gas pressure sensor error and/or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Fail: Gas pressure is unstable.	Blocked nebulizer.	Clean or replace the nebulizer. Refer to **Section 5.2** for details.
	Inlet gas pressure is unstable.	Check stability of gas source and remedy if necessary.
	Gas flow meter, valve, or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

6.2.2 GAS FLOW TEST

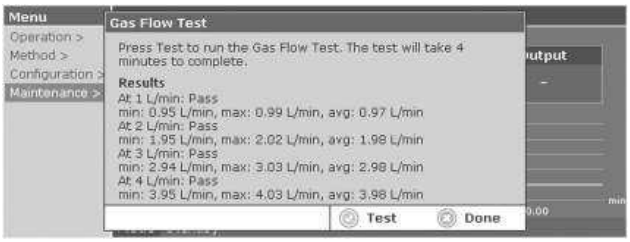
1. Make sure the inlet gas pressure is set between 65 and 80psig before starting the test.
2. Turn off mobile phase flow. Wait several minutes for the detector to stabilize.
3. In the software, select Maintenance/Tests/Gas Flow Test and press Enter. The testing screen will then appear:



4. The test takes 4 minutes to complete. A timer bar will appear showing how much time is left for the test.



5. The following steps occur during the Gas Flow Test:
   - *Gas flow is set to 1.0, 2.0, 3.0, and 4.0L/min and allowed to stabilize at each flow rate.*
   - *Minimum, maximum, and average gas flow rate values are determined at each flow rate.*
   - *Test results are displayed. The gas flow is returned to its pre-testing set point.*



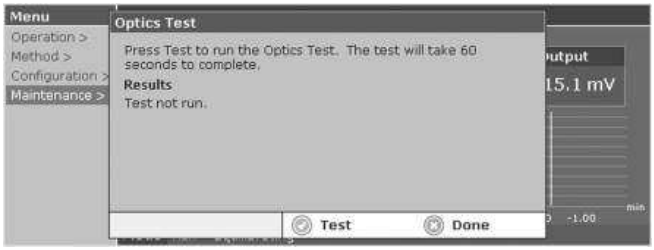
6. Passing requirements for the Gas Flow Test are:
   - *Gas flow must be within 0.2L/min at each gas flow set point (1.0, 2.0, 3.0, and 4.0L/min).*
7. Once the test is completed, press Done to return to the main screen, or press Test to repeat the test.
8. If the test has failed, refer to the following table, Gas Flow Test Results, for possible fail messages and their solutions.

GAS FLOW TEST RESULTS

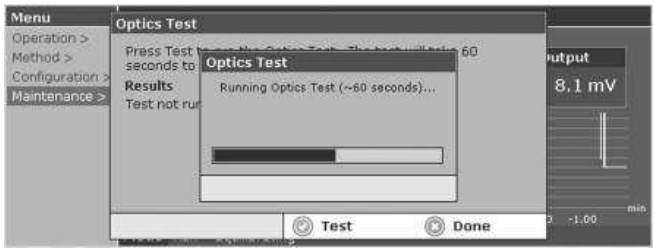
RESULT	CAUSE	SOLUTION
Fail: Gas flow is unstable.	Unstable inlet gas pressure. Gas flow meter and/or valve error. Blocked nebulizer.	Check stability of gas source and remedy if necessary. Contact your local BUCHI Customer Service office which can be found on www.buchi.com. Clean or replace nebulizer. Refer to **Section 5.2** for details.
Fail: Gas flow is lower than the set point.	Gas supply valve is closed. Gas supply is low or empty. Blockage or leaks in gas line before the detector.	Open gas supply valve. Check gas supply and replace if necessary. Check gas line for blockage and leaks; remedy/replace as necessary.
Fail: Gas flow is higher than the set point.	Gas flow calibration is not correct. Gas flow meter, valve, or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

6.2.3 OPTICS TEST

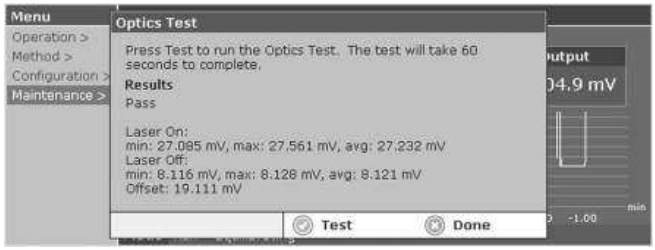
1. Make sure the inlet gas pressure is set between 65 and 80psig before starting the test. Make sure the gas flow source is connected to the 3300 ELSD with the valve open.
2. Turn off mobile phase flow. Wait several minutes for the detector to stabilize.
3. In the software, select Maintenance/Tests/Optics Test and press Enter. The Optics Test screen will then appear:



4. The test takes 60 seconds to complete and a timer bar appears indicating how much time is remaining on the test.



5. The following steps occur during the test:
   - *The laser is turned off and the detector is allowed to stabilize at Gain 1.*
   - *Minimum, maximum, and average signal data are collected for laser off.*
   - *The laser is turned on and the detector is allowed to stabilize.*
   - *Minimum, maximum, and average signal data are recorded for laser on.*
   - *The offset between laser on and laser off, and the amount of variation in laser signal are determined.*
   - *Test results are displayed.*



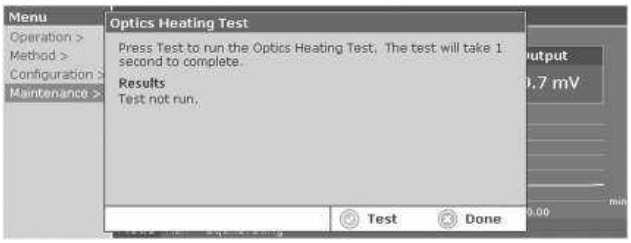
6. Passing requirements for the Optics Test are:
   - *Laser on/off offset must be between 0.5 – 80mV*
   - *Laser off stability must be within 1.5mV*
   - *Laser on stability must be within 1.5mV.*
7. Once the test is completed, press Done to return to the main screen, or press Test to repeat the test.
8. If the test has failed, refer to the following table, Optics Test Results, for possible fail messages and their solutions.

OPTICS TEST RESULTS

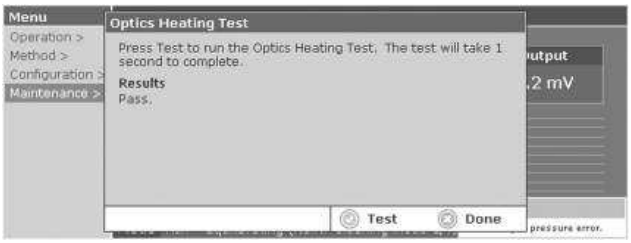
RESULT	CAUSE	SOLUTION
Fail: Noise level at laser off is too high.	Electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Fail: Offset between laser on and laser off is too high.	Optics need cleaning. Electronics error.	Clean optics. Refer to **Section 5.4** for details. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Fail: Offset between laser on and laser off is too low.	Laser or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Fail: Noise level at laser on is too high.	Optics need cleaning. Electronics error.	Clean optics. Refer to **Section 5.4** for details. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

6.2.4 OPTICS HEATING TEST

1. The detector must be in Run mode and the heaters equilibrated before the Optics Heating Test is run.
2. In the software, go to Maintenance/Tests/Optics Heating Test and press Enter. The testing screen will then appear:



3. Press Test to run the Optics Heating Test. The test takes 1 second to complete. The results screen will then appear.



4. Pass requirements for the test are:
   - *Optics Block must be 90% (+/-2%) of the Drift Tube Temperature set point.*

5. If the test has failed, refer to the following table, Optics Heating Test Results for possible causes and solutions.

OPTICS HEATING TEST RESULTS

RESULT	CAUSE	SOLUTION
Fail.	Detector is in Standby mode.	Put the detector in Run mode, allow to equilibrate, and then repeat the test.
	Detector is still equilibrating.	Wait for the detector to equilibrate and then repeat the test.
	Thermal fuse may be blown.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
	Optics block heater, sensor or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

6.3 DIAGNOSING BASELINE NOISE

There are many causes for baseline noise. Use the table below, Diagnosing Baseline Noise, to help identify the source.

Start diagnosing the noise at 'A' and work down the table until the source of the baseline noise is determined.

DIAGNOSING BASELINE NOISE

SYMPTOM	SOLUTION
A. Noise from Column • *Column in-line* • *Mobile phase on* • *Nebulizing gas on* • *Laser on* **Result:** Noise disappears when column is removed.	**1.** The column may be leaking silica or packing material. Replace the column.
B. Noise from Mobile Phase • *Column removed* • *Mobile phase on* • *Nebulizing gas on* • *Laser on* **Result:** Noise disappears when pump is stopped.	**1.** Current drift tube temperature and gas flow rate settings may not be providing adequate evaporation of the mobile phase. Re-optimize gas flow and/or drift tube temperature following the optimization procedure in **Section 4.6**. **2.** The nebulizer, drift tube, and/or optics may be dirty. Refer to **Sections 5.1 – 5.4** for cleaning procedures. **3.** The mobile phase may be contaminated with particulate matter. Filter the current mobile phase or replace it with freshly prepared and filtered mobile phase. **4.** The mobile phase may contain excess air bubbles. Degas the mobile phase. **5.** The pump may be the source. Check pump for pulsations. Make sure the pump has been sufficiently purged to remove air. Incorporate a pulse dampener into the system if necessary. Examine the pump check valve and seals and replace as necessary.
C. Noise from Gas • *Column removed* • *Mobile phase off* • *Nebulizing gas on* • *Laser on* **Result:** Noise disappears when gas is turned off.	**1.** The gas supply may be contaminated with particulates. Replace with better quality/higher purity gas. **2.** The nebulizer, drift tube, and/or optics may need cleaning. Refer to **Sections 5.1 – 5.4** for cleaning procedures.
D. Noise from Optics Block • *Column removed* • *Mobile phase off* • *Nebulizing gas off* • *Laser on* **Result:** Noise disappears when laser is turned off.	**1.** The optics may need cleaning. Refer to **Section 5.4** for optics cleaning procedure. **2.** Check data cable for noise. **3.** Check light trap for condensation. Refer to **Section 5.4** for details on removing the light trap.
E. Noise from Electronics • *Column removed* • *Mobile phase off* • *Nebulizing gas off* • *Laser off* **Result:** Baseline noise persists under the above conditions.	**1.** Possible electrical problem. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.

6.4 TROUBLESHOOTING CHARTS

Consult the following charts to assist in troubleshooting your system:

TROUBLESHOOTING

PROBLEM	CAUSE	SOLUTION
Baseline drift	Detector has not fully equilibrated.	Wait for detector to fully equilibrate. Refer to **Section 4.5**, Start-Up Sequence, for equilibration procedure.
Baseline noise	Follow the procedure in **Section 6.3**, Diagnosing Baseline Noise, to determine the source of the problem and possible solutions.	
Spiking	Drift tube temperature and/or gas flow rate set too low. Gas source contaminated or low purity. Mobile phase contaminated or made of low quality material. Nebulizer, drift tube, and/or optical cell dirty. Improper nebulization.	Re-optimize the drift tube temperature and gas flow rate following the optimization procedure in **Section 4.5**. Use clean, dry, inert gas, usually 99.9% pure nitrogen. Replace with fresh, filtered, higher-quality mobile phase. Refer to **Sections 5.1 – 5.4** for cleaning procedures. Nebulizer may be partially obstructed. Refer to **Section 5.2** for nebulizer cleaning procedure.
Drift tube temperature not reaching set point	Thermal fuse(s) may be blown. Drift tube heater, sensor, or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Optics block temperature not reaching set point	Thermal fuse may be blown. Optics block heater, sensor, or other electronics error.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
No gas flow	Gas source valve is closed. Source gas pressure is too low. Gas source may be low or empty.	Open gas valve. Adjust source pressure to 65-80psig. Replace gas source.
No power	Blown fuse.	Replace fuse. Refer to **Section 5.5**, Fuse Replacement, for details.

TROUBLESHOOTING

PROBLEM	CAUSE	SOLUTION
No LCD display	Electrical problem.	Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
No peak(s) detected	Sample is below the detection limit. Sample is volatile at current detector conditions. Sample is being retained on the column. Gain is set too low. Autosampler needle is not pulling up sample properly, or sample loop blockage. Bad laser or other electronics error.	Increase the sample concentration or injection volume and re-inject. Lower temperature settings are needed for semi-volatile compounds. Refer to **Section 4.6**, Optimization Procedure, for details. Use a different column for your separation. Increase the gain value. Repair or replace equipment as needed. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Change in peak height or loss in sensitivity	Nebulizer, drift tube, and/or optics are dirty. Autosampler needle not pulling up sample properly, or sample loop blockage. Bad laser or other electronics error.	Clean the nebulizer, drift tube, and optics as needed. Following the cleaning procedures in **Sections 5.1 – 5.4**. Repair or replace equipment as needed. Contact your local BUCHI Customer Service office which can be found on www.buchi.com.
Broad peaks	Leak(s) (especially between the column and detector) present. Tubing between column and detector is too long or too large of an i.d..	Check for loose fittings and tighten if necessary. Use a shorter piece of 0.005 - 0.010" i.d. tubing.
Cut-off peaks	Sample concentration too high. Gain set too high.	Decrease sample concentration until peaks are on-scale. Reduce gain value until peaks are on-scale.

7. APPENDIX

7.1 SPECIFICATIONS

MODEL 3300 ELSD SPECIFICATIONS	
Light Source:	Laser diode with collimating optics, 650nm, max output less than 30mW, Class IIIB
Product Class:	Class 1 laser product
Detector Element:	Silicon photodiode
Temperature Range:	Ambient to 120°C in 0.1°C increments
Nebulizer Gas:	Up to 4.0L/min, nitrogen preferred, 65psig min. pressure, 80psig max pressure
Mobile Phase Flow Rate:	50uL-3.0mL/min
Analog Outputs:	Selectable for either 0-1V or 0-10mV full scale
Communications:	**Remote Inputs:** TTL/Contact closure-Autozero, Gas Shutoff, Start, Standby **Outputs:** Contact closure--Fault Relay, External Output; RS232, USB (Device), USB (Host), Ethernet
Operating Parameter Selection & Display:	Windows®-Based Graphical LCD with alphanumeric keypad
Power Requirements:	120/240V, 50/60Hz, 5A
Dimensions:	10.0" H x 10.25" W x 19.0" D (25.4cm H x 26.0cm W x 48.3cm D)
Weight:	30 lbs (13.6 kg)
Environmental Operating Temperature:	15 to 40˚C
Relative Humidity:	10 – 90%, Non-Condensing

7.2 REPLACEMENT PARTS

MODEL 3300 ELSD REPLACEMENT PARTS		
Part No.	**Qty.**	**Description**
145136931	1	3300 ELSD Accessory Kit (includes all parts listed above the divider)
142112995	1	Signal Cable
142112993	1	Power Cord, 110V
142107259	1	Open-End Wrench, 3/8" x 7/16"
142106694	1	Open-End Wrench, 1/4" x 5/16"
142107268	1	Ball Driver, 3/32"
142112992	1	Ball Driver, 7/64"
142112880	1	Drift Tube Cleaning Brush
142110773	1	PEEK Tubing, 1/16" o.d. x .005" i.d., 10’
145126955	1	SofGrip™ Fittings, 10/pk
142110623	1	Gas Tubing, 10’
142111502	1	1/8" Brass Nut
142111503	1	1/8" Brass Ferrule
142112997	1	Drain Tubing, 5'
142112990	1	Clamp for Drain Tubing
143112777	1	Exhaust Tubing, 20’
142108384	1	Exhaust Adapter
142112994	1	Fuse, 5 Amp
11593900	1	Model 3300 ELSD Operating Manual
142112991	1	14 Pin Terminal Block
142113967	1	Nebulizer
142113968	1	Nebulizer Wear Band
142114811	1	Chemraz O-ring for Nebulizer
142107480	1	.25 x .79 Aperture
142107476	1	.31 x .74 Aperture
142107511	1	.36 x .74 Aperture
148606365	1	Gas Filter
142113956	1	Front Service Door
142113954	1	Inlet Gas Fitting

7.3 VOLATILE MOBILE PHASE MODIFIERS

VOLATILE BUFFERS AND MOBILE PHASE MODIFIERS					
	pKa	**pKb**	**pH Range**	**BP**	**MP**
Acids					
Trifluoroacetic Acid	0.3	13.70		72.4°C	
Formic Acid	3.75	10.25		100.7°C	
Acetic Acid	4.75	9.25		116.0°C	
Carbonic Acid	6.37	7.63		-	
Bases					
Ammonia	9.25	4.75		-33.35°C	
Methylamine	10.81	3.19		16.6°C	
Ethylamine	10.66	3.34		-6.3°C	
Triethylamine	11.01	2.99		89.3°C	
Buffers					
Ammonium Formate			3.0-5.0		120°C
Pyridinium Formate			3.0-5.0		
Ammonium Acetate			3.8-5.8		111°C
Pyridinium Acetate			4.0-6.0		
Ammonium Carbonate (used for reverse phase)			8.0 (adjusted)		
Ammonium Carbonate			5.5-7.5 and 9.3-11.3		
Ion-Pair Reagents					
Pentafluoropropionic Acid	~0.6			96-97°C	
Heptafluorobutyric Acid	~0.6			120°C	
Nonafluoropentanoic Acid	~0.6			140°C	
Pentadecafluorooctanoic Acid	~0.6			189°C	
Tridecafluoroheptanoic Acid	~0.6			175°C	

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9. USEFUL REFERENCES

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