171/172
Diode Array Detectors
User's Guide

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Safety

Read this section before installing and operating the 171 and 172 Diode Array Detectors.

The detector is intended to be used in a laboratory environment by trained technical personnel.

For safe and correct use of this instrument, it is recommended that both operating and service personnel follow the instructions contained in this guide when installing, cleaning, and maintaining the instrument.

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

The detector has been certified to safety standards specified for Canada, Europe, and the United States. Refer to the instrument rear panel label and the Declaration of Conformity document for the current standards to which the instrument has been tested.

Safety

The following electronic and hazard symbols may appear on the instrument:

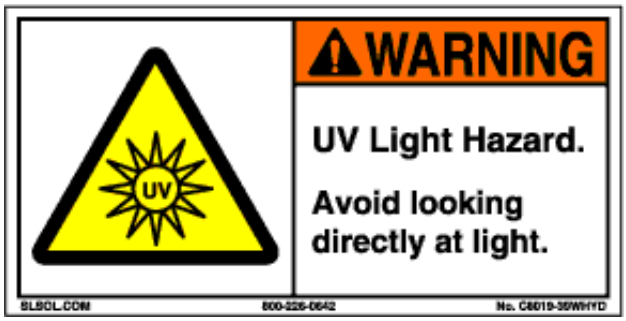
Symbol	*Explanation*		
~	Alternating current	Courant alternatif	Wechselstrom
⎓	Direct current	Courant continu	Gleichstrom
	Protective conductor terminal	Borne de terre de protection	Schutzleiteranschluss
|	Electrical power ON	Sous tension	Netzschalter ein
O	Electrical power OFF	Hors tension	Netzschalter aus
	Caution	Attention	Vorsicht
	Caution, risk of electric shock	Attention, risque de choc électrique	Vorsicht, Elektroschockgefahr
	Caution, hot surface	Attention, surface chaude	Vorsicht, heiße Oberfläche
	Caution, ultraviolet light	Attention, rayonnement ultraviolet	Vorsicht, Ultraviolettes Licht
	Fuse	Fusible	Sicherung

The following safety notices may appear in this document:

⚠WARNING	WARNING indicates a potentially hazardous situation which, if not avoided, may result in serious injury
⚠CAUTION	CAUTION indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury
NOTICE	NOTICE indicates a potentially hazardous situation which, if not avoided, may result in equipment damage

Ultraviolet (UV) Radiation

⚠WARNING Never look directly into the light from the deuterium lamp. Always use protective glasses with a UV filter, if you have to open the lamp housing while the deuterium lamp is on.



Warning: The deuterium lamp emits UV radiation that is harmful to the eyes. Always turn off the detector power before removing the lamp cover.

Avertissement: La lampe deuterium emettet de rayons ultraviolets, qui sont dangeroux pour les yeux. Voulez vous etteindre le detecteur avant d'enlever le couvercle de la lampe.

Warnung: Die Deuteriumlampe gibt UV-Strahlung ab; diese ist schädlich für die Augen. Schalten Sie den Detektor immer aus, ehe Sie die Abdeckung von der Lampe entfernen.

Fiber Optics

⚠WARNING Do not look directly at the light emitted from the fiber optics.

Please note the following when handling the fiber optics:

- Observe a minimum bend radius for optical fibers. For a fiber optic with a diameter of 0.6 mm, the smallest allowable bending radius is approximately 50 mm for internal fibers and approximately 120 mm for external fibers.
- Avoid contact with fingers or contamination of the optical fiber tip. Even very thin layers of contaminants will greatly impair measurements, particularly in the UV range.
- If the front surface of the optical fiber tip becomes dirty, it should be cleaned with a lint-free cloth.
- When not in use, the ends of the optical fibers should always be protected with the end caps delivered with the fibers.

Voltage

Access to the rear panel is necessary. Always disconnect the power cord from the detector before opening the top cover. The detector must be detached from all voltage sources before service, repair, or exchange of parts.

Under no circumstances is the detector to be operated with the top cover removed. For normal operation the detector is to be grounded through the AC line cord provided. Failure to do so can result in a potential shock hazard that could result in serious personal injury.

Use only fuses with the rated current and of the specified type (fast acting, normal blow, time delay) as listed on the rear panel of the instrument.

The instrument must only be operated with the voltage specified on the rear panel label of the instrument using a grounded AC line cord. Parts which conduct voltage can be exposed if covers are opened or parts removed.

Heat

The deuterium lamp can reach temperatures of 176°F (80°C) in normal operation. Allow 30 minutes for the lamp to cool before servicing/replacing.

Solvents

Observe safe laboratory practices when handling solvents. If dangerous liquids are used, adequate protection such as proper ventilation, safety glasses, etc., should be used.

Replacement Parts

Be sure to use only replacement parts mentioned in Chapter 4, Maintenance and Appendix B, Replacement Parts. Do not repair or change parts which are not listed in this user's guide. If it is necessary to change parts not listed, please contact your local Gilson representative.

Introduction 1

The Gilson 171 and 172 Diode Array Detectors are software-controlled absorbance diode array detectors. These detectors are intended to be used in a laboratory environment by trained technical personnel. Both detector models are compatible components of a Gilson modular HPLC system.

This chapter provides information on the following topics:

- Unpacking
- Customer Service
- Technical Specifications

Figure 1-1: 172 Diode Array Detector

Unpacking

The detector and its accessories are shipped in two boxes. Unpack the detector and its accessories carefully from the boxes.

- One box contains the detector, Ethernet cable, terminal block connector, fuses, power cords, documentation CD, IQ document, setup guide, and Declaration of Conformity.
- Another box contains the flow cell accessory kit and any other accessories for the detector.

Cross-check the contents against the Standard Equipment and against your purchase order's optional accessory list to verify that all parts are included and undamaged. Do this now, even if the detector will not be used immediately. Many carriers must receive concealed damage claims within seven days of delivery.

The detector is delivered with most major components already assembled. Keep the original container and packing assembly in case the detector must be returned to the factory.

Standard Equipment

After the detector and accessories are unpacked, you should have the following:

- 171 or 172 Diode Array Detector
- Ethernet cable
- terminal block connector
- fuses
- power cords

Documentation

The following documents are included with the detector:

- 171/172 Diode Array Detectors Documentation CD, which includes:
  - 171/172 Diode Array Detectors User's Guide
  - IQ Procedure 171/172 Diode Array Detectors
- 171/172 Diode Array Detectors Setup Guide
- Declaration of Conformity

Accessories

Based on your configuration, you also ordered and received additional accessories.

Flow Cell Accessory Kits

Each of the flow cell accessory kits includes a flow cell assembly with pre-connected inlet and outlet tubing, a plumbing package, and a 2.5 mm Allen wrench. The flow cell accessory kits for analytical applications also include a back pressure regulator.

Part number	*Description*
Analytical	
18004008	Flow cell accessory kit for analytical applications (with stainless steel inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
18004007	Flow cell accessory kit for analytical applications (with PEEK inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
Preparative	
18004006	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.2 mm pathlength, 0.7 µL volume, quartz
18004005	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.05 mm pathlength, 0.16 µL volume, quartz

Remote Flow Cell Holder

Part number	*Description*
18004004	Remote cell holder Includes the remote cell holder and fiber optic cables. Flow cell accessory kits are ordered separately.

Customer Service

Gilson, Inc. and its worldwide network of authorized representatives provide customers with the following types of assistance: sales, technical support, applications, and instrument repair.

If you need assistance, please contact your local Gilson representative. Specific contact information can be found at www.gilson.com. To help us serve you quickly and efficiently, please refer to Before Calling Us on page 5-6.

Technical Specifications

Please be aware of the following before operating the detectors.

NOTICE Changes or modifications to this device not expressly approved by Gilson could void the warranty.

The instrument complies with part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) This instrument may not cause harmful interference, and (2) this instrument must accept any interference received, including interference that may cause undesired operation.

Shielded cables must be used with the detector to ensure compliance with the FCC Class A limits.

Technical Specification	*Definition*
Communication	Ethernet
Contact Control	Two open collector outputs and two contact inputs
Data Acquisition	Between 5 and 10 points/sec with TRILUTION® LC Software
Detection Type (Pixels)	256-element photodiode array (171) 512-element photodiode array (172)
Dimensions	26.5 x 43.5 x 15.6 cm (10.4 x 17.1 x 6.2 in)
Drift	**Baseline drift*** <300 x 10^{-6} AU/hour **Wavelength temperature drift** <0.006 nm/°C (171) <0.005 nm/°C (172) *Value was obtained using the following conditions: Wavelength: 254 nm, Lamp Warm Up Time: 10 hours, Ambient Temperature: 21°C ± 2°C. Refer to ASTM E685-93 (2005).
Short Term Noise (Signal/Noise Ratio)	<10 x 10^{-6} AU Values were obtained with air block installed using the following conditions: Wavelength: 254 nm, Lamp Warm Up Time: 10 hours, Ambient Temperature: 21°C ± 2°C. Refer to ASTM E685-93 (2005).
Environmental Conditions	Indoor use Altitude: up to 2000 m Temperature range: 5°–40°C Air pressure: 75–105 kPa Humidity: Maximum relative humidity 80% for temperatures up to 31°C, decreasing linearly to 50% relative humidity at 40°C
Flow Cells	The following quartz flow cells are available:

Flow cell	*Pathlength*	*Volume*	*Pressure*		
			psi	*bar*	*MPa*
Analytical	5 mm	12 µL	500	34.5	3.45
Preparative	0.05 mm	0.16 µL	500	34.5	3.45
Preparative	0.2 mm	0.7 µL	500	34.5	3.45

Technical Specification	*Definition*
Front Panel	POWER/STANDBY soft key and LED indicator lights for POWER, UV, VIS, and ERROR
Fuse	Two 5 x 20 mm, "TD" type, 1.60A
Lamp Warm-Up Time	15–30 minutes
Light Source	Deuterium and tungsten lamps (2000 hours expected lamp life)
Power Requirements	Frequency: 50 to 60 Hz Voltage: 100–240V 100W
Remote Flow Cell	Optional
Resolution	2.2 nm/pixel (171) 0.8 nm/pixel (172)
Safety and Compliance	The detector has been certified to safety standards specified for Canada, Europe, and the United States. Refer to the instrument rear panel label and the Declaration of Conformity document for the current standards to which the instrument has been tested.
Software Control	Computer control via Ethernet and TRILUTION® LC Software
Wavelength Accuracy	± 1 nm Repeatability <0.1 nm
Wavelength Range	190–720 nm (171) 190–610 nm (172)
Weight	6.8 kg (15 lbs) for 171 Diode Array Detector 7.3 kg (16 lbs) for 172 Diode Array Detector

Installation 2

This chapter takes you through the steps for setting up your detector, which includes:

- Operating Environment
- Rear Panel Connections
- Flow Cell Assembly Installation
- Remote Flow Cell Holder (Optional)
- Plumbing Connections

Operating Environment

You can use the detector in normal laboratory and cold room environments.

The following factors will influence the performance of your detector.

Temperature

Changes in ambient temperature can produce noticeable output variations.

Avoid areas that are in direct sunlight for part of the day and those that are subject to draft; for example, do not place the detector near an open window or doorway.

Contaminants

Smoke, vapors, and acid fumes can cause baseline drift and noise and can cause damage to the detector.

Ventilation

Leave at least 10 cm (4 in) of clearance behind the detector to allow exhaust from the cooling fan to escape. Also, do not block the ventilation holes on the bottom of the detector.

Condensation

Before starting, the detector must have reached the temperature range specified (refer to Technical Specifications for more information). If the detector has been very cold, it should stand for at least two hours in its new environment in order to allow any condensation on the surfaces to evaporate.

Rear Panel Connections

Refer to the diagram below when making the connections described in this section.

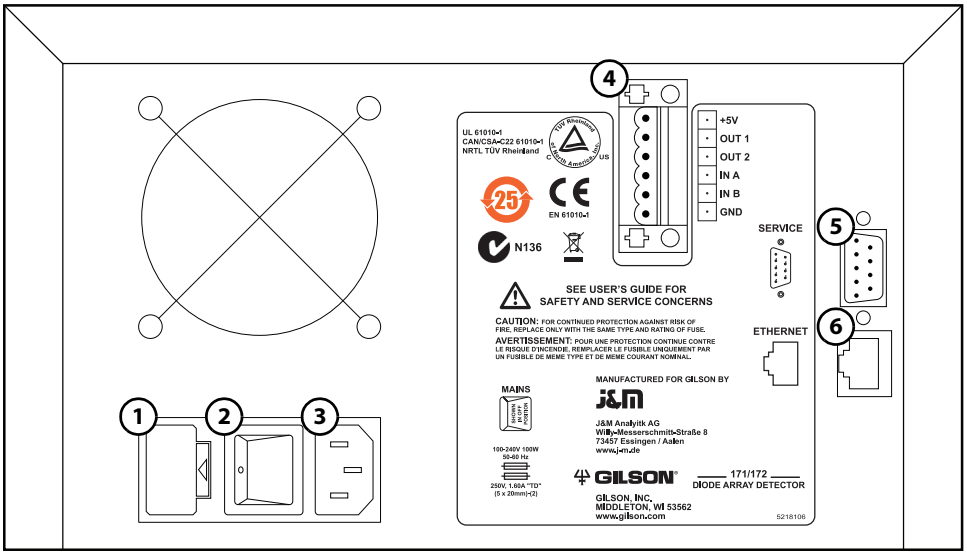


Figure 2-1: 171/172 Diode Array Detectors Rear Panel

1 Fuse drawer

2 Power switch (MAINS)

3 Power receptacle

4 Input/output ports

5 Service (factory use only)

6 Ethernet

Ethernet

To make the Ethernet connection to the instrument, a router and Ethernet cables are needed.

Note: The router is not available from Gilson, Inc.

1 Connect power to the router

Connect the power supply to the router and then connect the power supply to a power source.

2 Connect the instrument to the router

Before connecting, ensure that the instrument is powered OFF.

Locate the Ethernet cable that was included with the detector. Plug one end of the cable into the Ethernet port on the instrument and the other end to an Ethernet port on the router.

3 Connect the computer to the router

Connect one end of another Ethernet cable to an Ethernet port on the router and the other end to an Ethernet port on the network adapter in the computer.

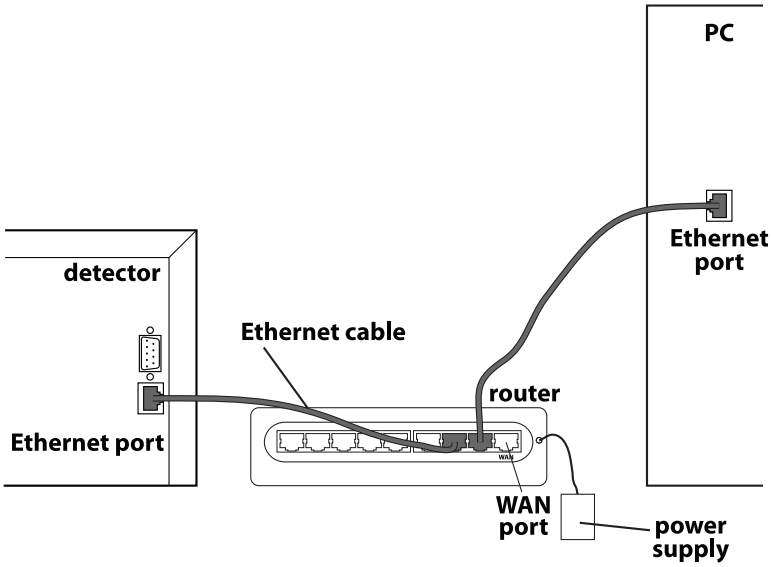


Figure 2-2: Ethernet Connections

Input/Output Ports

Use the input and output contacts on the rear panel of the detector to control peripheral devices.

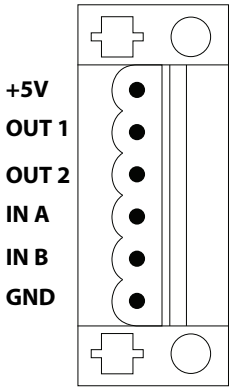


Figure 2-3:
Input/Output Ports

Contact Inputs

The detector has two contact inputs, labeled IN A and IN B.

Open Collector Outputs

The detector has two open collector outputs, labeled OUT 1 and OUT 2.

Make Connections

The following are needed to make connections:

- terminal block connector
- 2-conductor cable (22–30 gauge for each wire)

  You can purchase a 6-foot piece of suitable cable (part number 709910206) or a package of five cables with identification markers (part number 36078155) from Gilson.
- wire insulation stripper
- small-blade screwdriver

To make connections using the 2-conductor cable:

1 Cut the cable into pieces of appropriate length.

2 Strip about 3 mm of insulation from each end of the cable.

3 Remove the terminal block connector from the detector. Insert each wire into the appropriate terminal on the terminal block connector.

Note: When making connections, be sure to maintain the correct orientation of the connector relative to the port.

Push the wire all the way in; then tighten its corresponding pin screw.

4 Reconnect the terminal block connector to the detector. Push the connector in as far as it will go. It is designed to fit snugly into its receptacle.

5 Connect the opposite ends of the wires to the other equipment. Be sure to match ground connections.

6 Label each cable to identify the purpose of the connection.

Power Cord Connection

Locate the appropriate power cord for the voltage.

Use the power cord to connect the detector to a power source.

Flow Cell Assembly Installation

Follow these instructions to install the flow cell assembly on the detector. To install the flow cell assembly on a remote flow cell holder, refer to Remote Flow Cell Holder (Optional) on page 2-7.

NOTICE Be extremely careful when working with the flow cell and its fittings. Flow cells are considered expendable and are not covered by warranty if damaged or broken during installation.

1 Open the front door of the detector.

2 Remove the fiber optic cables and set aside. The fiber optic cables are located in a bag inside the door of the detector.

3 Locate the box containing the flow cell assembly and the two thumbscrews that will be used to attach the flow cell assembly to the detector.

4 Orient the flow cell assembly with the labels IN and UP facing you. IN should be at the bottom and UP at the top.

5 Align the holes in the flow cell assembly with the holes on the flow cell assembly bracket.

6 Place the thumbscrews through the bracket and into the flow cell assembly and tighten.

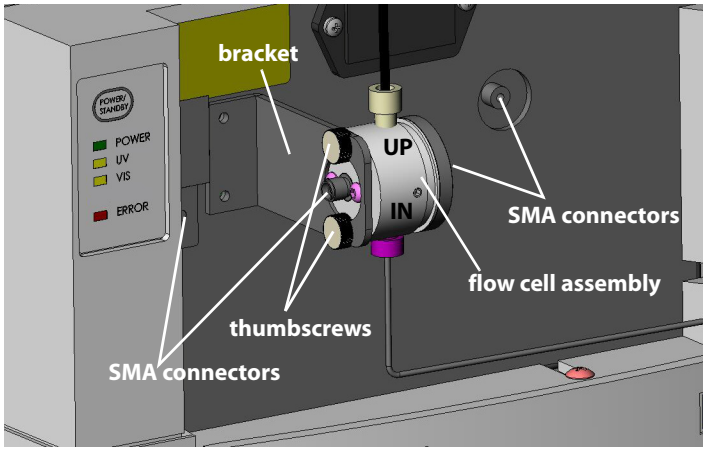


Figure 2-4: Flow Cell Assembly Installed

7 Remove the fiber optic cables from the bag and then remove the protective caps from the ends of the cables.

NOTICE Be careful not to touch the connector ends of the fiber optic cables.

The caps can be stored in the bag and placed inside the detector door for future use.

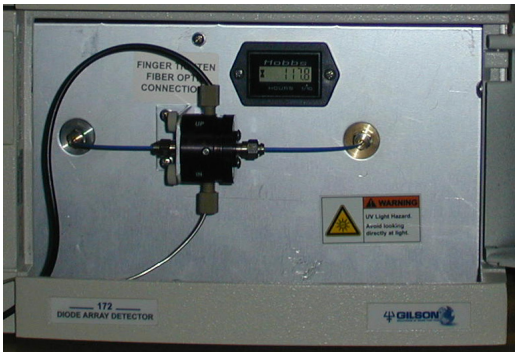


Figure 2-5: Fiber Optic Cables Connected

8 Connect one end of a fiber optic cable to the SMA connector on the left side of the detector. Finger tighten.

9 Connect the other end of the fiber optic cable to the SMA connector on the left side of the flow cell assembly. Finger tighten.

NOTICE The fiber optic cables should not be over-bent. To avoid bending the cable too much, place your finger on the inside (right side) of the fiber optic cable and then curve the cable around your finger and connect it to the SMA connector on the left side of the flow cell assembly.

10 Connect one end of a fiber optic cable to the SMA connector on the right side of the detector. Finger tighten.

11 Connect the other end of the fiber optic cable to the SMA connector on the right side of the flow cell assembly. Finger tighten.

NOTICE The fiber optic cables should not be over-bent. To avoid bending the cable too much, place your finger on the inside (left side) of the fiber optic cable and then curve the cable around your finger and connect it to the SMA connector on the right side of the flow cell assembly.

12 Connect one end of a fiber optic cable to the SMA connector on the left side of the detector. Finger tighten.

Remote Flow Cell Holder (Optional)

Refer to these instructions to assemble the remote flow cell holder and then install the flow cell assembly.

Remote Flow Cell Holder Assembly

The remote flow cell holder assembly (part number 18004004) includes the remote flow cell holder base and bracket, two screws, and two fiber optic cables.

To assemble the remote flow cell holder:

1 Place the remote flow cell holder base on a flat surface with the label facing the front.

2 Orient the bracket so it makes an L-shape when attached to the base and then align the holes on the bracket with the holes in the base.

3 Insert the two screws and tighten using the Allen wrench included with the flow cell assembly.

Flow Cell Assembly Installation

To install the flow cell assembly on the remote flow cell holder:

1 Open the front door of the detector.

2 Locate the box containing the flow cell assembly and the two thumbscrews that will be used to attach the flow cell assembly to the detector.

3 Orient the flow cell assembly with the labels IN and UP facing you. IN should be at the bottom and UP at the top.

4 Align the holes in the flow cell assembly with the holes on the remote flow cell holder bracket.

5 Place the thumbscrews through the bracket and into the flow cell assembly and tighten.

6 Remove the fiber optic cables from the box and then remove the protective caps from the ends of the cables. The caps can be stored inside the detector door for future use.

NOTICE Be careful not to touch the connector ends of the fiber optic cables.

7 Connect one end of a fiber optic cable to the SMA connector on the left side of the detector. Finger tighten the fiber optic connections.

8 Connect the other end of the fiber optic cable to the SMA connector on the left side of the flow cell assembly. Finger tighten the fiber optic connections.

NOTICE The fiber optic cables should not be over-bent.

9 Connect one end of a fiber optic cable to the SMA connector on the right side of the detector. Finger tighten the fiber optic connections.

10 Connect the other end of the fiber optic cable to the SMA connector on the right side of the flow cell assembly. Finger tighten the fiber optic connections.

NOTICE The fiber optic cables should not be over-bent.

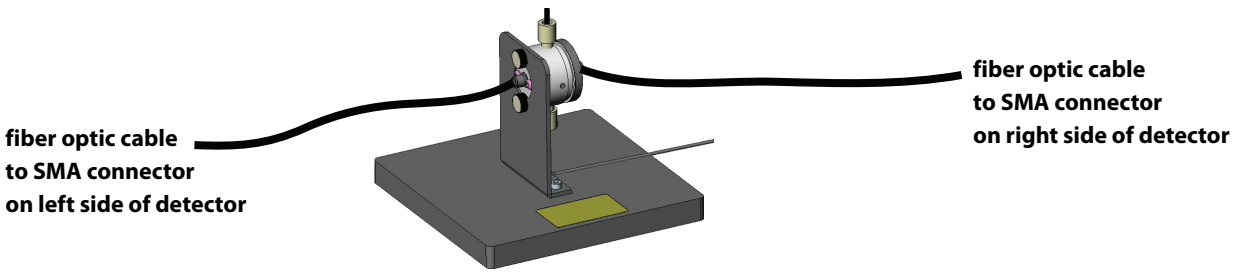


Figure 2-6: Fiber Optic Cables Attached to Remote Flow Cell Holder

Plumbing Connections

This section describes the following plumbing connections:

1 Inlet tubing to the column outlet

2 Outlet tubing to a back pressure regulator, fraction collector, or waste container

Note: The flow cell accessory kits for analytical applications include a back pressure regulator.

Inlet Tubing

Connect the inlet tubing to the column outlet using the F-100N Fingertight fitting, included in the plumbing package (part number 18007000). This fitting accommodates most existing female column outlets.

To connect the fitting, insert it onto the tubing. Screw the fitting into the column outlet and then finger tighten.

Instructions for replacing the inlet tubing and fittings are found in Chapter 4, Maintenance.

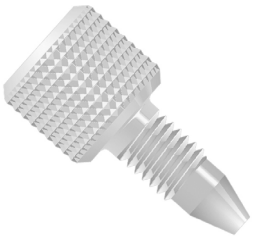


Figure 2-7: F-100N Fingertight Fitting

Outlet Tubing

The outlet tubing from the detector can be directed three different ways:

- to a back pressure regulator
- to the inlet of the fraction collector in order to collect the column effluent
  Use the appropriate coupler to make the connection. Universal couplers are included in the plumbing package (part number 18007000).
- to a waste container if the column effluent does not need to be saved

Back Pressure Regulator

When using an HPLC flow cell, additional back pressure on the outlet tubing may be needed to reduce bubble formation. If additional back pressure is needed, connect the outlet tubing to a back pressure regulator before directing the effluent to the fraction collector or waste container.

The flow cell accessory packages for analytical applications include a 75 psi back pressure regulator as shown. The arrow on the back pressure regulator identifies the direction of flow. The recommended operating flow rate range is 0.1–10 mL/min.*

* Information provided by Upchurch Scientific® Inc

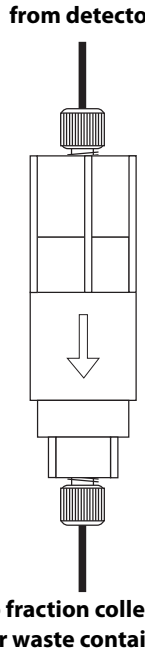


Figure 2-8: Back Pressure Regulator

Operation 3

TRILUTION® LC Software provides programmed control of the 171 and 172 Diode Array Detectors during a chromatographic run. For each injection made during the run, up to eight channels of data can be acquired and viewed in TRILUTION LC.

After the run, a chromatogram at any wavelength and a spectrum at any time point can be displayed. Additionally, the iso-electric or 3D plot for collected data can be displayed.

This chapter provides the following information:

- A description of the Front Panel of the detector
- How to Start Up the detector
- Instructions for using TRILUTION LC to Turn Lamps On
- Instructions for Detector Control from TRILUTION LC
- Instructions for viewing Results

Front Panel

Figure 3-1: 172 Diode Array Detector Front Panel

The front panel of the detector contains a POWER/STANDBY soft key and indicator lights for POWER, UV, VIS, and ERROR.

POWER/STANDBY When the rear panel MAINS switch is ON, this key can be used to turn the detector OFF or ON. Press and hold this key to turn the detector OFF. To turn the detector ON again, press this key. The rear panel MAINS (power) switch will still be in the ON position if this key is used to turn the detector OFF.

POWER The POWER indicator illuminates when the rear panel MAINS (power) switch is switched ON. This indicator remains ON as long as the detector is ON. When the POWER/STANDBY key is used to turn the detector OFF, the MAINS (power) switch in the back will still be in the ON position but the POWER light will not be ON.

UV The UV indicator illuminates when the deuterium lamp is lit. (The lamps are controlled using TRILUTION LC.)
After the lamps are turned on, the UV indicator will flash until the UV lamp is lit.

VIS The VIS indicator illuminates when the tungsten lamp is lit. (The lamps are controlled using TRILUTION LC.)

ERROR The ERROR indicator illuminates when there is an instrument error. Refer to ERROR LED is On on page 5-2.
The ERROR indicator will flash intermittently until the detector is fully powered.
The ERROR indicator will illuminate briefly when the lamps are turned on. (The lamps are controlled using TRILUTION LC.)

Start Up

Follow the instructions in Chapter 2, Installation, to make all rear panel and plumbing connections.

Ensure that the power supply is connected to the router and that the power supply is connected to a power source.

Switch the detector ON using the rear panel MAINS (power) switch.

- The POWER indicator on the front panel illuminates.
- The ERROR indicator on the front panel flashes intermittently until the detector is fully powered.

Turn Lamps On

TRILUTION LC is required to turn the detector lamps on. Ensure that the detector is powered ON before starting TRILUTION LC.

1 Create a TRILUTION LC method with the detector in the configuration.
   a) Click **Scan**. The software searches for the detector.
   b) From the Available Instruments window, drag and then drop the detector in the workspace.

2 On the Control tab, drag and then drop the Turn Lamp On task into the control workspace.

3 Save the method and then click **Run**. The Application Run window appears.

4 On the Application Run window, select the method name from the Method Configuration drop-down list and then click  .

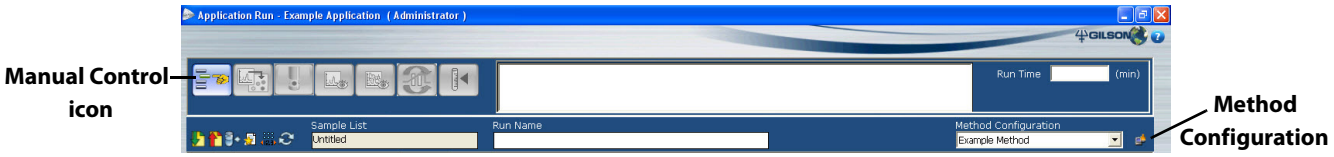


Figure 3-2: Application Run Window

5 Click the Manual Control icon. The Manual Control window appears.

6 Drag and then drop the Turn Lamp On task into the workspace. Click **OK**.

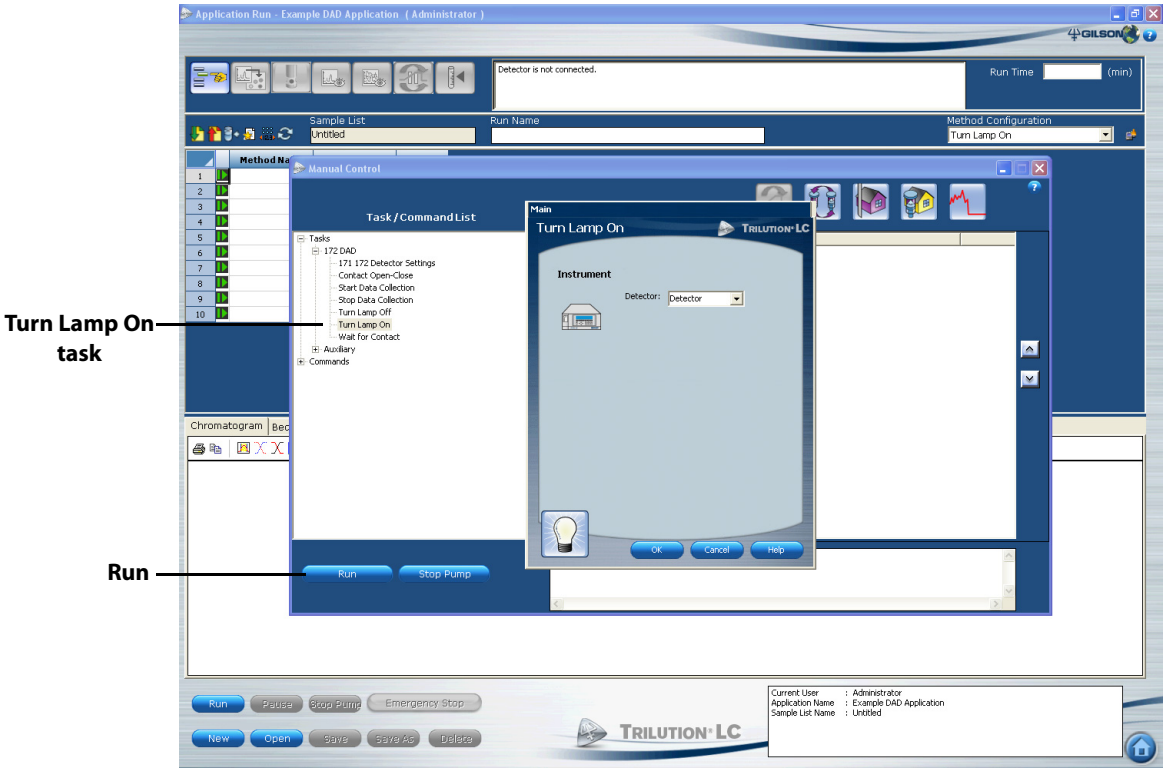


Figure 3-3: Turn Lamp On Task

7 Click **Run** on the Manual Control window.
   - The UV indicator on the detector front panel flashes until the lamp is lit and then remains lit while the lamp is on.
   - The VIS indicator on the detector front panel illuminates.
   - The ERROR indicator on the detector front panel illuminates briefly.

   Note: The detector requires a 15–30 minute warm-up period to achieve a stable baseline.

Detector Control from TRILUTION LC

The following pages describe how to set conditions for detector control via TRILUTION LC and view spectral data. For additional information about method setup and the chromatography run in TRILUTION LC, refer to its on-line help or user's guide.

Configuration

On the Configuration tab in the Method Builder, tell the software that a diode array detector will be used and how many data channels on that detector will be used.

How to Create a Configuration

1 In the Method Builder, select the Configuration tab.

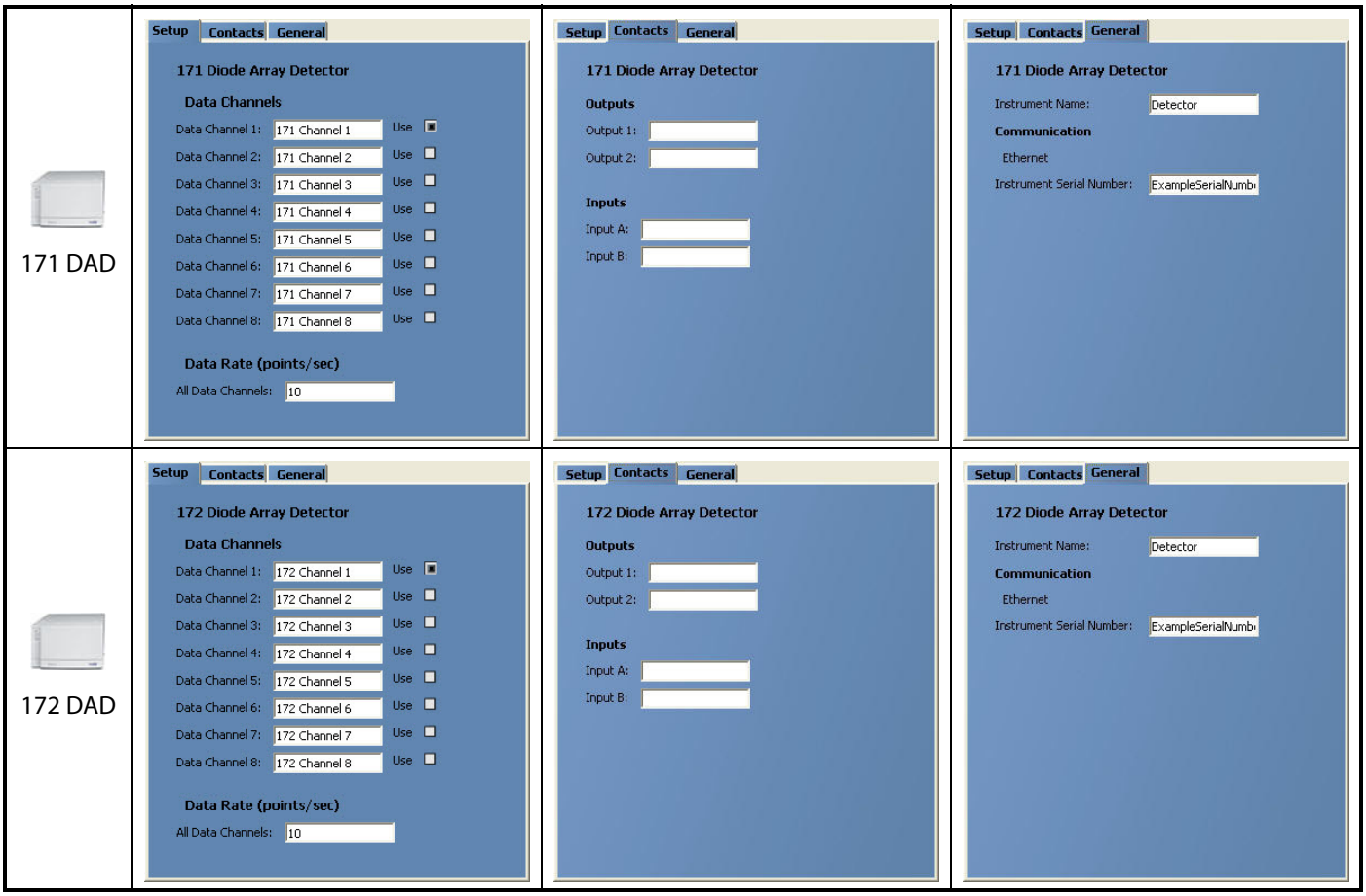
2 Click **Scan**. The software searches for connected instruments.

3 From the Available Instruments window, drag and then drop the 171 Detector or 172 Detector in the workspace.

4 For the Detector, right-click on a data channel marked for use and then select **Primary Channel**. One channel per configuration must be specified as the primary channel.

Note: When collecting fractions, the software monitors the primary channel.

Configuration Properties



171/172 Diode Array Detectors Properties

Name	*Brief Description*	*Default Value*	*Range*
Data Channels	Optionally, type a unique description for each data channel. This description and the data channel's signal will be displayed in the status box while data is being collected during an Application Run if the Use check box is selected for that data channel. The Use check box for Data Channel 1 defaults selected. There are eight data channels available (labeled 17X Channel 1 through 17X Channel 8, where X is 1 or 2, dependent on the model of the detector).	171 Channel 1 172 Channel 1	N/A
Data Rate (points/sec)	The number of data points collected per second for all channels. One rate is set for all channels. The recommended setting is between 5 and 10 points/sec.	10	1–10
Contacts	Optionally, type a description for the contact. This description and the contact's status will be displayed at all times in the status box during an Application Run. There are two Outputs available (labeled OUT 1, OUT 2) and two Inputs available (labeled IN A, IN B).	N/A	N/A
Instrument Name	The name that will be used to identify this instrument in a task.	Detector	N/A
Instrument Serial Number	The serial number of the detector.	N/A	N/A

171/172 Diode Array Detectors Properties

Control

The Control tab window in the Method Builder is a graphical interface used to organize the tasks used in a method.

171/172 Diode Array Detectors Tasks

The table below lists the tasks for the 171 and 172 Diode Array Detectors.

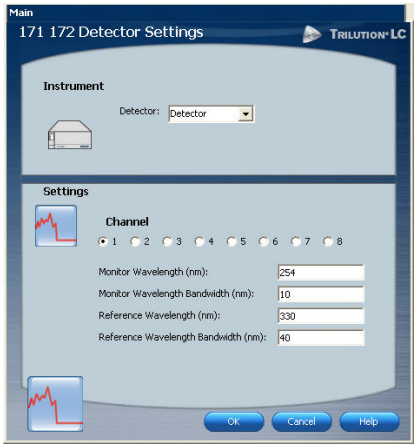
171/172 Diode Array Detectors Tasks

	Gilson Task Name and Description
	171 172 Detector Settings Sets the DAD wavelengths and bandwidths to be monitored based on each selected Channel for the selected Detector.
	Contact Open-Close This task opens and powers off, closes and powers on, or pulses the specified output contact on a specified instrument.
	Start Data Collection This task tells TRILUTION LC to begin collecting data. This task has no properties to set.
	Stop Data Collection This task tells TRILUTION LC to stop collecting data. This task has no properties to set.
	Turn Lamp Off This task turns off power to the lamps while maintaining power to the Detector.
	Turn Lamp On This task turns the UV and visible lamps on for the specified Detector.
	Wait for Contact This task initiates a wait in the method until any contact state change or a user-specified contact state change for the specified contact is detected. Sync to the end of the task (using a Sync Task) to prevent other tasks in the method from executing during the wait.

171/172 Diode Array Detectors Tasks

171 172 Detector Settings

Sets the DAD wavelengths and bandwidths to be monitored based on each selected Channel for the selected Detector.



171 172 Detector Settings Properties

Property Name	*Description*	*Default Value*
Detector	The detector that the task will affect.	Detector
Channel	Select Channel number for which wavelength and bandwidth values will be set. Repeat for each channel to be used. (1–8)	1
Monitor Wavelength	The wavelength at which chromatogram data will be extracted and displayed on-screen during the run. Range for 171 Diode Array Detector (190–720 nm): [Monitor Wavelength - (0.5 x Monitor Wavelength Bandwidth)] > 186 and [Monitor Wavelength + (0.5 x Monitor Wavelength Bandwidth)] < 723 Range for the 172 Diode Array Detector (190–610 nm): [Monitor Wavelength - (0.5 x Monitor Wavelength Bandwidth)] > 186 and [Monitor Wavelength + (0.5 x Monitor Wavelength Bandwidth)] < 612	254 nm
Monitor Wavelength Bandwidth	The number of wavelengths that TRILUTION LC will use in the chromatogram calculation for the monitor wavelength. Optimizing the bandwidth improves the signal-to-noise ratio to obtain the optimum signal for peak detection. (Range 1–15 nm, range dependent on setting for Monitor Wavelength)	10 nm
Reference Wavelength	The reference wavelength is used to correct for background noise and instabilities in the detector. Choose a wavelength that is close to the monitor wavelength and in a region of non-absorbance. Range for the 171 Diode Array Detector (190–720 nm): [Reference Wavelength - (0.5 x Reference Wavelength Bandwidth)] > 186 and [Reference Wavelength + (0.5 x Reference Wavelength Bandwidth)] < 723 Range for the 172 Diode Array Detector (190–610 nm): [Reference Wavelength - (0.5 x Reference Wavelength Bandwidth)] > 186 and [Reference Wavelength + (0.5 x Reference Wavelength Bandwidth)] < 612	330 nm
Reference Wavelength Bandwidth	The reference wavelength bandwidth sets the number of wavelengths that TRILUTION LC will use in the chromatogram calculation for the reference wavelength. Optimizing the bandwidth improves the signal-to-noise ratio to obtain the optimum signal for peak detection. (Range 1–50 nm, range dependent on setting for Reference Wavelength)	40 nm

171 172 Detector Settings Properties

Analysis

Using the options in the Method Builder - Analysis window, you indicate how collected data is analyzed and reported. TRILUTION LC uses the information in the analysis to report on peaks detected in samples.

To create an analysis, right-click on a data channel in the Configured Instruments Panel and then select **New Analysis**.

Peak Purity

Access Peak Purity options from the Method Builder - Analysis - Peak Integration tab window.

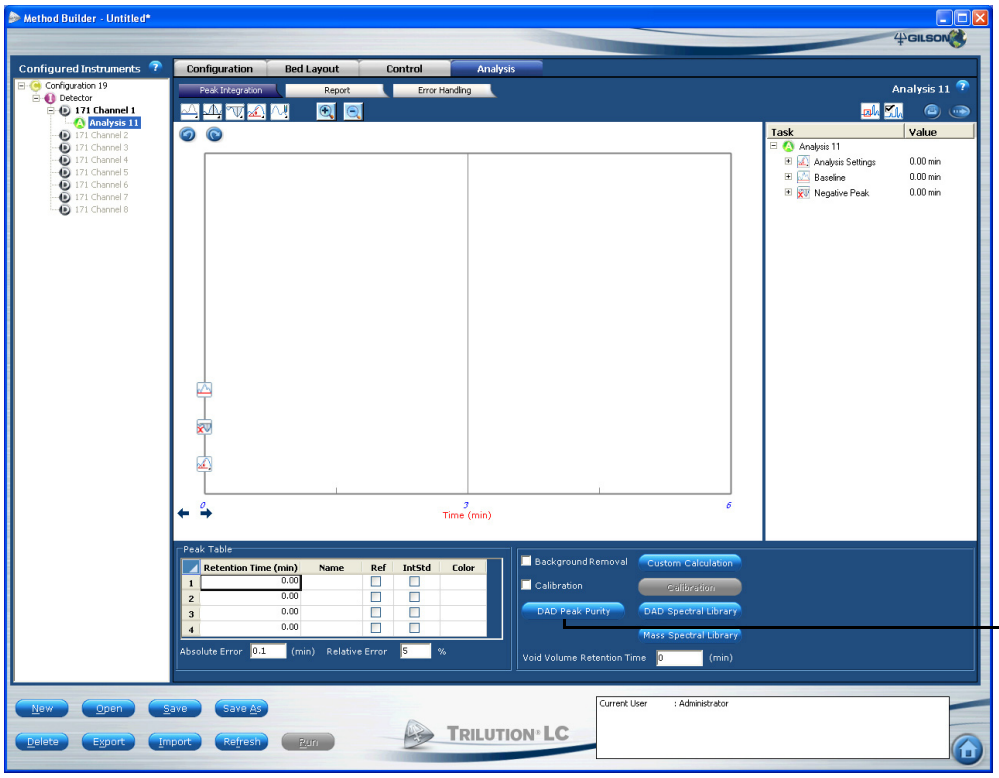


Figure 3-4: Method Builder - Analysis - Peak Integration Tab Window

Peak Purity is a spectral comparison of the apex spectrum to spectra from across the peak. This comparison is used to check the purity of the peak. The spectra that are compared depend on the Number of Sampled Spectra selected.

For more information about the peak purity options, refer to the on-line help.

Figure 3-5: Peak Purity

Spectral Library

In TRILUTION LC, you can assemble reference databases of spectral peaks of known compounds. You can then search and match the database contents to the chromatogram for an unknown sample and report the outcome.

Access the DAD Spectral Library from the Method Builder - Analysis - Peak Integration tab window.

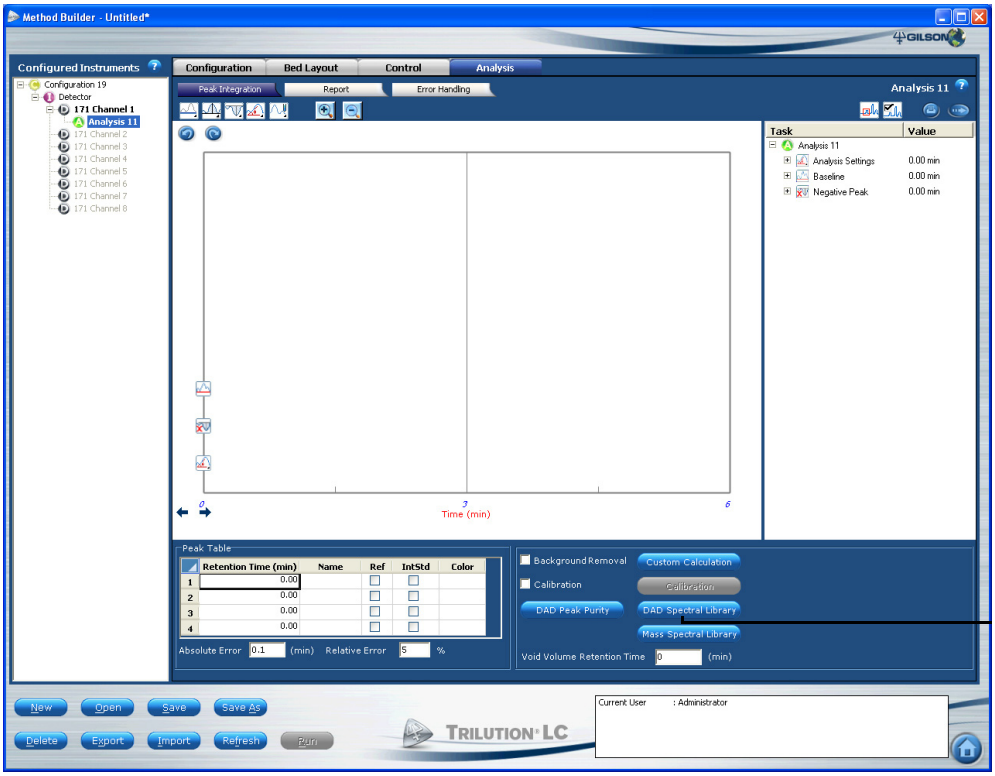


Figure 3-6: Method Builder - Analysis - Peak Integration Tab Window

To perform spectral matching at run time:

1 Click **DAD Spectral Library** to display the Search DAD Spectral Library dialog.

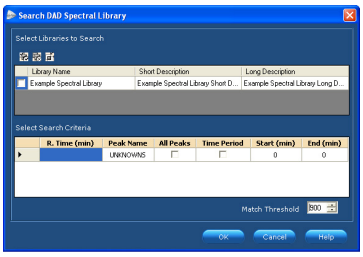


Figure 3-7:
Search DAD Spectral Library

2 In the Search DAD Spectral Library dialog:

a) Select the check box next to the **Library Name** for each library to be searched.

b) For each peak spectrum to match, do one of the following:

- Select the check box next to the **Peak Name** (UNKNOWNS and/or named peaks from the Peak Table) and under **All Peaks** to search all Spectral Library peaks.
- Select the check box next to the **Peak Name** (UNKNOWNS and/or named peaks from the Peak Table) and under **Time Period** and then enter **Start** and **End** times to limit the search to Spectral Library peaks whose retention times are within that time period.

c) In the **Match Threshold** field, indicate the smallest match value to be reported. A match value of 1000 indicates a perfect match.

3 Click **OK**.

Report Options

The report tab in the Method Builder - Analysis, provides options for adding spectral match results and peak purity values to Analysis Reports (refer to Analysis Report Elements), Calibration Reports, and Summary Reports.

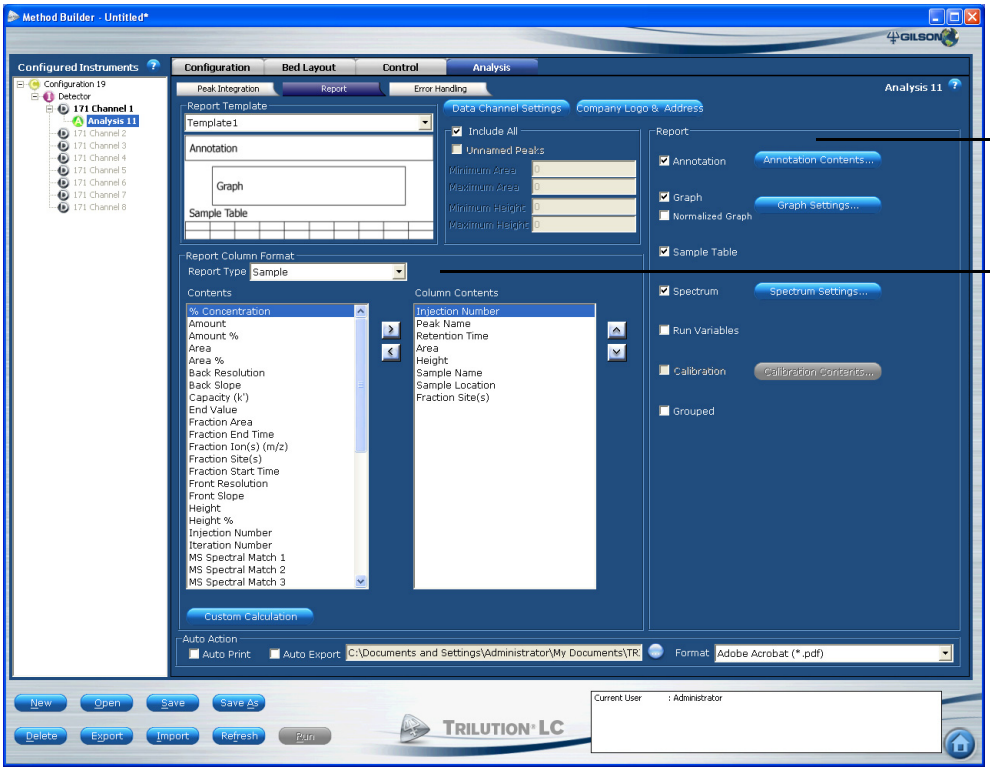


Figure 3-8: Method Builder - Analysis - Report Tab Window

Report Column Contents

Peak Purity - The calculated peak purity value.

Spectral Match 1 - The best spectral match according to spectral search criteria defined in the Method Builder - Analysis.

Spectral Match 2 - The second best spectral match according to spectral search criteria defined in the Method Builder - Analysis.

Spectral Match 3 - The third best spectral match according to spectral search criteria defined in the Method Builder - Analysis.

Set Report Contents

To set report contents:

1 Select a Report Type from the drop-down list.

2 From the Column Contents list, select the information type to be added to or removed from the report.

- Click the right arrow ( ) to add the information to Column Contents and to the report.
- Click the left arrow ( ) to remove information from the Column Contents and report.

3 Click the up ( ) or down ( ) arrow to reorganize the columns. Columns listed up to down in the Column Contents will appear left to right in the report.

Analysis Report Elements

A report can contain any or all of the following:

- Annotation
- Graph and/or Normalized Graph
- Sample Table
- Spectrum
- Run Variables
- Grouped Peaks

Select the check box for each element to include.

Spectrum

You can choose whether spectral views will be displayed and how they will be displayed in the Analysis Report.

1 On the Method Builder - Analysis - Report tab, select the **Spectrum** check box and then click **Spectrum Settings...** .

2 When the Spectrum Settings dialog appears, use the check boxes on the Diode Array Spectrum tab to select which spectral views to display in the Analysis Report.

3 Specify the minimum and maximum wavelengths to display.

Note: When setting the monitor and reference wavelengths in the 171 172 Detector Settings task, those values must be between the minimum and maximum wavelength values.

4 Specify the color scheme (3D and Iso-electric plots only) and elevation angle and rotation (3D plot only).

5 Specify the size of the spectrum as a percentage of the page.

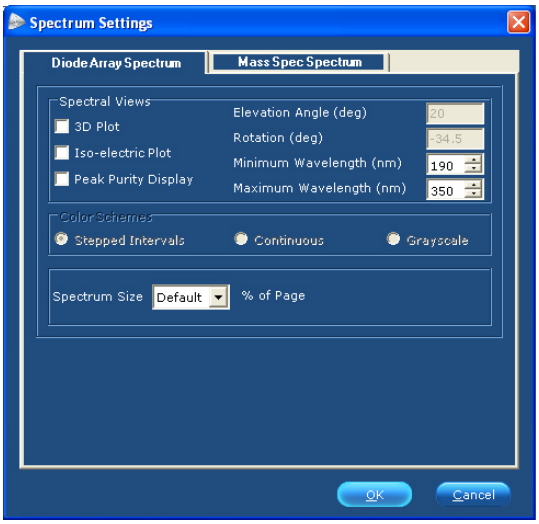


Figure 3-9: Spectrum Settings

Application Run

In the Application Run window, specify the list of steps, called a sample list, to execute during a run. A step in the sample list identifies the method to run. Access the Application Run window by:

- right-clicking an application in the Project Library and then selecting **Run** or
- selecting an application in the Project Library and then clicking **Run** or
- opening a saved method in the Method Builder and then clicking **Run**.

Ensure that the lamp is on before collecting any data, either during your run or while monitoring the baseline in Manual Control.

After setting up the sample list, click **Run** in the Application Run window to begin executing the steps in the run.

Sample Re-injection

Optionally, in the Sample List, choose to re-inject a sample based on either a named peak or an unknown peak meeting specified peak criteria (Peak Purity, for example) in the Default Analysis on the Primary Channel.

To set peak criteria for re-injecting a sample:

1 Right-click on a column name and then select **Sample Re-injection**.

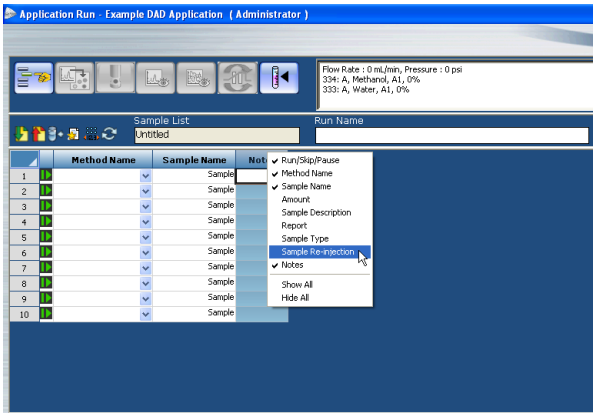


Figure 3-10: Select Sample Re-injection

2 Ensure that a Method has been selected and then click ....

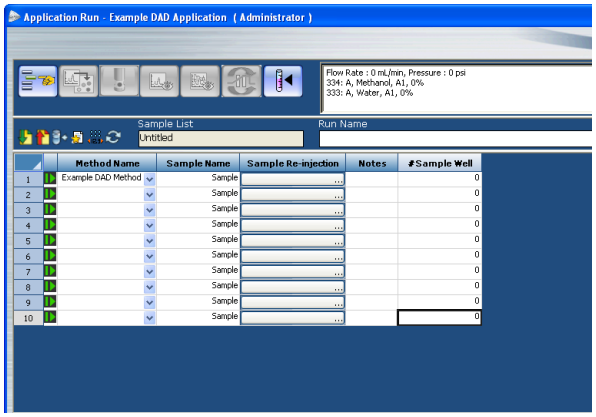


Figure 3-11: Select a Method

3 The Sample Re-injection Criteria dialog appears.

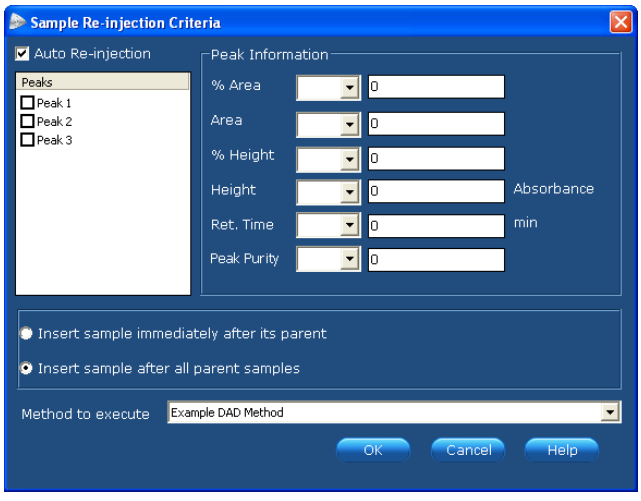


Figure 3-12: Sample Re-injection Criteria Dialog

Refer to the on-line help for more information about setting the criteria.

Results

From a run, the software produces Results containing the channel data collected and stored for all injected samples. Results contain time and signal information for each sample.

Results are accessed in the Project Library. Click on an application and double-click the Results icon ( ). The Results window is displayed, listing all runs for the selected application.

Double-click on a Run Name to display the Run Results window.

Run Results Window

The Run Results window enables viewing chromatogram plots and analysis information for collected data. To view chromatogram results, preview the report.

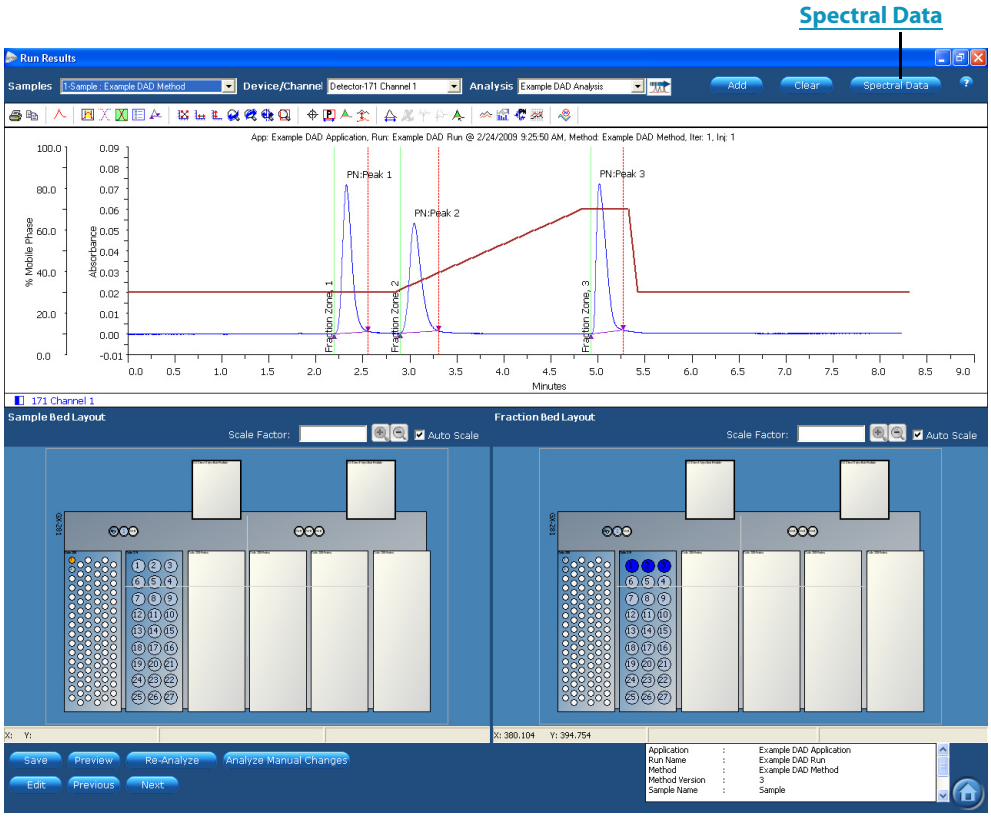


Figure 3-13: Run Results Window

Spectral Data

Clicking the Spectral Data button in the Run Results window enables you to view chromatogram and spectral plots for one sample and create spectral libraries using data collected by the 171 or 172 Diode Array Detector. Information is provided on three tabs: Spectral Data Tab below, Peak Purity Tab on page 3-16, and Library Tab on page 3-17.

Spectral Data Tab

Use the Spectral Data tab to view diode array data.

By default, the following are displayed:

- a 3D plot of the spectral data set
- a spectrum plot showing the APEX of:
  - the first named peak in the peak table found
  - or the first integrated peak found (if no peaks were named in the Peak Table)
- the chromatogram

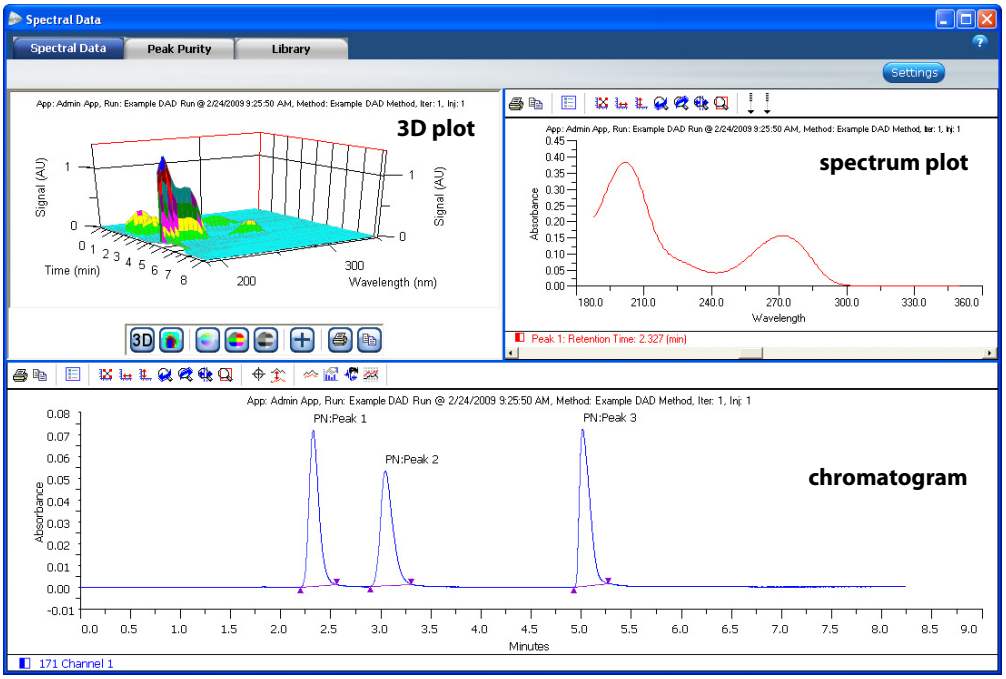


Figure 3-14: Spectral Data Window - Spectral Data Tab

3D plot/Iso-electric plot

Zoom (3D plot)

To zoom toward the center of the plot, right-click while dragging the mouse downward.

To zoom away from the center of the plot, right-click while dragging the mouse upward.

Zoom (Iso-electric plot)

To zoom in, right-click on the plot.

To zoom out, right-click outside the plot on either axis.

Display Legend

To display the legend, right-click next to the toolbar and then select **Control Panel**. The legend is displayed identifying the absorbance range associated with each color pattern.

Toolbar

Choose key functions using the icons in the toolbar.

Icon	*Description*
	Displays the 3D plot.
	Displays the iso-electric plot.
	Displays the Time, Wavelength, and Absorbance at the intersect of the cross hair when the iso-electric plot is displayed. Click on the iso-electric plot to update the wavelength on the chromatogram and the retention time on the spectrum.

Chromatogram

Shows the chromatogram plot for the selected data channel and analysis. Optionally shows an overlaid data trace at a user-selected wavelength (using cross hair tool on the iso-electric plot or **Settings** button).

Settings

Click the **Settings** button and then modify any or all of the parameters. After clicking **OK**, the overlaid chromatogram trace updates to reflect those settings. These settings cannot be saved and will revert to the run time settings upon exiting the Spectral Data window.

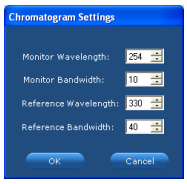


Figure 3-15: Settings

Spectrum Plot

Displays a spectrum plot showing the APEX of:

- the first named peak in the peak table found
- or the first integrated peak found (if no peaks were named in the Peak Table)

To display a spectrum for a different peak, double-click in the peak on the chromatogram.

Peak Purity Tab

Use the Peak Purity tab to compare the apex spectrum to spectra across the peak to check the purity of the peak. Peak Purity options are set in the Method Builder - Analysis - Peak Integration tab. By default, the chromatogram, the sampled spectra for the first peak found, and a similarity curve are displayed.

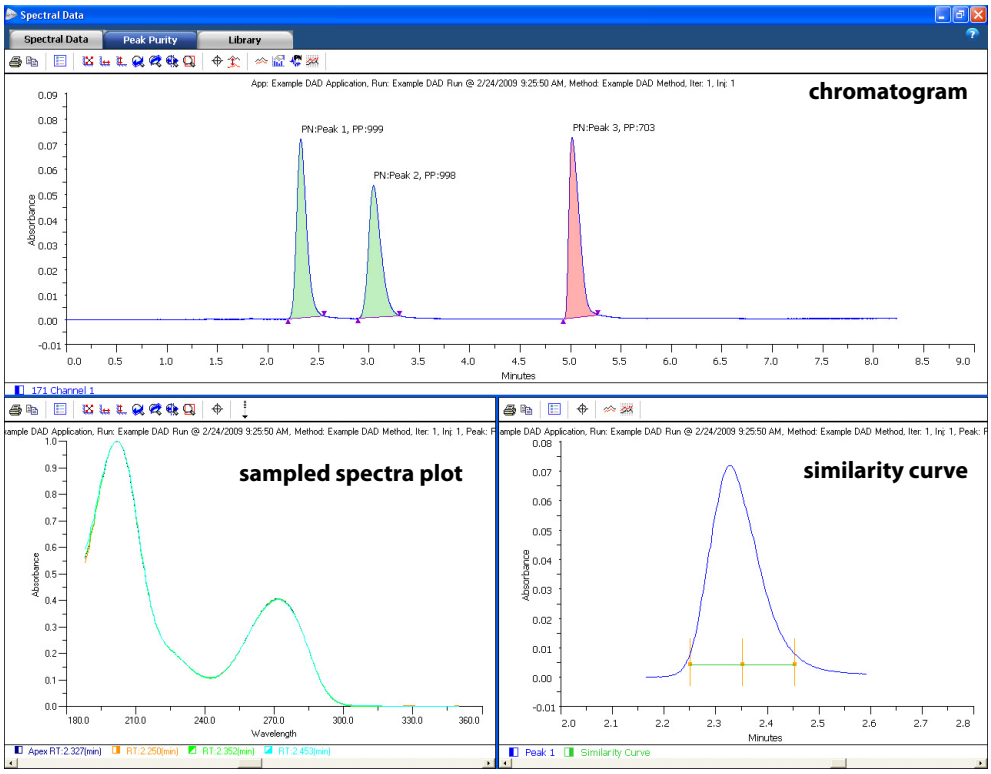


Figure 3-16: Spectral Data Window - Peak Purity Tab

Chromatogram

Shows the chromatogram plot for the selected data channel and Analysis and displays the peak purity values for each of the integrated peaks. Peak Purity values are preceded by **PP:**. If TRILUTION LC calculates a peak purity that is below the Purity Threshold value set in the Peak Purity options in the method, the peak is filled red. If the calculated peak purity is above the Purity Threshold value, the peak is filled green.

Sampled Spectra Plot

Displays the sampled spectra for the first peak found. To display spectra for a peak other than the first one found, double-click in the peak on the chromatogram.

Similarity Curve Plot

The similarity curve is a representation of individual purity calculations during elution. The number of points on the curve are the Number of Sampled Spectra selected in the Peak Purity options in the Method Builder - Analysis - Peak Integration tab.

Library Tab

In TRILUTION LC, you can assemble reference databases of spectra for known compounds. You can then search and match the database contents to the chromatogram for an unknown sample and report the outcome.

You can perform spectral matching at run time or during post-run re-analysis.

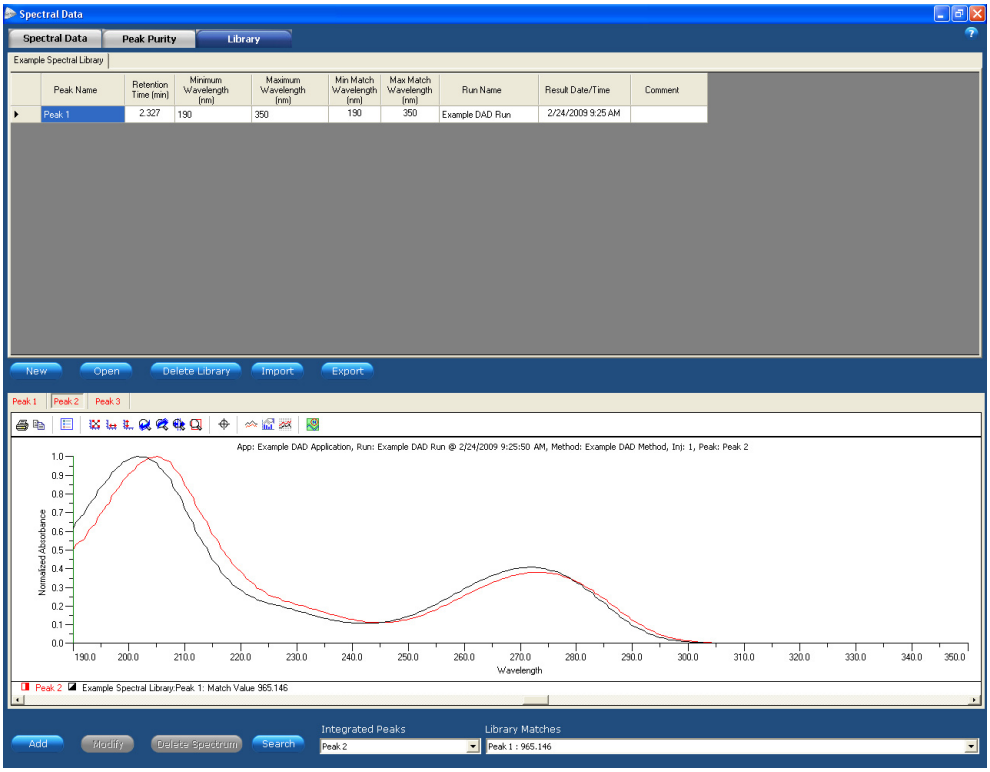


Figure 3-17: Spectral Data Window - Library Tab

Build a Spectral Library

1 To access the spectral libraries, do one of the following:
   - From the Run Results, click **Spectral Data** and then select the **Library** tab.
   - From the Project Library, double-click **DAD Spectral Libraries**.

2 Click **New** to create a new library. The New Spectral Library dialog appears.

3 In the New Spectral Library dialog:
   a) In the **Spectral Library Name** field, type a name.
   b) In the **Short Description** field (optional), type a brief description of the library.
   c) In the **Long Description** field (optional), type a detailed description of the library.
   d) Click **OK**. The new library opens.

Add a Peak Spectrum to Spectral Libraries

1 From the Run Results, click **Spectral Data** and then select the **Library** tab.

2 Select the name or number of the integrated peak from the drop-down list of Integrated Peaks.

3 Click **Add**. The Add Spectral Library Entry dialog appears.

4 In the Add Spectral Library Entry dialog:

a) In the **Peak Name** field, change the peak name, if desired. Peak names must be unique to ALL spectral libraries.

b) In the **Comment** field (optional), type a brief description of how data for the peak was acquired. For example, include mobile phase and column information.

c) In the **Comparison Wavelength Range** fields, specify the wavelengths TRILUTION LC will use when comparing the Spectral Library peak against an unknown peak. The default values identify the entire wavelength range collected during a run.

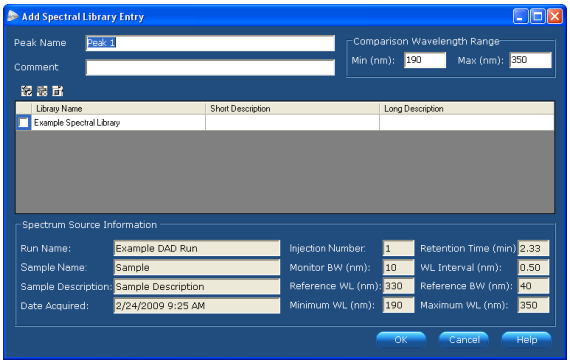


Figure 3-18: Add Spectral Library Entry Dialog

d) Select the check box next to the **Library Name** for each library to which the peak should be added.

e) Click **OK** to exit the Add Spectral Library Entry dialog and save the peak spectrum.

5 View the spectrum for a peak by clicking the table row in the Spectral Library.

Columns in the Spectral Library can be shown or hidden. Right-click on a column name and then select the column name from the menu to toggle it to show or to hide.

By default, columns for Peak Name, Retention Time, Minimum Wavelength, Maximum Wavelength, Min. Match Wavelength, Run Name, Result Date/Time, and Comment are shown in the Spectral Library.

Additional columns available to show are: Sample Name, Sample Description, Injection Number, Monitor Bandwidth, Reference Wavelength, Reference Bandwidth, Wavelength Interval, Application Name, Method Name, Wavelength Start, and Wavelength End.

Search for Spectral Matches

1 From the Run Results, click **Spectral Data** and then select the **Library** tab.

2 Select the name or number of one or more integrated peaks from the drop-down list of Integrated Peaks. As each peak is selected, a tab displays above the spectrum.

3 Click **Search**. The Search Spectral Library dialog appears.

4 In the Search Spectral Library dialog:

a) Select the check box next to the **Library Name** for each library to be searched.

b) For each peak spectrum to match, do one of the following:

- Select the check box next to the **Peak Name** and under **All Peaks** to search all Spectral Library peaks.
- Select the check box next to the **Peak Name** and under **Time Period** and then enter **Start** and **End** times to limit the search to Spectral Library peaks whose retention times are within that time period.

c) In the **Match Threshold** field, indicate the smallest match value to be reported. A match value of 1000 indicates a perfect match.

Figure 3-19: Search Spectral Library Dialog

5 Click **OK** to begin the search.

6 When the search completes, TRILUTION LC displays the five closest matches.

7 In the tabbed spectrum view, select a spectrum. From the drop-down list of **Library Matches**, select a spectrum to overlay. The integrated peak spectrum displays red, the matched spectrum displays black.

8 The peak spectrum and the spectral match are normalized. To view the originals, click .

9 To remove the overlaid spectrum, right-click on the spectrum tab and then select **Close Tab** to close the spectrum tab.

Modify a Spectral Library Entry

1 To open a Spectral Library, do one of the following:

- From the Run Results, click **Spectral Data** and then select the **Library** tab. Click **Open.** The Open Spectral Library dialog appears. Select the check box next to the **Library Name** for each library to open and then click **OK**.
- From the Project Library, double-click the Spectral Library name. The library opens.

2 Click to select a spectrum in the library. The spectrum opens graphically below in a tabbed view.

3 Click **Modify**. The Modify Spectral Library Entry dialog appears.

4 In the Modify Spectral Library dialog:

a) In the **Comment** field (optional), type a brief description of how data for the peak was acquired. For example, include mobile phase and column information.

b) In the **Comparison Wavelength Range** fields, specify the wavelengths TRILUTION LC will use when comparing the Spectral Library peak against an unknown peak. The default values identify the entire wavelength range collected during a run.

c) Select the check box next to the **Library Name** for each library to which the peak should be added.

d) In the **Minimum WL** and **Maximum WL** fields, specify the wavelength range for the spectrum to be saved to the library.

5 Click **OK**.

Delete Spectral Library entries

1 To open a Spectral Library, do one of the following:
- From the Run Results, click **Spectral Data** and then select the **Library** tab. Click **Open.** The Open Spectral Library dialog appears. Select the check box next to the **Library Name** for each library to open and then click **OK**.
- From the Project Library, double-click the Spectral Library name. The library opens.

2 Click to select a spectrum in the library. The spectrum opens graphically below in a tabbed view.

3 Click **Delete Spectrum**. The Select Spectral Library dialog appears.

4 In the Select Spectral Library dialog, select the libraries from which the open spectrum should be deleted.

5 Click **OK**.

Delete Spectral Libraries

1 To access the spectral libraries, do one of the following:
- From the Run Results, click **Spectral Data** and then select the **Library** tab.
- From the Project Library, double-click **DAD Spectral Libraries**.

2 Click **Delete Library**.The Delete Spectral Library dialog appears.

3 Select the check box next to the **Library Name** for each library to delete and then click **OK**.

Export Libraries

1 To access the spectral libraries, do one of the following:
- From the Run Results, click **Spectral Data** and then select the **Library** tab.
- From the Project Library, double-click **DAD Spectral Libraries**.

2 Click **Export** to display the Export window.

3 In the Export window:
a) In the **Select the items to be exported** field, select the spectral libraries.
b) Designate a path to which the libraries should be exported:
1) Click  to display the Browse For folder window.
2) On the Browse For folder window, select a folder and then click **OK**.

4 Click **OK**. On completion of the export operation, the spectral libraries are saved in the specified folder with a .LCSL extension.
a) When the export success/fail dialog appears:
1) To view the log information of the export operation, click **Details**.
2) Click **OK**.

Import Libraries

1 To access the spectral libraries, do one of the following:
- From the Run Results, click **Spectral Data** and then select the **Library** tab.
- From the Project Library, double-click **DAD Spectral Libraries**.

2 Click **Import** to display the Import window.

3 From the Import window:
a) Browse for and select the Spectral Library Export Files (.LCSL).
b) Click **Open**.

4 When the import success/fail dialog appears:
a) To view the log information of the import operation, click **Details**.
b) Click **OK**.

Maintenance 4

The procedures in this chapter should only be performed by trained personnel.

This chapter contains the following information to help you maintain your detector:

- Flow Cell Maintenance
- Lamp Maintenance
- Fuse Replacement

Note: Refer to Chapter 5, Troubleshooting to determine if your detector needs these maintenance procedures.

These procedures are the first line of defense against problems with the detector. Many problems can be corrected simply by cleaning the flow cell, replacing a lamp, or changing a fuse.

If these do not solve the problem, contact your local Gilson representative. Refer to Before Calling Us on page 5-6.

Flow Cell Maintenance

Follow these instructions if you need to clean the flow cell, unclog the flow cell, replace the inlet or outlet tubing, or replace the flow cell.

NOTICE Be extremely careful when working with the flow cell and its fittings. Flow cells are considered expendable and are not covered by warranty if damaged or broken during any maintenance procedure.

Clean the Inside of the Flow Cell

There are two ways to clean an active flow cell that has become coated with light absorbing material:

- solvent cleaning
- acid cleaning

Solvent Cleaning

If you suspect that droplets of an organic-soluble contaminant or immiscible solvent contaminate the active flow cell, draw or pump a series of miscible solvents through the cell.

If you were pumping a polar mobile phase, draw methanol, tetrahydrofuran, methylene chloride, and then methanol through the flow cell.

If you were pumping a non-polar mobile phase, draw hexane, isopropanol, methanol, then water through the flow cell.

⚠CAUTION To prevent injury, observe good laboratory practices when handling solvents. Know the physical and chemical properties. Refer to the Material Safety Data Sheets for the solvents used.

To clean the inside of the flow cell:

1 Disconnect the inlet and outlet tubing from your system.

2 Using a 1/4-28 coupler, connect a Luer-lock fitting to the fitting on the outlet tubing. Then attach a large glass syringe to the Luer-lock fitting.

3 Place the end of the inlet tubing into a reservoir of the first solvent of the series.

4 Draw solvent through the cell.

5 Repeat for each remaining solvent in the series.

6 Reconnect the inlet and outlet tubing to the system.

7 Check detector performance by running a sample through the system.

Acid Cleaning

If you suspect that residual proteins contaminate the flow cell, draw 50% nitric acid through the cell. Acid cleaning is a more thorough technique, though much more care must be taken.

⚠WARNING Take all necessary precautions so that acid does not come into contact with eyes, skin, clothing and equipment surfaces.

To clean the inside of the flow cell:

1 Disconnect the inlet and outlet tubing from your system.

2 Using a 1/4-28 coupler, connect a Luer-lock fitting to the fitting on the outlet tubing. Then attach a large glass syringe to the Luer-lock fitting.

3 Place the end of the inlet tubing into a reservoir of 50% nitric acid.

4 Draw nitric acid through the cell.

NOTICE Use nitric acid. Do not use halogenated acid. Halogenated acid will damage stainless steel.

⚠WARNING Do not pump acid through the cell. Never put acid under pressure.

5 After the acid flush, draw HPLC-grade water through the cell.

6 To be sure that all acid has been removed, monitor the pH of the final outflow.

7 Reconnect the inlet and outlet tubing to the system.

8 Check the detector performance by running a sample through the system.

Unclog the Flow Cell

To try to eliminate a blockage in the active cell or in its inlet or outlet tubing, pump solvent backwards through the flow cell.

1 Connect a high-pressure pump to the outlet and direct the inlet to waste.

2 For the pump, set the maximum pressure below 500 psi (34 bar).

3 Beginning at a flow rate of 0.1 mL/min, gradually increase the flow rate, in increments of 0.1 mL/min.

A significant pressure drop indicates that the blockage has been removed.

Replace the Flow Cell Assembly

Refer to the instructions in this section when replacing the entire flow cell assembly. To replace tubing and/or the flow cell individually, refer to Replace Tubing and/or the Flow Cell on page 4-5.

Refer to the table below for flow cell accessory kit part numbers. Each of the flow cell accessory kits includes a flow cell assembly with pre-connected inlet and outlet tubing, a plumbing package, and a 2.5 mm Allen wrench. The flow cell accessory kits for analytical applications also include a back pressure regulator.

Part number	*Description*
Analytical	
18004008	Flow cell accessory kit for analytical applications (with stainless steel inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
18004007	Flow cell accessory kit for analytical applications (with PEEK inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
Preparative	
18004006	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.2 mm pathlength, 0.7 µL volume, quartz
18004005	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.05 mm pathlength, 0.16 µL volume, quartz

To replace the entire flow cell assembly:

1 Open the front door of the detector.

2 Disconnect the fiber optic cable from the left side of the flow cell assembly. The protective cap can be placed over the end of the fiber optic cable while it is not connected to the flow cell assembly.

NOTICE Be careful not to touch the connector ends of the fiber optic cables.

3 Disconnect the fiber optic cable from the right side of the flow cell assembly. Place the protective cap over the end of the fiber optic cable.

4 Unscrew the two thumbscrews on the flow cell assembly bracket and remove the flow cell assembly.

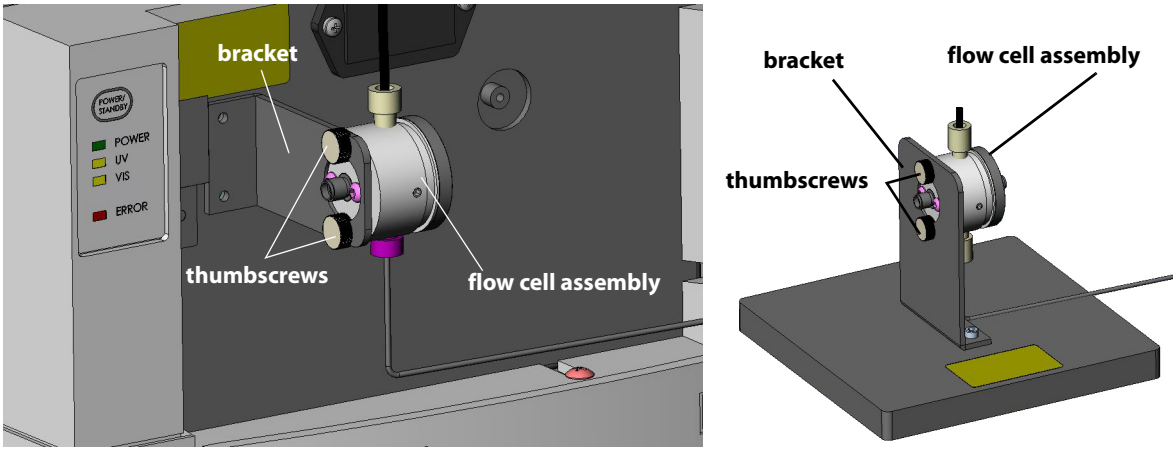


Figure 4-1: Flow Cell Assembly Installed on the Detector (Left) and on the Remote Flow Cell Holder (Right)

5 Align the holes in the replacement flow cell assembly with the holes on the flow cell assembly bracket.

6 Replace the two thumbscrews.

7 Re-connect the SMA connectors to the flow cell assembly.

8 Close the front door of the detector.

Replace Tubing and/or the Flow Cell

Refer to the instructions in this section when replacing tubing and/or the flow cell individually. To replace the entire flow cell assembly, refer to Replace the Flow Cell Assembly on page 4-3.

All of the flow cells fit into the same flow cell holder; however, the inlet and outlet tubing varies. Refer to Appendix B, Replacement Parts for ordering information.

The steps in this section should be followed in the order shown below.

1 Remove the Flow Cell Assembly

2 Remove the Flow Cell

3 Replace the Flow Cell

4 Replace the Outlet Tubing

5 Replace the Inlet Tubing

6 Check for Leaks

7 Replace the Flow Cell Assembly

Remove the Flow Cell Assembly

To remove the flow cell assembly from the detector or from the remote flow cell holder:

1 Open the front door of the detector.

2 Disconnect the fiber optic cable from the left side of the flow cell assembly.

The protective cap can be placed over the end of the fiber optic cable to protect the end of the cable while it is not connected to the flow cell assembly.

3 Disconnect the fiber optic cable from the right side of the flow cell assembly. Place the protective cap over the end of the fiber optic cable.

4 Unscrew the two thumbscrews on the flow cell assembly bracket and remove the flow cell assembly.

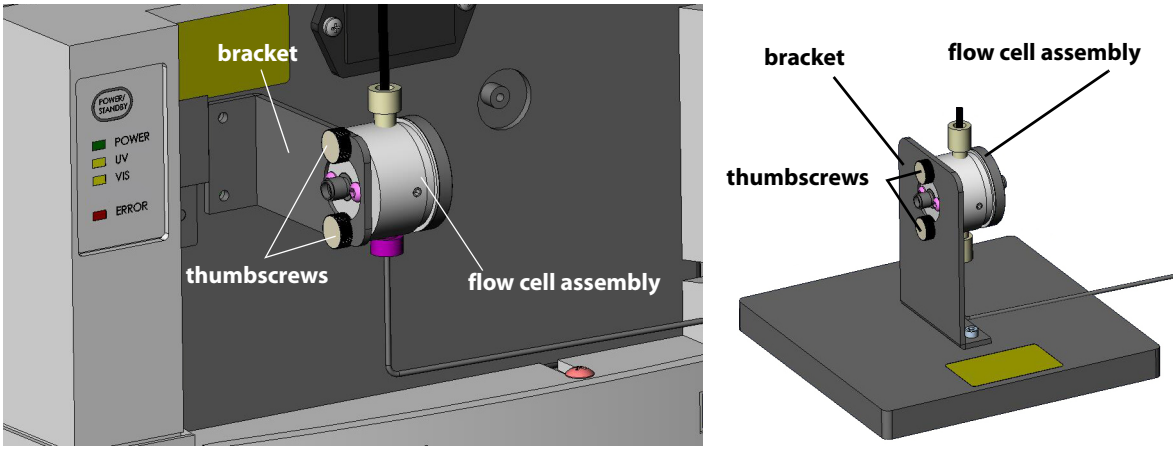


Figure 4-2: Flow Cell Assembly Installed on the Detector (Left) and on the Remote Flow Cell Holder (Right)

Remove the Flow Cell

It is recommended to have a tray in which to place the parts while disassembling the flow cell assembly.

To remove the flow cell from the flow cell assembly:

1 Loosen the fittings for the inlet and outlet tubing. On the flow cell assembly, the inlet tubing port is labeled IN and the outlet tubing port is labeled UP.

2 Using the 2.5 mm Allen wrench, remove the three screws from the cover of the flow cell holder and then remove the cover.

3 Using the 2.5 mm Allen wrench, loosen the set screw on the left side of the flow cell holder.

4 Carefully remove the flow cell using a pair of tweezers or by tilting the flow cell holder until the flow cell drops into the palm of your hand.

There are two gaskets, one on each side of the flow cell. It is recommended to replace these gaskets when replacing the flow cell.

- The black gasket (part number 18001024) fits between the flow cell holder and the flow cell.
- The gray gasket (part number 18001020) fits between the flow cell and the cover.

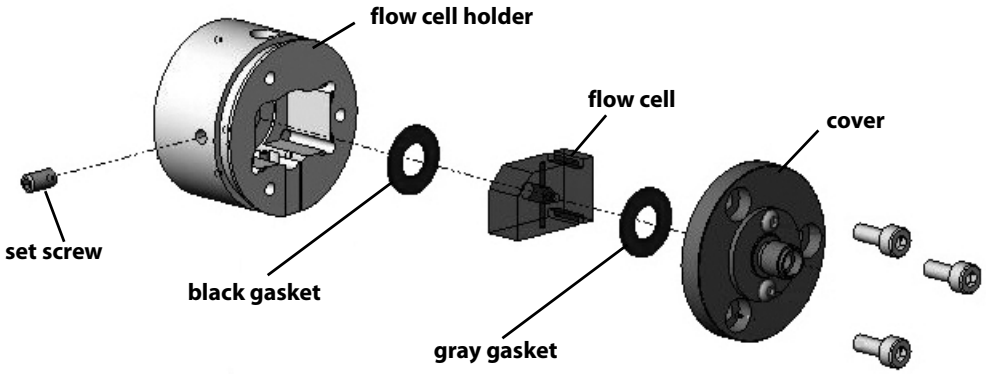


Figure 4-3: Flow Cell Assembly

Replace the Flow Cell

Refer to the table below for part numbers for replacement flow cells.

Part number	*Description*
100405	Analytical flow cell (5 mm pathlength, 12 µL volume, quartz)
100401	Preparative flow cell (0.2 mm pathlength, 0.7 µL volume, quartz)
100407	Preparative flow cell (0.05 mm pathlength, 0.16 µL volume, quartz)

To replace the flow cell:

1 Before inserting the replacement flow cell, dampen the quartz windows with alcohol or a laboratory cleaning solution. Then wipe the windows with a piece of lens paper or lint-free towel.

2 Wipe the inside of the flow cell holder with a lint-free towel soaked in alcohol.

3 Carefully dry all surfaces of the assembly.

4 Place the black gasket (part number 18001024) in the inner recessed circle on the flow cell holder.

5 Without touching the quartz windows, insert the flow cell into its chamber. Push the cell into the chamber as far as it will go. A poorly aligned flow cell may restrict or block flow.

6 Using the 2.5 mm Allen wrench, tighten the set screw.

7 Place the gray gasket (part number 18001020) in the inner recessed circle on the cover and then place the cover over the flow cell holder. Align the groove on the lower half of the flow cell holder with the groove on the lower half of the cover.

8 Using the 2.5 mm Allen wrench, replace the three screws.

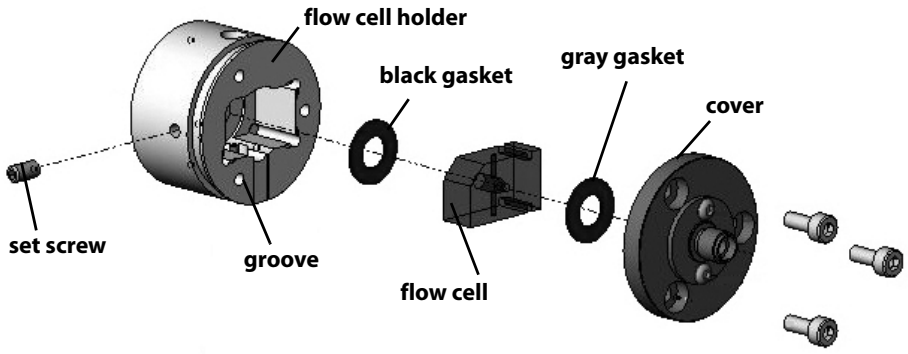


Figure 4-4: Flow Cell Assembly

Replace the Outlet Tubing

When replacing the outlet tubing, ensure that its ID is appropriate for the flow cell. Refer to the table below for part numbers for replacement tubing.

Part number	*Description*
18007001	Analytical outlet tubing, 0.3 mm (0.010") ID Teflon, with fittings
18007002	Preparative outlet tubing, 1.0 mm (0.040") ID Teflon, with fittings

To replace the outlet tubing:

1 Disconnect the outlet tubing from the flow cell holder and, if necessary, from the other HPLC system component.

2 Connect the fitting on the replacement tubing to the flow cell holder. Finger tighten the fitting.

3 Connect the other end of the tubing, if necessary.

Replace the Inlet Tubing

When replacing the inlet tubing, ensure that its ID is appropriate for the flow cell. Refer to the table below for part numbers for replacement tubing.

Part number	*Description*
18007004	Analytical inlet tubing, 0.3 mm (0.010") ID stainless steel, with fittings to connect to flow cell
18007005	Analytical inlet tubing, 0.3 mm (0.010") ID PEEK, with fittings to connect to flow cell
18007006	Preparative inlet tubing, 1.0 mm (0.040") ID stainless steel, with fittings to connect to flow cell

To replace the inlet tubing:

1 Disconnect the inlet tubing from the flow cell holder and the column.

2 Connect the fitting on the replacement tubing to the flow cell holder. Finger tighten the fitting.

3 Connect the other end of the tubing to the column outlet.

Check for Leaks

To determine whether the fittings are leak-free:

1 Use a syringe to initiate flow through the cell.

2 Check carefully along the sides of the cell for beads of liquid. A flashlight may be helpful.

3 If leakage is detected visually, or if the area around the fitting feels cool to the touch, remove and clean the flow cell, as well as its chamber. Then re-insert the flow cell and re-tighten the fittings.

Replace the Flow Cell Assembly

To replace the flow cell assembly on the detector or on the remote flow cell holder:

1 Align the holes in the flow cell assembly with the holes on the flow cell assembly bracket.

2 Replace the two thumbscrews.

3 Re-connect the fiber optic cables to the flow cell assembly and to the detector.

4 Route the tubing through one of the cutouts on the lower left or right of the front door of the detector.

5 Close the front door of the detector.

Lamp Maintenance

⚠CAUTION

Caution: To avoid possible electric shock, disconnect the power cord before servicing the instrument.

Attention: Afin d'éviter toute possibilité de commotion électrique, débranchez le cordon d'alimentation de la prise avant d'effectuer la maintenance de l'instrument.

Vorsicht: Zur Vermeidung von Stromschlägen sollte das Gerät vor der Wartung vom Netz getrennt werden.

The detector contains a deuterium lamp for monitoring UV wavelengths and a tungsten lamp for monitoring visible wavelengths.

Both lamps have an expected lamp life of 2000 hours.

⚠CAUTION Always wear protective eye wear when looking at a UV-radiating lamp. Prescription glasses are acceptable, if they have Plasticon lenses. Plastic safety goggles provide maximum protection.

⚠CAUTION Never touch a hot lamp. After turning off the lamps, always wait 30 minutes or more before replacing a lamp.

Prepare the Detector

To prepare the detector for lamp maintenance:

1 Turn off the detector using the rear panel MAINS (power) switch.

2 Unplug the power cord from the detector and from the outlet.

3 Remove the two screws on the sides of the detector and the six screws from the back and then lift the cover off the detector. The lamps are located on the right side, near the front.

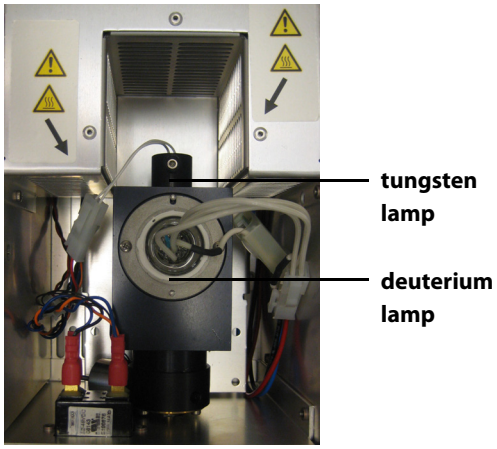


Figure 4-5: Lamp Locations

Deuterium (UV) Lamp Replacement

Before beginning this procedure, ensure that the detector is powered OFF and that the power cord has been removed from the detector and outlet.

1 Locate the deuterium lamp and disconnect the lamp connector.

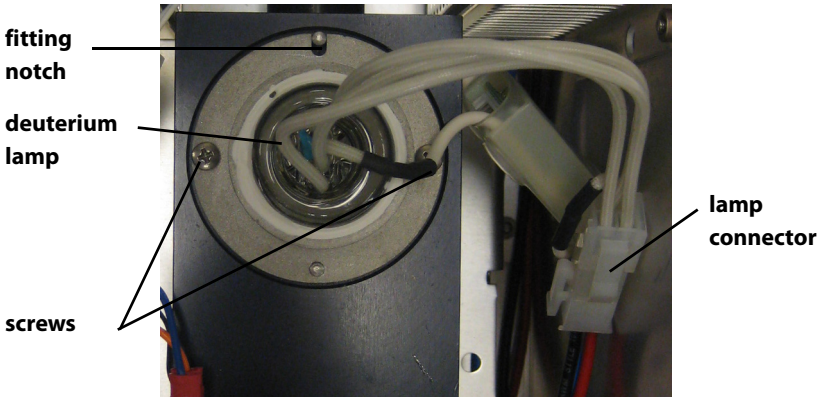


Figure 4-6: Deuterium Lamp Replacement

2 Using a #1 Phillips screwdriver, remove the two screws holding the deuterium lamp in place.

3 Grasp the lamp by its base and remove the lamp from the lamp housing. Keep the lamp vertical while pulling it out. Otherwise, the fitting notch may get stuck.

⚠CAUTION Always handle the lamp by the base, avoiding contact with its glass window. A hot lamp can cause a severe burn, and fingerprints can etch the window. To clean a lamp, use a soft cloth moistened with alcohol.

4 Make sure the replacement lamp is free of fingerprints and other blemishes.

5 Install the replacement lamp and replace the screws.

The deuterium lamp should be oriented so the fitting notch fits over the post nearest the tungsten lamp (and the rear of the detector). If the deuterium lamp is not oriented correctly, the screw holes in the lamp assembly will not align with the screw holes in the lamp socket.

6 Re-connect the leads of the new lamp and replace the screws.

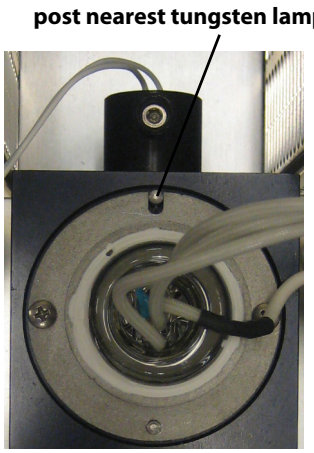


Figure 4-7:
Deuterium Lamp Orientation

Tungsten (VIS) Lamp Replacement

Before beginning this procedure, ensure that the detector is powered OFF and that the power cord has been removed from the detector and outlet.

1 Locate the tungsten lamp and disconnect the lamp connector.

2 With your fingers, loosen the plastic thumbscrew holding the tungsten lamp in place. Do **NOT** use an Allen wrench to loosen the thumbscrew.

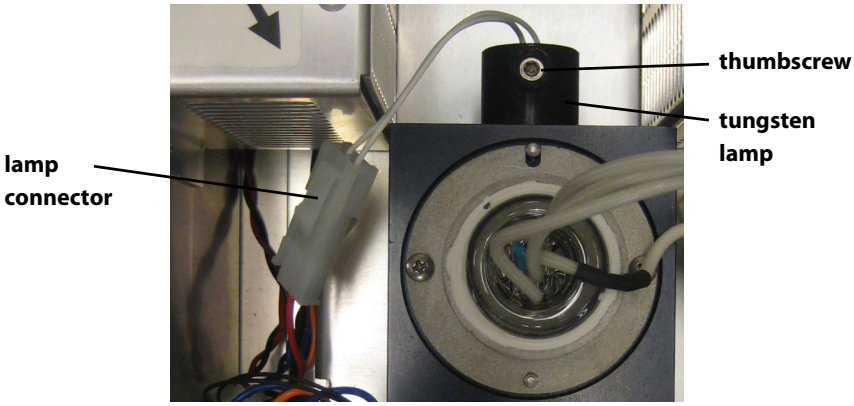


Figure 4-8: Tungsten Lamp Replacement

3 Pull the tungsten lamp out.

4 Insert the replacement tungsten lamp. Take care not to touch the lamp glass bulb.

5 Look at the black line drawn on the lamp near the wire connections. Turn the lamp so that this line is vertical.

6 Tighten the thumbscrew.

7 Re-connect the lamp connector.

Lamp Timer Reset

After the deuterium (UV) lamp has been replaced, the lamp timer needs to be reset.

1 Place the cover on top of the detector and then slide the cover back about two inches as shown in the photo below.

Figure 4-9: Top View of Detector

2 Re-connect the power cord to the detector and then to a power source.

3 Press and hold the lamp reset button for 1–2 seconds to set the lamp hours back to zero.

4 Replace the cover on the detector.

Check Out the New Lamp

1 Replace the cover on the detector.

2 Plug the power cord of the detector into an outlet.

3 Turn on MAINS (power) switch.

4 Use TRILUTION® LC Software to turn on the detector lamps. Refer to Turn Lamps On on page 3-3.

If the lamp fails again, there may be an internal alignment, a power supply, or a starting circuitry problem. Contact your local Gilson representative; refer to Before Calling Us on page 5-6.

Fuse Replacement

To change a fuse:

1 Turn off the detector using the rear panel MAINS (power) switch.

2 Disconnect the power cord from the power outlet and from the rear panel receptacle.

3 Locate the fuse drawer on the rear panel.

4 Insert a small screwdriver into the notch under the fuse drawer.

5 Using the screwdriver, lift up to remove the fuse drawer. The fuse drawer contains two 1.60A "TD" type fuses.

6 Remove the old fuse(s) and insert the new fuse(s).

7 Insert the fuse drawer into its receptacle in the detector.

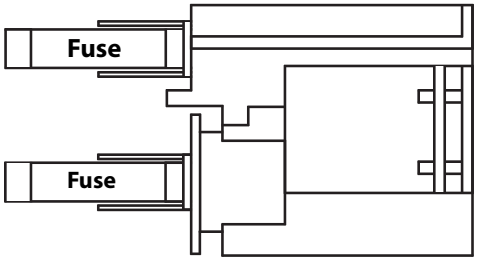


Figure 4-10: Fuses in Fuse Drawer

A blown fuse may indicate the existence of another problem in the instrument. If the replacement fuses blow, do not try others. Contact your local Gilson representative. Refer to Before Calling Us on page 5-6.

⚠CAUTION

Caution: To protect against fire hazard, replace fuses with those of the same type and rating.

Attention: Remplacez toujours les fusibles par d'autres du même type et de la même puissance afin d'éviter tout risque d'incendie.

Vorsicht: Zum Schutz gegen Feuergefahr die Sicherungen nur mit Sicherungen des gleichen Typs und Nennwertes ersetzen.

Troubleshooting 5

This chapter provides information on the following topics:

Troubleshooting

No POWER LED

- Check cord and power connection.
- Fuses may be blown. Refer to Fuse Replacement on page 4-12.

If the problem persists, contact your local Gilson representative.

ERROR LED is On

- Turn the MAINS (power) switch OFF then ON again. After the detector is powered ON, restart TRILUTION® LC Software.

  If the ERROR LED is still on after restarting the detector and TRILUTION LC, contact your local Gilson representative.

Note: The ERROR LED may flash briefly as the detector is powered ON and also when the lamps are turned on.

A Lamp Fails to Ignite/A Lamp Fails While On

- Check for loose lamp connector or replace lamp. Refer to Lamp Maintenance on page 4-9.

If the problem persists, contact your local Gilson representative.

Baseline Spikes

- Be sure solvents are degassed.
- Check fittings for a leak in system. Repair as needed.
- Reduce pump noise by changing the compressibility setting on the pump and by examining check valves.
- Check grounds on all instruments. Eliminate ground loops by connecting the detector and recorder to the same power outlet.

Baseline Noise

- Allow each lamp to warm up for at least 15–30 minutes.
- Eliminate drafty air currents and variations in ambient temperature. Move the detector away from windows or doors.
- Eliminate smoke from the environment.
- Check grounds on all instruments. Eliminate ground loops by connecting the detector and recorder to the same power outlet.
- Be sure solvents are degassed.
- Check fittings for a leak in system. Repair as needed.
- If you see baseline deflection when back pressure is exerted on the flow cell, you probably have bubbles trapped in the cell. Try one of these methods to eliminate bubbles:
  - Increase back pressure on the cell by connecting pressure regulator tubing to the flow cell outlet.
  - Increase back pressure by raising flow rate and using a syringe or septum to momentarily block exit stream.
  - Flush flow cell using a syringe filled with fresh solvent.
  - Raise the fraction collector to a level higher than the flow cell.
- A lamp may be weak. Refer to Lamp Maintenance on page 4-9.
- Check flow cell for contamination. Refer to Flow Cell Maintenance on page 4-2.

Baseline Drift

- Allow each lamp to warm up for at least 15–30 minutes.
- Eliminate changes in ambient temperature. If drift is dependent on temperature, eliminate drafts and avoid areas of direct sunlight.
- Check for contaminated or "bleeding" column. If drift disappears after removing column connections, regenerate or replace column.
- Be sure solvents are degassed.
- Check fittings for leak in system. Repair as needed.
- If you see baseline deflection when back pressure is exerted on the flow cell, you probably have bubbles trapped in the cell. Try one of these methods to eliminate bubbles:
  - Increase back pressure on the cell by connecting pressure regulator tubing to the flow cell outlet.
  - Increase back pressure by raising flow rate and using a syringe or septum to momentarily block exit stream.
  - Flush flow cell using a syringe filled with fresh solvent.
  - Raise the fraction collector to a level higher than the flow cell.
- Check pump or delivery system to determine that flow rate is constant.
- A lamp may be weak. Refer to Lamp Maintenance on page 4-9.
- Check flow cell for leak or contamination. Refer to Flow Cell Maintenance on page 4-2.

Ethernet Connections

For communication to occur, the instrument must be connected via an Ethernet connection to a router. If there are communication problems between the instrument and the computer, verify that the procedure below has been followed.

1 Connect the router to a power source and then wait 30 seconds for the router to initialize.

2 With the instrument powered OFF, connect one end of an Ethernet cable to a port on the router and the other end to the ETHERNET port on the rear panel of the instrument.

3 Power the instrument ON and then wait 1 minute for it to initialize. The instrument will be assigned an IP address via DHCP from the router at this time.

4 Using an Ethernet cable, connect one end of the cable to the connector on the computer and the other end to a port on the router.

Ethernet Communication

After the connections have been verified, use the procedure below to confirm Ethernet communication.

1 Close software programs controlling Gilson system.

2 Turn off power to the router and the Gilson system.

3 Wait five minutes.

4 Turn power on to the router.

5 Turn power on to the Gilson system.

6 Start the control software.

Mobile Phase Tips

Degas Solvents and Buffers

Always use degassed, HPLC-grade solvents (including water) and buffers for your mobile phase.

If the back pressure on the flow cell is sufficiently low, gas bubbles may still appear, even when using degassed solvents. You can avoid this by using a back pressure regulator. The flow cell accessory kits for analytical flow cells include a back pressure regulator. Refer to Back Pressure Regulator on page 2-8 for installation instructions.

⚠CAUTION To prevent injury, observe good laboratory practices when handling solvents. Know the physical and chemical properties. Refer to the Material Safety Data Sheets for the solvents used.

Check UV and Visible Absorbance of Solvents

Remember to use only HPLC-grade solvents for your mobile phase. Impure solvents can absorb significant quantities of UV or visible light even when the cutoff may be below the monitoring wavelength. This can result in an inability to accurately set the baseline to zero.

Manufacturer's specifications list the UV and visible cutoff for a solvent. This is the wavelength below which a solvent should not be used for UV or visible detection. The cutoff is the wavelength at which the solvent is 90% opaque (absorbance 3, 1.0 AUFS). Interference can still occur above the rated limit, particularly when the solvent composition changes during a run. This frequently appears as a gradual baseline shift.

The table below lists commonly used solvents with low UV and visible cutoffs.

Solvent	*UV/VIS Cutoff*	*Solvent*	*UV/VIS Cutoff*	*Solvent*	*UV/VIS Cutoff*
Acetonitrile	190 nm	Hexadecane	190 nm	Pentane	205 nm
Cyclohexane	200 nm	Hexane	195 nm	i-Pentanol	208 nm
Cyclopentane	200 nm	Hexanes	210 nm	i-Propanol	205 nm
Decahydronaphthalene	200 nm	Isooctane	197 nm	n-Propanol	240 nm
Ethanol	210 nm	Methanol	205 nm	Tetrahydrofuran	212 nm
Heptane	200 nm	Methylene chloride	233 nm	Water	<190 nm

Repair and Return Policies

Before Calling Us

Your local Gilson representative will be able to serve you more efficiently if you have the following information:

- the serial number and model number of the instruments involved. The serial number is located on the door frame and is visible when the door is open.
- the installation procedure you used
- list of concise symptoms
- list of operating procedures and conditions you were using when the problem arose
- list of other devices connected to the detector and a description of those connections
- list of other electrical connections in the room

Warranty Repair

Units covered under warranty will be repaired and returned to you at no charge. If you have any questions about applicability, please contact your local Gilson representative.

Non-Warranty Repair

For out-of-warranty repairs, contact your local Gilson representative who will discuss service options with you and can assist in making arrangements to return the equipment, if necessary.

Return Procedure

Contact your local Gilson representative to obtain authorization before returning any Gilson equipment. To return a piece of equipment:

- Carefully pack the unit to prevent damage in transit. Check with your local Gilson representative regarding proper method of shipment. No responsibility is assumed by Gilson or your local Gilson representative for damage caused by improperly packaged instruments. Indicate the authorization on the carton and on the packing slip.
- Always insure for the replacement value of the unit.
- Include a description of symptoms, your name, address, phone number, and purchase order to cover repair costs, return and shipping charges, if your institution requires it.

Unit End-of-Life

When a unit reaches the end of its useful life, refer to www.gilson.com for directions and information on the end-of-life policy. This is in accordance with the European Union Directive 2002/96/EC on Waste Electrical and Electronic Equipment (WEEE).

Exercises A

The following exercises take you through reviewing spectral data. From the exercises, you will also learn how to create a Spectral Library and perform spectral matching. The examples were created from a chromatographic run of samples containing three compounds.

The following exercises are included in this chapter:

Example Results are supplied on the 171/172 Diode Array Detectors Documentation CD.

Import Example Results

An example result file (.LCRA) is provided on the 171/172 Diode Array Detectors Documentation CD to illustrate the recommended method setup and to give an example of the acquired data. By importing the results, the run results are imported and the method used to generate the results can be extracted.

To import (restore) the example file to TRILUTION LC:

1 Copy the Example TLC vx.x Results folder on the 171/172 Diode Array Detectors Documentation CD to …\My Documents\TRILUTION LC x.x\Export. TRILUTION LC x.x indicates the version of TRILUTION LC.

2 Start TRILUTION LC and then log on.

3 Click **Project Library**.

4 In the Project Library, create a new Project (i.e., Example Project).

5 Create a new Application in the new Project (i.e., Example Application).

6 Open the Results window. The Results window is accessed by right-clicking on an Application in the Project Library and then choosing Results or by double-clicking the Results icon ( ).

7 On the Results window, click the Restore Run button ( ) to display the Import window.

8 On the Import window:

a) Browse for and then select the Result Export File EXAMPLE DAD RUN.LCRA located at …\My Documents\TRILUTION LC x.x\Export\Example TLC vx.x Results.

b) Click **Open**.

9 When the import success/fail dialog appears, click **OK**.

10 In the Results window, click **Refresh** to show the imported example results.

Extract Method

1 Right-click on the Run Name in the Results window and then select **Extract Methods** to display the Extract Methods dialog.

2 On the Extract Methods dialog, click **OK** to extract the selected method and display the Import dialog.

3 On the Import dialog, click **Overwrite All** and then click **Import**. When asked to confirm the overwrite, click **Yes**.

4 In the Project Library, click **Refresh** to show the extracted method.

Exercise 1 - Review Data

Using the options in the Spectral Data window, you can review spectral information.

1 Access results by right-clicking on an application in the Project Library and then choosing **Results** or by double-clicking the Results icon ( ). The Results window appears. Double-click the run name to show the run results.

When the Run Results window appears, you see the trace for the data channel that was marked for use in the configuration. This is also the trace that was displayed real-time.

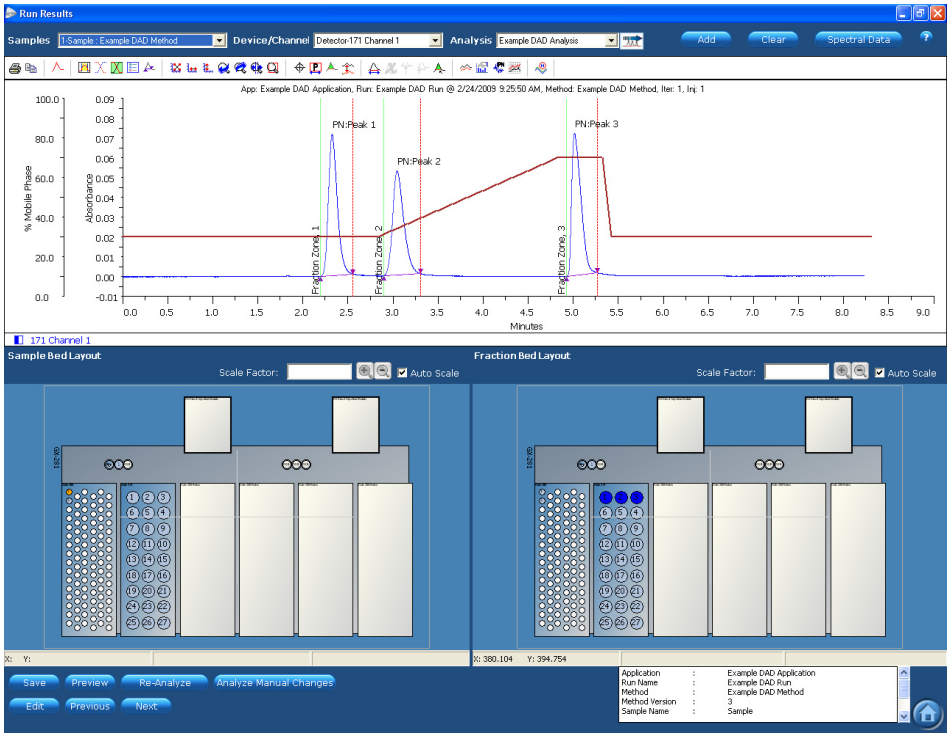


Figure A-1: Run Results Window

2 Click the **Spectral Data** button. The Spectral Data window appears for viewing chromatogram and spectral plots and creating spectral libraries using data collected by the 171 or 172 Diode Array Detector.

3 View the peak purity values for each of the integrated peaks by selecting the Peak Purity tab. Peak Purity values are preceded by **PP:**. If TRILUTION LC calculates a peak purity that is below the Purity Threshold value set in the Peak Purity options in the method, the peak is filled red. If the calculated peak purity is above the Purity Threshold value, the peak is filled green.

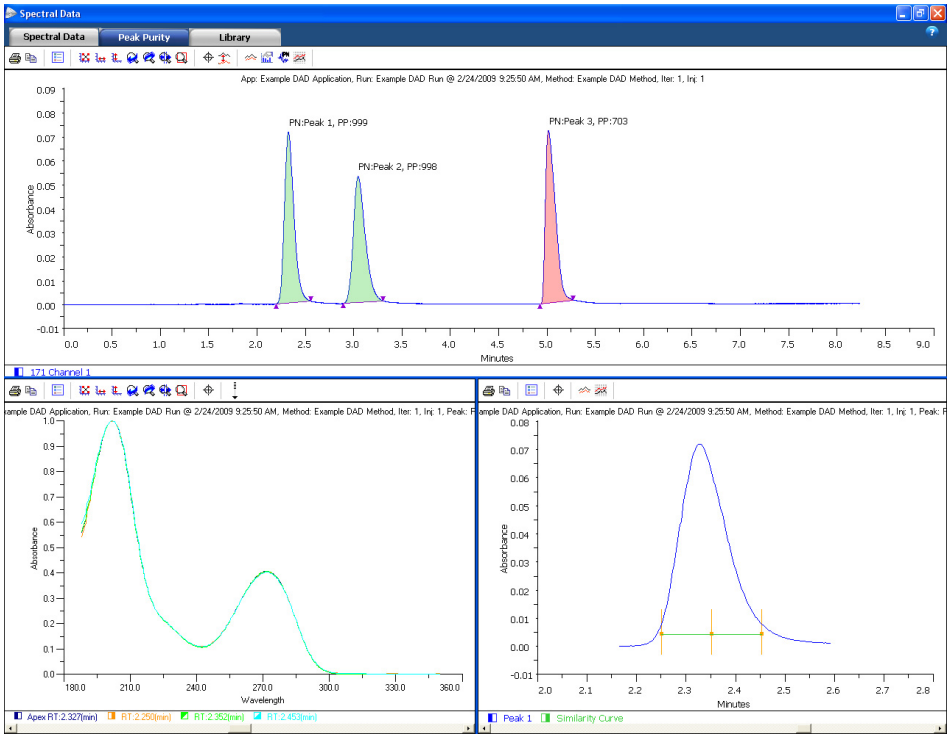


Figure A-2: Spectral Data Window - Peak Purity Tab

View the peak purity values in the Analysis Report, by adding the Peak Purity column to Report Type Sample.

Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (mAUmin x100)	Height (Absorbance)	Sample Location	Fraction Site(s)	Peak Purity
1	Peak 1	2.327	813.1354	0.071	Sample Zone->1	Fraction Zone-1	999.294
1	Peak 2	3.048	750.4139	0.052	Sample Zone->1	Fraction Zone-2	998.29
1	Peak 3	5.02	847.3379	0.072	Sample Zone->1	Fraction Zone-3	703.031

Figure A-3: Analysis Report - Sample Table

Display and Update the Spectrum Plot

1 Display a spectrum.

a) In the Spectral Data window, click to select the **Spectral Data** tab.

By default, the spectrum displayed in the upper right is the APEX for:

- the first named peak in the peak table found
- or the first integrated peak found (if no peaks were named in the Peak Table)

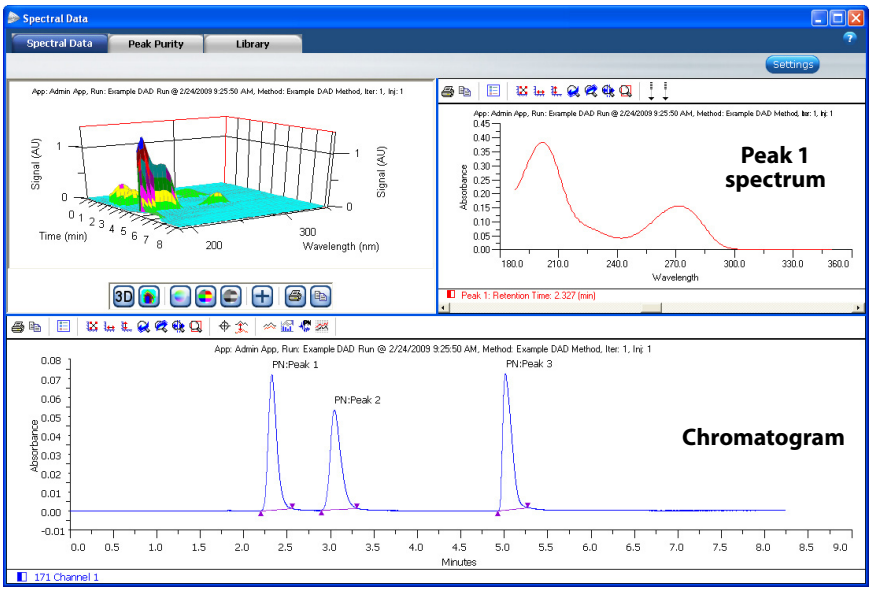


Figure A-4: Spectral Data Window - Spectral Data Tab

b) In the Chromatogram toolbar, click to select the cross hair tool ( ) which displays the Time and Absorbance at the intersect of the cross hair.

c) Move the mouse pointer to a location within Peak 3. Notice how the time and absorbance readings update. Double-click. The spectrum at the apex of Peak 3 appears in the upper-right corner.

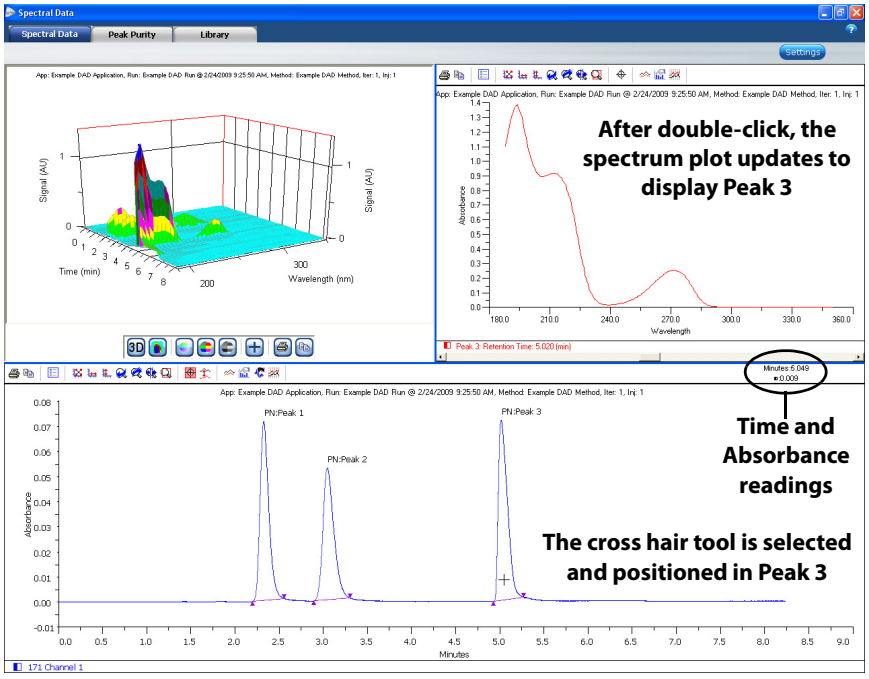


Figure A-5: Spectral Data Window - Spectral Data Tab

2 Display the spectrum at the APEX for a different peak by double-clicking in the peak on the chromatogram.

Display and Update the Iso-electric Plot

1 Display the iso-electric plot:

a) In the Spectral Data window, click to select the **Spectral Data** tab.

b) Click ( ) to show the iso-electric plot.

2 Use the iso-electric plot to update the chromatogram plot and spectrum plot.

a) Click to select the cross hair tool ( ) which displays the Time, Wavelength, and Absorbance at the intersect of the cross hair.

b) Move the mouse pointer over the iso-electric plot. Notice how the time, wavelength, and absorbance readings update. Click to update the wavelength of the overlaid trace on the chromatogram plot and the retention time on the spectrum plot.

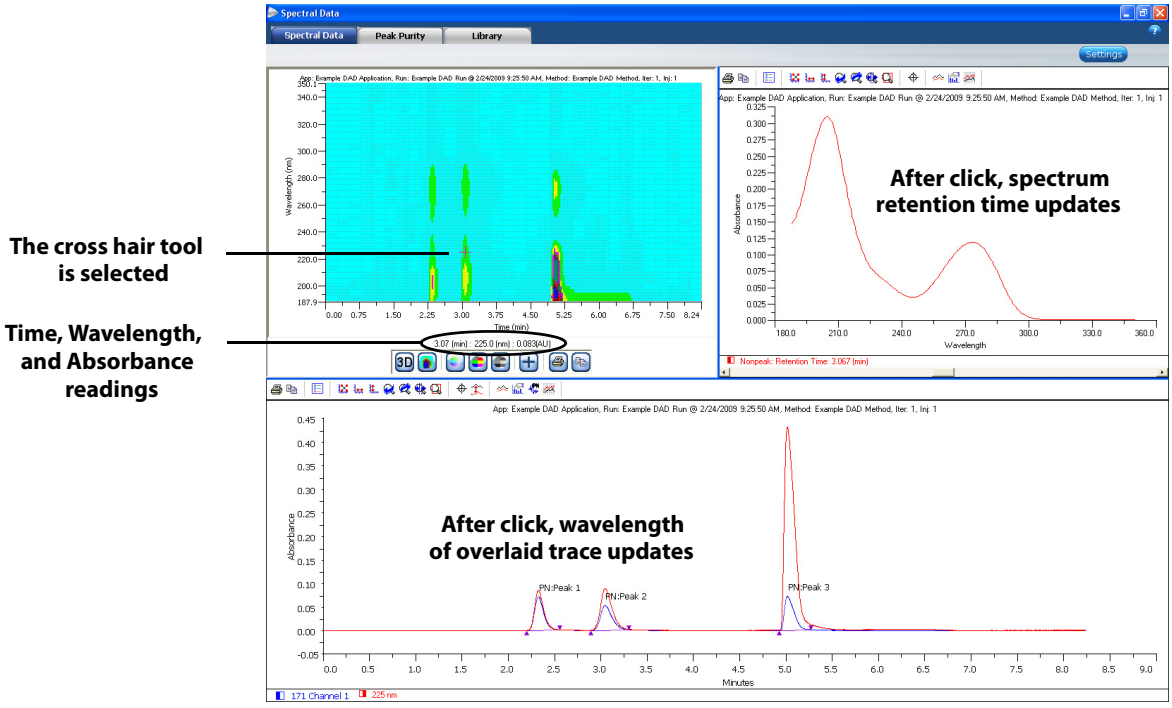


Figure A-6: Spectral Data Window - Spectral Data Tab

3 Reduce and enlarge the iso-electric plot.

a) To zoom in, right-click on the plot.

b) To zoom out, right-click outside the plot on either axis.

Display and Update the 3D Plot

1 Display the 3D plot.

a) In the Spectral Data window, click to select the **Spectral Data** tab.

b) Click ( ) to show the 3D plot. Note that the 3D plot is displayed by default. You only need to click the icon if the iso-electric plot is being displayed.

Display options for the legend and updating the 3D plot by right-clicking next to the toolbar and then selecting **Control Panel**.

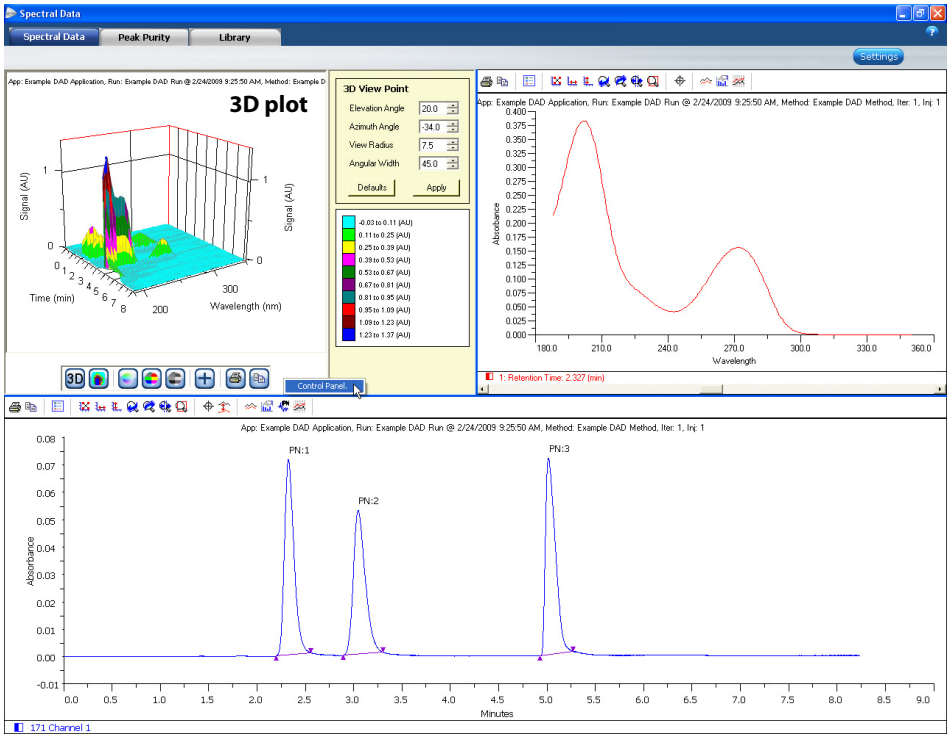


Figure A-7: Spectral Data Window - Spectral Data Tab

2 The default displayed wavelength range is 190–350 nm. Display the traces for the wavelength range from 200–300 nm.

a) Close the Spectral Data window by clicking the x in the upper right corner. The Run Results window appears.

b) On the Run Results window, click **Edit**. The Result Analysis Builder appears.

c) Click to select the Report tab.

d) On the Report tab, select the **Spectrum** check box and then click **Spectrum Settings...**. The Spectrum Settings dialog appears.

e) On the Diode Array Spectrum tab of the Spectrum Settings dialog, change the Minimum Wavelength to 200 and the Maximum Wavelength to 300 then click **OK**.

f) Click **OK** to exit the Result Analysis Builder.

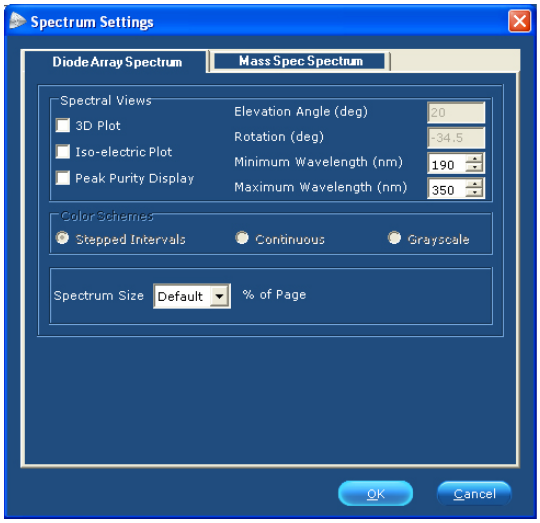


Figure A-8: Spectrum Settings Dialog

g) Click the **Spectral Data** button to access the Spectral Data window and Spectral Data tab where the 3D plot is displayed showing the updated wavelength range.

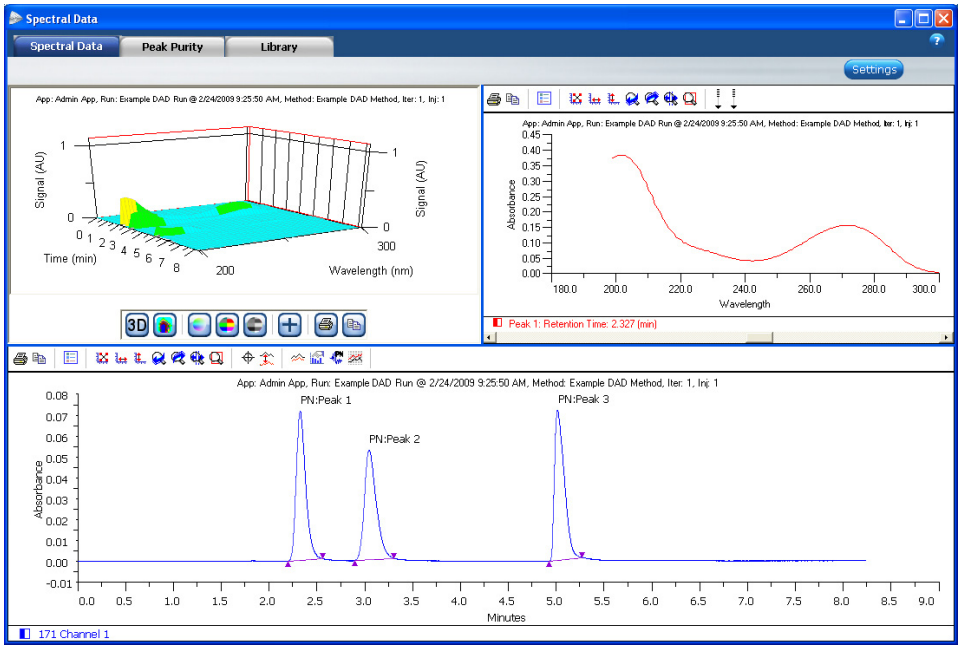


Figure A-9: Spectral Data Window - Spectral Data Tab

3 Return the wavelength axis to the previous minimum and maximum values by repeating step 2 and changing the values accordingly.

4 Reduce and enlarge the 3D plot.

a) Zoom toward the center of the plot by right-clicking while dragging the mouse downward.

b) Zoom away from the center of the plot by right-clicking while dragging the mouse upward.

View Spectral Data for Another Sample

View the 3D plot, chromatogram plot, and spectrum plot for another sample.

1 Close the Spectral Data window by clicking the x in the upper right corner. The Run Results window appears.

2 On the Run Results window:

a) Select a different sample from the **Samples** drop-down list.

b) Select the data channel from the **Device/Channel** drop-down list and the Analysis from the **Analysis** drop-down menu.

3 Click the **Spectral Data** button to view the chromatogram and spectral plots for the selected sample.

4 Repeat for any other samples.

Exercise 2- Modify Peak Purity Options

Peak Purity is a spectral comparison of the apex spectrum to spectra from across the peak. This comparison is used to check the purity of the peak.

Set values pre-run in the Method Builder as described below or set values post-run and prior to a re-analysis as described in the Modify Peak Purity Options Post-Run example on page A-10.

1 Open the method and click to select the Analysis tab.

2 Click **DAD Peak Purity**. The Peak Purity dialog appears.

3 In the **Purity Threshold** field, change the Purity Threshold value to 990.

4 Add peak purity values to the Analysis Report.

a) With the method still open, click to select the Report tab.

b) Under Report Type, click to select Sample.

c) Under Contents, click to select Peak Purity.

d) Click the right arrow ( ) to add it to the bottom of the Column Contents list.

5 After the run is complete, view the Analysis Report (either from the Results or the Reports menu). A purity value will be reported for all peaks.

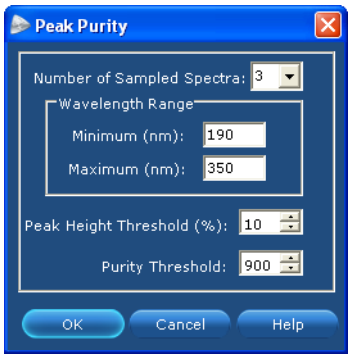


Figure A-10: Peak Purity Dialog

Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (mAUmin x100)	Height (Absorbance)	Sample Location	Fraction Site(s)	Peak Purity
1	Peak 1	2.327	813.1354	0.071	Sample Zone->1	Fraction Zone-1	999.294
1	Peak 2	3.048	750.4139	0.052	Sample Zone->1	Fraction Zone-2	998.29
1	Peak 3	5.02	847.3379	0.072	Sample Zone->1	Fraction Zone-3	703.031

Figure A-11: Analysis Report - Sample Table

View the effects of the Purity Threshold setting by accessing the Spectral Data window - Peak Purity tab. TRILUTION LC calculated a peak purity value higher than the Purity Threshold setting for the first and second peaks so those peaks are color filled green on the Peak Purity Display graph. The peak purity value was calculated lower than the Purity Threshold setting for the third peak so that peak is color filled red on the Peak Purity Display graph.

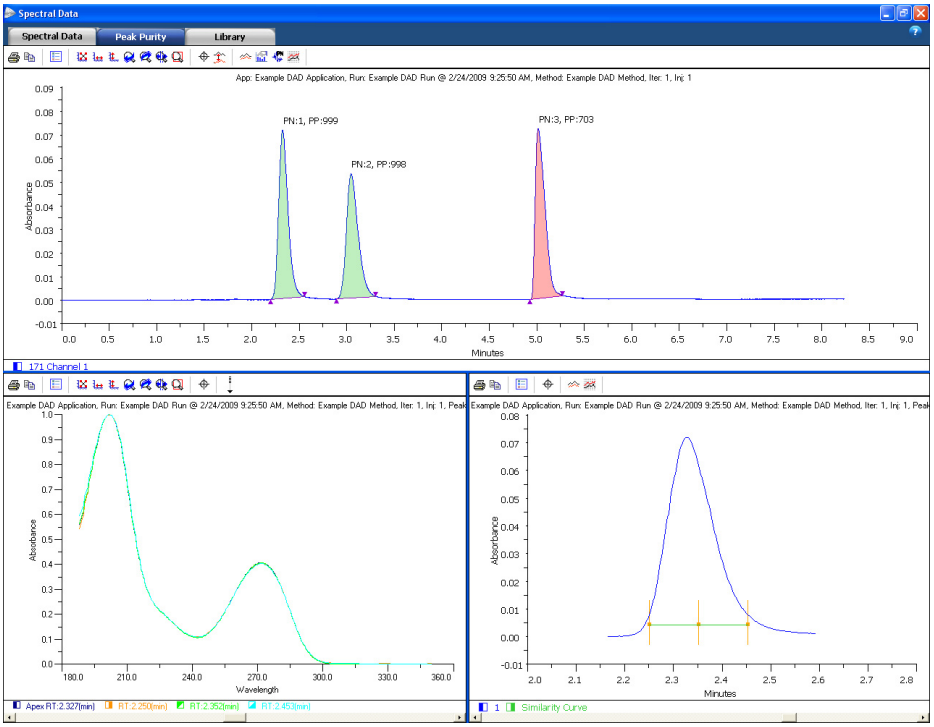


Figure A-12: Spectral Data Window - Peak Purity Tab

Modify Peak Purity Options Post-Run

The Peak Purity options can be modified during post-run re-analysis.

To modify the Peak Purity Options:

1 On the Run Results window, click **Edit**. The Result Analysis Builder appears.

2 On the Result Analysis Builder - Peak Integration tab, click **DAD Peak Purity**.

3 Modify the peak purity options and then click **OK**.

4 Click **OK** to return to the Run Results window.

5 In the Run Results window, click **Re-Analyze**. The changes are not saved until you click **Save** or click **Yes** when asked if you want to save data.

Exercise 3- Build and Use a Spectral Library

In TRILUTION LC, reference databases of spectral peaks of known compounds can be assembled. Search and match the database contents to the chromatogram for an unknown sample and report the outcome.

Perform spectral matching at run time or during post-run re-analysis. Each Analysis can contain unique library search parameters—searching one library, specific libraries, or all libraries.

Build a Spectral Library

1 Create a Spectral Library.

a) From the Run Results, click **Spectral Data** and then select the **Library** tab.

b) Click **New** to create a new library. The New Spectral Library dialog appears.

c) In the New Spectral Library dialog:

1) In the **Spectral Library Name** field, type a name.

2) In the **Short Description** field (optional), type a brief description of the library.

3) In the **Long Description** field (optional), type a detailed description of the library.

4) Click **OK**. The new library opens.

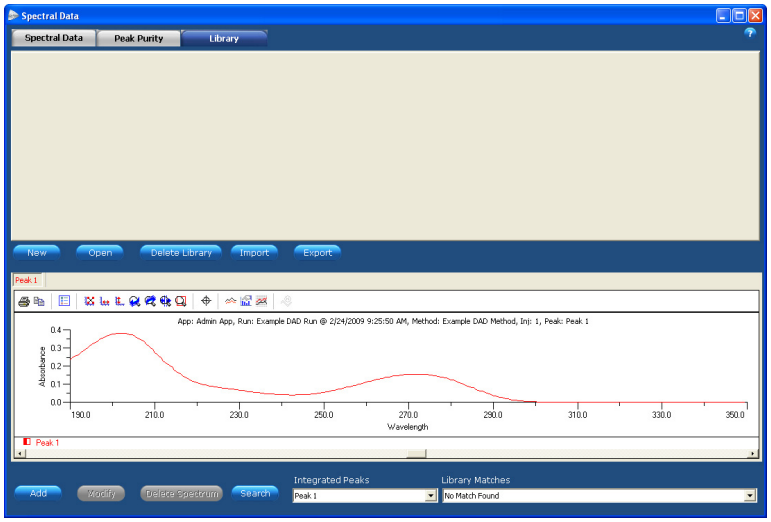


Figure A-13: Spectral Library

2 Add a peak spectrum into the Spectral Library for the peak.

a) Select Peak 1 from the drop-down list of Integrated Peaks.

b) Click **Add**. The Add Spectral Library Entry dialog appears.

3 In the Add Spectral Library Entry dialog:

a) In the **Peak Name** field, change the peak name, if desired. Peak names must be unique to ALL spectral libraries.

b) In the **Comment** field (optional), type a brief description of how data for the peak was acquired. For example, include mobile phase and column information.

c) In the **Comparison Wavelength Range** fields, specify the wavelengths TRILUTION LC will use when comparing the Spectral Library peak against an unknown peak. The default values identify the entire wavelength range collected during a run.

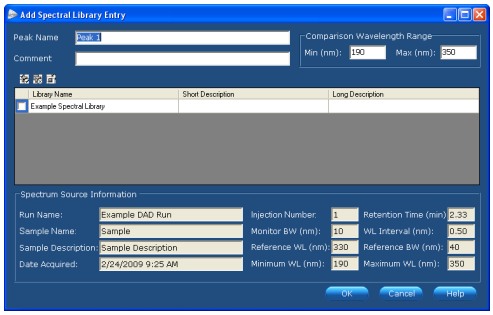


Figure A-14: Add Spectral Library Entry Dialog

d) Select the check box next to the **Library Name** for each library to which the peak should be added.

e) Click **OK** to exit the Add Spectral Library Entry dialog and save the peak spectrum.

4 View the spectrum for a peak by clicking the table row in the Spectral Library.

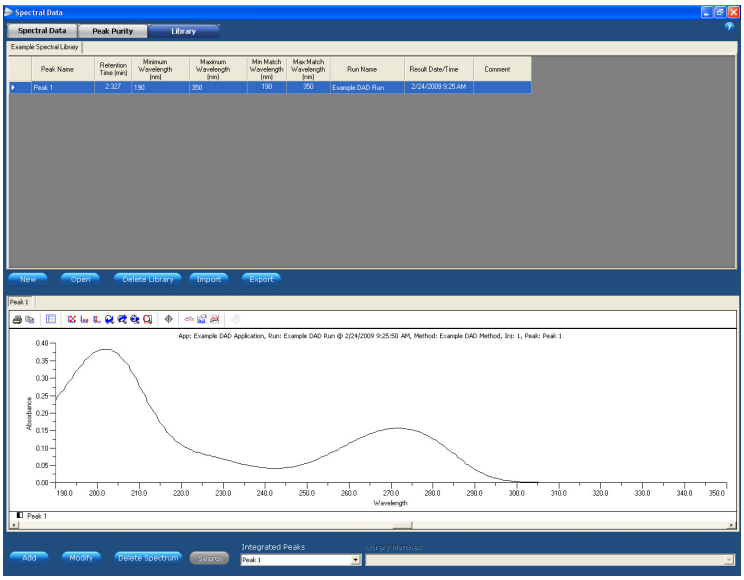


Figure A-15: Spectral Library

Search for Spectral Matches

In this part of Exercise 3, manually search for matches in the Spectral Library that was just created.

1 Open results containing integrated peaks.

2 Access the Spectral Data window - Library tab.

3 Select the name or number of one or more integrated peaks from the drop-down list of Integrated Peaks. Only selected peaks can be used when searching for spectral matches. As each peak is selected, a tab displays above the spectrum with the name in red.

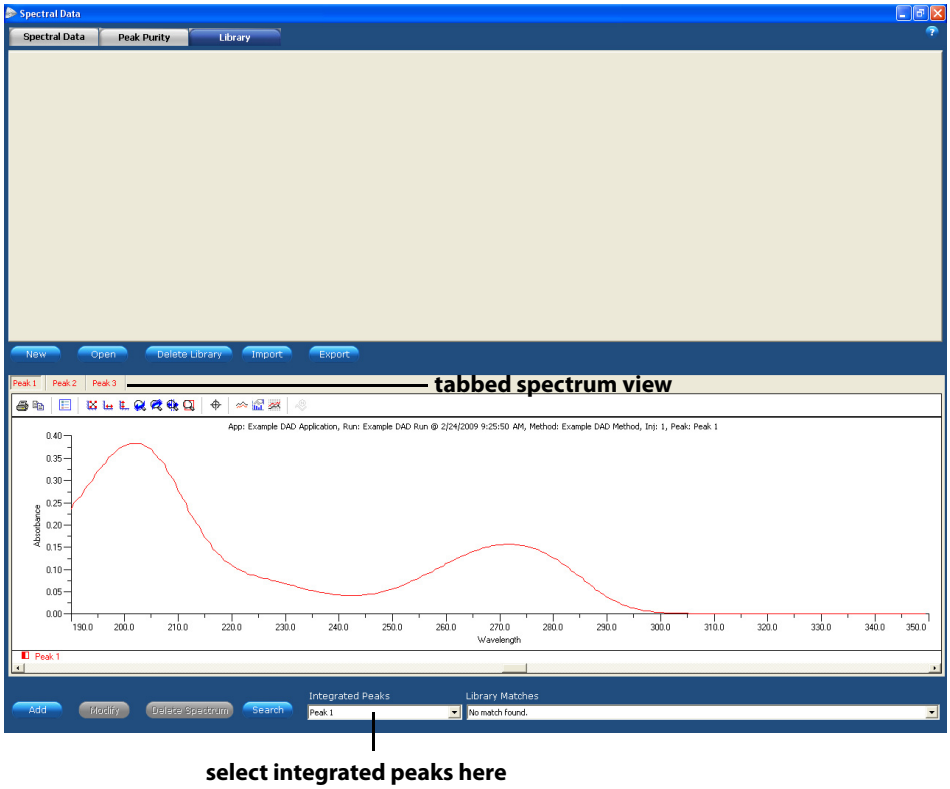


Figure A-16: Spectral Library

4 Click **Search**. The Search Spectral Library dialog appears.

5 In the Search Spectral Library dialog:

a) Select the check box next to the **Library Name** for each library to be searched.

b) For each of the first two peak spectra, select the check box next to the **Peak Name** and under **All Peaks** to search all Spectral Library peaks.

c) For the last peak spectra, select the check box next to **Time Period** and then type a **Start** time of 4.75 and an **End** time of 5.25 to search Spectral Library peaks with a retention time between 4.75 and 5.25 min.

d) In the **Match Threshold** field, change the Match Threshold to 500. A match value of 1000 indicates a perfect match.

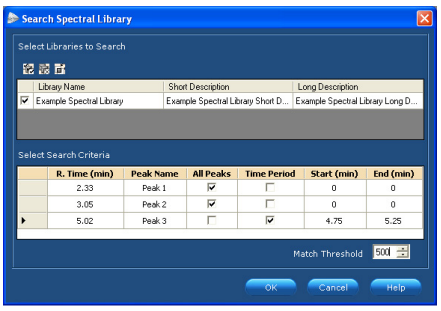


Figure A-17: Search Spectral Library Dialog

6 Click **OK** to begin the search.

7 When the search completes, TRILUTION LC displays the five closest matches.

8 In the tabbed spectrum view, select a spectrum. From the drop-down list of **Library Matches**, select a spectrum to overlay. The integrated peak spectrum displays red, the matched spectrum displays black.

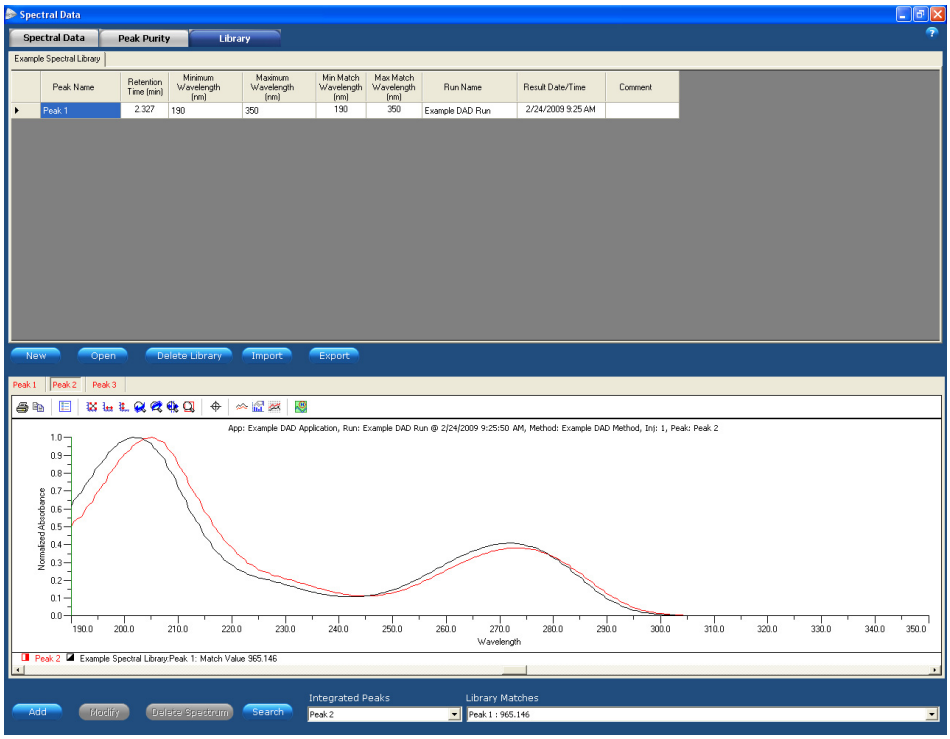


Figure A-18: Spectral Library

9 Remove the overlaid spectrum by right-clicking on the spectrum tab and then selecting **Close Tab** to close the spectrum tab.

Specify a Spectral Library in the Analysis

In this part of Exercise 3, set up the Analysis to perform spectral matching on unknown peaks automatically at run time.

1 Open the method and click to select the Analysis tab.

2 Click **DAD Spectral Library**. The Search DAD Spectral Library dialog appears.

3 In the Search DAD Spectral Library dialog:

a) Select the check box next to the **Library Name**.

b) Select the check box next to the **Peak Name** (UNKNOWNS) and under **All Peaks** to search all Spectral Library peaks.

c) In the **Match Threshold** field, change the Match Threshold to 500. A match value of 1000 indicates a perfect match.

d) Click **OK** to exit the Search Spectral Library dialog.

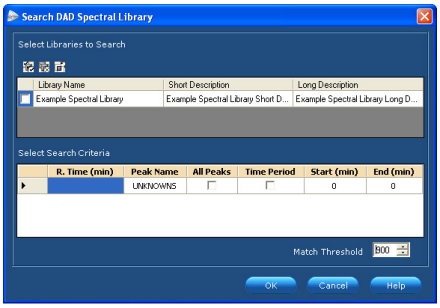


Figure A-19: Search Spectral Library Dialog

4 Add spectral match columns to the Analysis Report.

a) With the method still open, click to select the Report tab.

b) Under Report Type, click to select Sample.

c) Under Contents, click to select Spectral Match 1.

d) Click the right arrow ( ) to add it to the bottom of the Column Contents list.

e) Repeat steps c and d to add Spectral Match 2 and Spectral Match 3 to the list.

Note: The Sample Table in the Analysis Report can contain a maximum of ten columns. By default, eight columns are included. To add three spectral matching columns, one other column must be removed.

f) After the run is complete, view the Analysis Report (either from the Results or the Reports menu). Matches will be reported. If there was no match, the cell in the Sample Table will be empty.

Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (mAUmin x100)	Height (Absorbance)	Peak Purity	Spectral Match 1	Spectral Match 2	Spectral Match 3
1	1	2.327	813.1354	0.071	999.294	2.33(min), Example Spectral Library, Peak 1 [1000.000]	3.05(min), Example Spectral Library, Peak 2 [965.150]	5.02(min), Example Spectral Library, Peak 3 [795.828]
1	2	3.048	750.4139	0.052	998.29	3.05(min), Example Spectral Library, Peak 2 [1000.000]	2.33(min), Example Spectral Library, Peak 1 [965.146]	5.02(min), Example Spectral Library, Peak 3 [787.908]
1	3	5.02	847.3379	0.072	703.031	5.02(min), Example Spectral Library, Peak 3 [1000.000]	2.33(min), Example Spectral Library, Peak 1 [795.830]	3.05(min), Example Spectral Library, Peak 2 [787.909]

Figure A-20: Analysis Report - Sample Table

Replacement Parts B

Detectors

Part Number	*Description*
18100001	171 Diode Array Detector
18100002	172 Diode Array Detector

Lamp Assemblies

Part Number	*Description*
18001063	Deuterium lamp assembly for 171/172 Diode Array Detectors (UV wavelengths)
18001069	Tungsten lamp assembly for 171/172 Diode Array Detectors (VIS wavelengths)

Flow Cell Accessory Kits

Part Number	*Description*
Each of the flow cell accessory kits include a flow cell assembly with pre-connected inlet and outlet tubing, a plumbing package, and a 2.5 mm Allen wrench. The flow cell accessory kits for analytical applications also include a back pressure regulator.	
Analytical	
18004008	Flow cell accessory kit for analytical applications (with stainless steel inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
18004007	Flow cell accessory kit for analytical applications (with PEEK inlet tubing) flow cell: 5 mm pathlength, 12 µL volume, quartz
Preparative	
18004006	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.2 mm pathlength, 0.7 µL volume, quartz
18004005	Flow cell accessory kit for preparative applications (with stainless steel inlet tubing) flow cell: 0.05 mm pathlength, 0.16 µL volume, quartz

Flow Cells

Part Number	*Description*
Analytical	
100405	Analytical flow cell (5 mm pathlength, 12 µL volume, quartz)
Preparative	
100401	Preparative flow cell (0.2 mm pathlength, 0.7 µL volume, quartz)
100407	Preparative flow cell (0.05 mm pathlength, 0.16 µL volume, quartz)

Gaskets

Part Number	*Description*
18001020	Gasket, .062 cell holder (gray)
18001024	Gasket, .031 cell holder (black)

Inlet and Outlet Tubing

Part Number	*Description*
18007000	Plumbing package for 171/172 Diode Array Detectors
49041001	Upchurch F-100N Fingertight fitting, 1/16", 10–32, Kel-F (PCTFE), natural, for connection to column output
495018	PTFE adapter coupling with female luer (1/4"–28)
495019	Luer fitting, male (1/4"–28)
F1410050	PVDF 1/4"–28 coupling; package of 5.
490410255	Upchurch P-255 nut, super flangeless, 1/16"–1/32", 1/4–28, PEEK, natural
49041034	Upchurch P-250 ferrule, super flangeless, with SS ring, 1/16", PEEK, natural
Analytical	
18007001	Analytical outlet tubing, 0.3 mm (0.010") ID Teflon, with fittings
18007004	Analytical inlet tubing, 0.3 mm (0.010") ID stainless steel, with fittings to connect to flow cell
18007005	Analytical inlet tubing,0.3 mm (0.010") ID PEEK, with fittings to connect to flow cell
Preparative	
18007002	Preparative outlet tubing, 1.0 mm (0.040") ID Teflon, with fittings
18007006	Preparative inlet tubing, 1.0 mm (0.040") ID stainless steel, with fittings to connect to flow cell

Miscellaneous

Part Number	*Description*
36070905	Back pressure regulator (75 psi), PEEK
36070906	Back pressure regulator (75 psi), stainless steel
4320252	Allen wrench, 2.5 mm
7080318107	Power cord, 110V
7080316106	Power cord, 220V
6730164007	Fuse, 1.6 A, T-1.6
18004004	Remote flow cell holder
18001052	Fiber optic cable, 105 mm
18001059	Fiber optic cable for remote flow cell
638304512	Terminal block connector, 4 pin