SolventTrak

Liquid Chromatography
Mobile Phase Recycler

Operators Manual

Distributed by:

Carl Stuart Limited

ADVANCED APPLIED TECHNOLOGIES

Contact Us:
Irl Ph: 01 4523432
UK Ph: 08452 30 40 30
Web: www.carlstuart.com
Email: info@carlstuart.com

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1.0. Introduction

The **SolventTrak** Solvent Recycler for isocratic HPLC applications allows you to automatically and reliably recycle eluting mobile phase free of component peaks back into the solvent reservoir for reuse over multiple injections. Substantial cost savings can be achieved with the assurance that recycled solvent is clear of contamination.

All eluate fractions containing detectable peaks are "sensed" by **SolventTrak**'s internal integrator, in real time, and these fractions are diverted to waste by switching the position of a solenoid-controlled diverter valve. **SolventTrak** incorporates a high-performance analog-to-digital (A/D) converter operating with a 200 Hz sampling rate, which executes continuous first derivative calculations in real time to test the incoming signal waveform for slope changes corresponding to the starting or trailing edges of peaks. The slope changes found are compared to a value set by the operator on the front panel.

The diverter valve is activated when this value is exceeded as a peak begins, "cutting" the peak from the recycled solvent stream. When the slope again falls below the value as the peak ends, the diverter valve is switched back again to return clear solvent to the reservoir. A peak width slope threshold value is also set by the user via the control panel to change the effective signal bunching rate in order to provide a wider range of detection sensitivity.

An independent "delay" can also be preset to delay the diverter valve operation to compensate for the contained volume between the detector cell and the actual valve mounting position. This delay setting can also be used for fine adjustment of the "peak cut" cycle to account for slowly tailing peaks and overlap between individual fused peaks which are not baseline resolved. Both positive and negative peaks are handled.

SolventTrak can also be set to respond to each peak detection event by closing a contact, which can be connected to activate another device, fraction collector, or alarm, as well as activating visual and audible (defeatable) signals that a peak has appeared. The user may also activate a small "tick mark" on each chromatogram baseline at the exact time the beginning and end of each peak is detected, for a permanent record of peak processing on your integrator or data system -- or separately on an independent chart recorder.

SolventTrak includes an autozero function for the outgoing signal. Both peak detection and autozero can be activated remotely by a computer, data system, or

integrator for complete automation. The front panel LCD Display shows incoming signal level (autozeroed) in real time. An "override" function permits the diverter valve to be forced to the "waste" position regardless of peak detection status via manual or automated contact closure switch.

Because **SolventTrak** does not depend on simple "threshold" based peak detection, it can be extremely accurate in finding peaks reliably in situations where the detection method creates routinely rising or falling baseline, or when very small peaks are found in the same chromatogram along with very large ones. Moreover, the main determinant influencing accurate peak detection in **SolventTrak** is the shape of peaks, which is often consistent over a wide range of applications, methods, and samples. **SolventTrak** therefore will generally not require the frequent resetting needed for threshold-based detection.

2.0. Specifications

Peak Detection

Signal Input Range: -1.000V to +1.000V

Sampling Rate: 200 Hz, continuous during operation

Number of Inputs Supported: One

Peak Width Range: User-settable to 1, 5, 15, 30, 45, 60, 75, 90, 105, 120, 150, 180, 210, 240, 270, or 300 seconds

Peak Slope Ranges: User-settable, 15 to 14,400

Diverter Valve Delay Timer: User-settable, 0 to 99 seconds, 1 second increments

Peak Waste/Valve Open Marker: Defeatable, tick duration 0.1 second, appears in Signal Out

Contact Closure Outputs: Peak Waste/Valve Open, active while valve switched to waste

Contact Closure Inputs: Enable/Disable Autozero, 1 second duration
Enable PeakWaste/Valve Override (continuous)
Start (Start CleanUp Cycle)

Peak-to-Waste/Valve Open Indicators: LCD Display Status , audio beep (defeatable)

Display: LCD, Active input voltage display, Peak Detection & Valve Position Status

Autozero: Manual Autozero Key or contact closure enable (1 second duration)

Diverter Valve

Type: Solenoid-controlled, 3-way, high efficiency, zero dead volume

Wetted Surfaces: All internal Teflon/PEEK or Teflon body

Mounting Ports: 1/4 - 28 Flat Bottom (valve can be mounted in any orientation)

Operating Pressure: Up to 30 psi (2 bar)

Response Time: Approx. 5 milliseconds

Requirements

Power: External Adapter (included) 110V/60 Hz or 220V/50 Hz, self switching, 12VDC Output

Environment: 40 to 90 F (4.4 to 32.2 C), 10% to 60% relative humidity (non-condensing)

Physical

Size: 9"W x 10"D x 3"H (22.8 cm x 25.4cm x 7.6 cm)

Weight: 5 lbs (2.28 kg)

3.0. Installation

Unpacking SolventTrak Unit

Remove the SolventTrak unit from its shipping container and check to be sure all components and accessories are present.

Items shipped with SolventTrak:

(1) SolventTrak instrument
(2) Input/Output signal cables
(1) External Power Adapter 12VDC Output
(1) Power cord, grounded
(1) RS232 Serial Cable for connection to PC Computer
(1) Diverter Valve Assembly
(3) 1/4" - 28 flangeless fittings (for connections to diverter valve ports)
(3) 1/4" - 28 ferrules (for diverter valve connections)
(1) 10' length, 1/16" Teflon tubing (0.030" ID) for valve connections
(1) Operators Manual

If any items are missing, call Axxiom Chromatography or your local Axxiom dealer or distributor.

Connecting Diverter Valve to Detector Cell

SolventTrak is intended to be positioned adjacent to or above your HPLC pump or detector [Figure 3.1] so that column eluant exiting the detector flow cell can be plumbed directly into the SolventTrak diverter valve. The SolventTrak unit is self-contained and requires 12VDC power from the adapter included with the instrument.

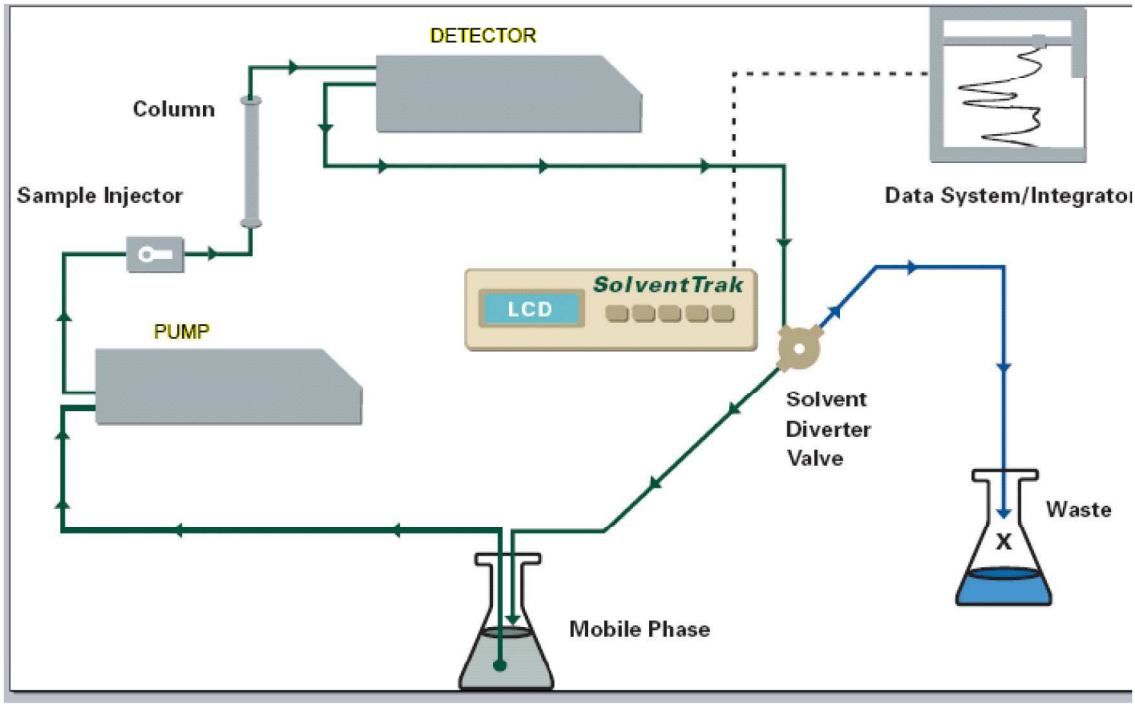


Figure 3.1 Placement of SolventTrak Unit in HPLC System

Normally, to minimize back pressure into and out of the diverter valve, it is advisable to use the shortest possible tubing lengths between the flow cell detector outflow connection and the valve input fitting, and from both the "waste" and "recycle" valve outlet fittings to the waste container and solvent reservoir, respectively.

Using the standard signal cables supplied, the distance between SolventTrak and the detector cannot exceed six feet. A considerably shorter tubing connection between the two is recommended. Tubing of any desired material can be fitted to and from the diverter valve if it is compatible with the 1/4 - 28 ferrules needed to mate with the valve.

All internal valve surfaces are Teflon for maximum inertness. The standard diverter valve will handle up to a maximum of 30 psi. If higher pressure or capacity is required, please contact Axxiom or your local dealer for assistance.

For normal operation, the valve can be mounted on either side of the SolventTrak chassis by using the bracket supplied. Alternatively, it can be mounted onto the detector chassis near to the flow cell....or it can be connected directly to the detector flow cell output tubing via direct fitting/ferrule connection.

To begin installation, position the SolventTrak unit as desired and plumb the valve using Teflon, or other appropriate tubing and fittings. For most applications you should use the Teflon tubing and fittings supplied with the unit for both the valve-to-reservoir and valve-to-waste lines exiting the valve, and for the flow-cell to valve line if the tubing can be mated with the detector cell outlet fitting.

Be certain to connect to the correct valve ports so that the input, waste and recycle lines are properly identified and directed [Figure 3.2]. After fittings and tubing are installed, you can check for correct plumbing before operating the system by forcing some solvent through the valve with SolventTrak power OFF (recycle/through valve position). Observe that the solvent moves through the valve and into the line to be routed to the solvent reservoir. If this is not the case, the tubing lines will need to be reversed for correct operation. Be sure that the waste and recycle tubing lines from the valve are completely and firmly anchored in their appropriate reservoirs or bottles.

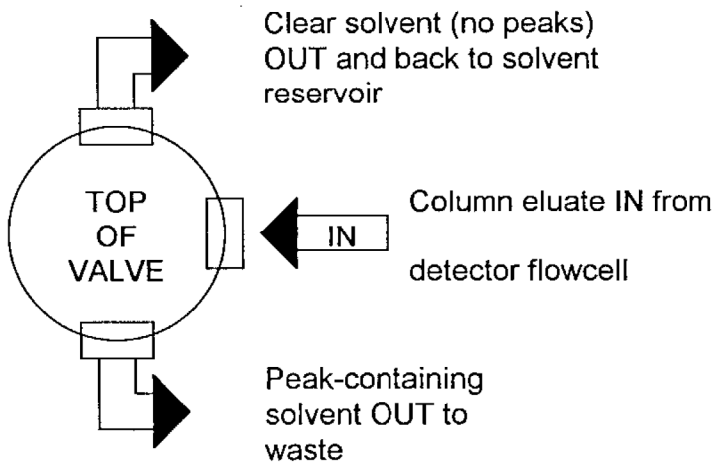


Figure 3.2 Flow Orientation for Diverter Valve Ports

Making System Connections

Figure 3.3 shows the rear panel of the SolventTrak unit where all connections are made for 12VDC power, input and output of signal, diverter valve, and input or output contact closures. Make these connections only after the unit has been placed in its operating position and all plumbing has been finished.

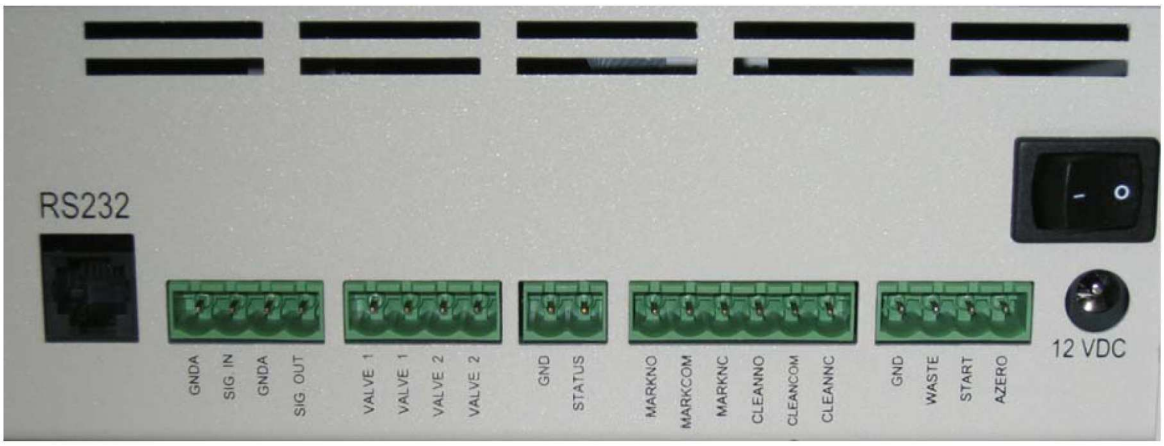


Figure 3.3 SolventTrak Rear Panel

Looking at the rear panel, the power supply connection and power On/Off switch are located on the right side. The detector signal input and output connections, contact closure connections, diverter and Cleanup valve connections are made on the terminals of the rear panel.

To begin the electrical installation, take one of the Input/Output cables provided and connect one end to the "Signal In" terminals on the rear panel and the other end to the output of the detector that will be used with SolventTrak. This detector must produce an analog (not digitized) signal in the range -1.000 to +1.000 Volts. Signals exceeding this range will be "clipped" and will not be properly evaluated by SolventTrak during its operation.

If necessary, a connector may be wired onto the free end of the cable for appropriate mating with the analog output terminals of the detector. [Contact Axxiom if special cabling and connector is needed for your detector that you are not able to find or make.] Otherwise, simply fix the leads of the cable to the detector's output screw terminals. The black wire should be considered ground; it may be necessary to jumper this "negative" side to the detector chassis or shield cable to eliminate noise. The cable shield wire can be connected to ground on the detector chassis or housing. Note that the total length of this cable should not exceed 15 feet (approximately 5 meters) in order to minimize noise and signal degradation. [Figure 3.4 shows typical wiring of a SolventTrak installation.]

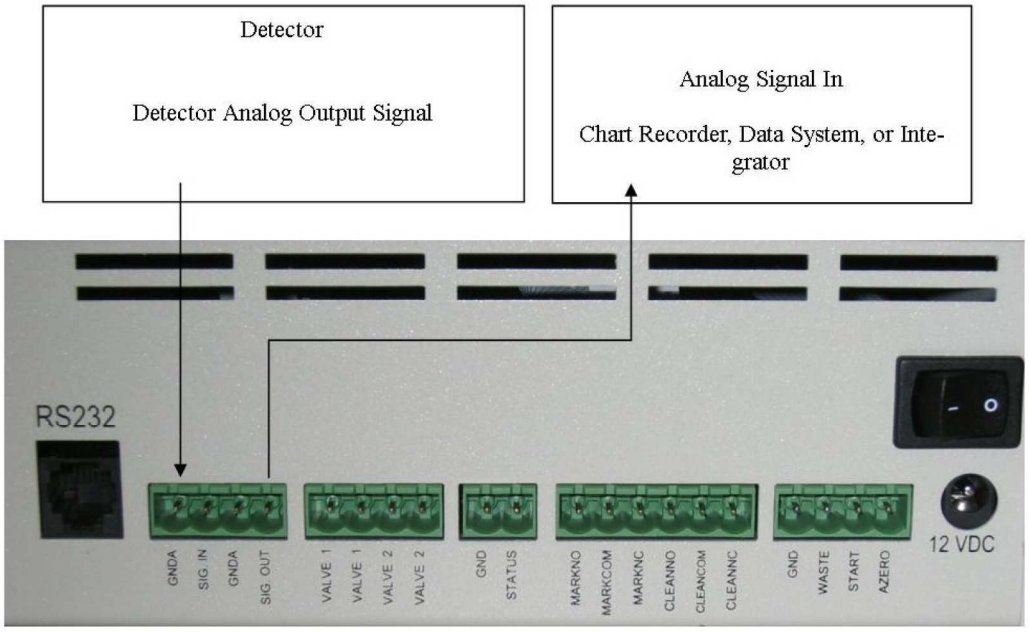


Figure 3.4. SolventTrak Signal In/Out Connections

If the detector output signal is to be transferred to a chart recorder, integrator, data system, or other computer, install the second cable between SolventTrak and the recording or integrating instrument. Connect one end of the signal cable to the rear panel terminals marked "Signal Out", and connect the other ends of the cable to the appropriate inputs on the recorder, integrator, or data system (after attaching the required connector, if any). The black wire is common and the shield wire can be connected to grounded housing of the recording device.

Assignments of the connectors on the rear panel, from left to right, are as follows:

RS232	Output Connector	This is an RJ11 connector plug. Used for connection of a special RS232 cable. This RS232 output is for making a connection to a computer serial port in order to obtain data from the SolventTrak for GLP logging.

Position	Label on Terminal	Function
1	GNDA	Negative detector input signal (+/- 1 volt range)
2	SIG INPUT+	Positive detector input signal (+/- 1 volt range)
3	GNDA	Negative output signal (+/- 1 volt range)
4	SIG OUTPUT +	Positive output signal (+/- 1 volt range)
5	VALVE 1	Recycle Valve Power connection
6	VALVE 1	Recycle Valve Return
7	VALVE 2	Cleanup Valve Power connection
8	VALVE 2	Cleanup Valve Return connection
9	STATUS	Analog output signal that shows when valve is active
10	MARK NO	Peak sense contact closure, Normally Closed contact
11	MARK COM	Peak sense contact closure, Common contact
12	MARK NC	Peak sense contact closure, Normally Open contact
13	CLEAN NO	Cleanup contact closure, Normally Closed contact
14	CLEAN COM	Cleanup contact closure, Common contact
15	CLEAN NC	Cleanup contact closure, Normally Open contact
16	GND	Ground input for zero & Start inputs
17	WASTE	TTL or Contact closure input to force valve to waste
18	START	Input TTL or Contact closure input to trigger Cleanup method
19	AZERO	TTL or Contact closure input to trigger Autozero

If necessary, connect the two leads from the recycle diverter valve assembly to two terminals marked "VALVE1". Either lead can be connected to either terminal; there is no polarity requirement.

THESE LEADS MUST BE CONNECTED PROPERLY FOR CORRECT SOLVENTTRAK OPERATION!

If you will want to "override" the normal SolventTrak peak detection/valve operation to force the valve to the "waste" position via an external contact closure or push button switch, connect the appropriate leads to your switch, integrator, data system, or other controller device, then to the terminals marked WASTE and GND [Figure 3.5 on the following page].

As an example, the OVERRIDE capability can be used in conjunction with a data system that is able to execute timed contact closure events during the course of a chromatographic method to "block out" and divert to waste a group of unretained peaks which elute quickly at the start of each run. This would be accomplished by connecting the WASTE and GND leads to a contact closure relay on the data system that would close at the start of each method and open again as soon as the peaks to be discarded have cleared. Connecting these leads to a manually actuated push button switch near the HPLC unit is also a convenient way to add a manual "bypass" function for quick diversion of any material passing through the detector cell to waste.

If the autozero function is to be switched by an external device, right most terminal (AZERO) and GND should be wired to the outputs from that device [Figure 3.5]. This autozero switching can be done by a data system, integrator, or remote push button. The logic must be set so that autozeroing occurs when the switching relay is closed. Note that the front panel "ZERO" key on SolventTrak performs the same function manually at any time.

Use the MARK connections for output of the peak sense contact closure if, for example, you wish to drive a fraction collector to repeatedly collect column eluate passing through the "waste" line. If for the functionality required the relay is to be closed when a peak is sensed (normally open), the leads for this relay are connected to terminals MARKCOM (common) and MARKNO. If the relay is to be open when a peak is sensed (normally closed), the leads for this relay are connected to terminals MARKNC and MARKCOM (common). These connections can be made to the contact closure input terminals for an alarm, valve, fraction collector, or other device to be activated in response to the presence of a peak. **Connection of any of these terminals is <u>not</u> required for standard SolventTrak operation.**

It is possible to enable or disable the overall diverter valve operation of SolventTrak using an externally programmed device such as a data system, integrator, or remote switch, by wiring an appropriate contact closure in series with the VALVE solenoid power connections made to either VALVE terminals. Opening such a contact will prevent the valve from actuating when peaks are detected and effectively disable SolventTrak's function; however, peak detection itself will not be disabled under such circumstances (both the visual and audible peak divert indicators will continue to respond in the usual fashion when peaks are found). You must use a contact closure or switch which will handle the current load (about 200 mA) used by the diverter valve.

All relay closure inputs have a requirement or duration of 1 second. If an external switching input does not equal or exceed one second, a simple RC network can be used to effectively extend the closure duration time. Contact Axxiom Chromatography for assistance in this event. Likewise, if a 1 second closure output from SolventTrak is insufficient, an RC network can be used to lengthen it.

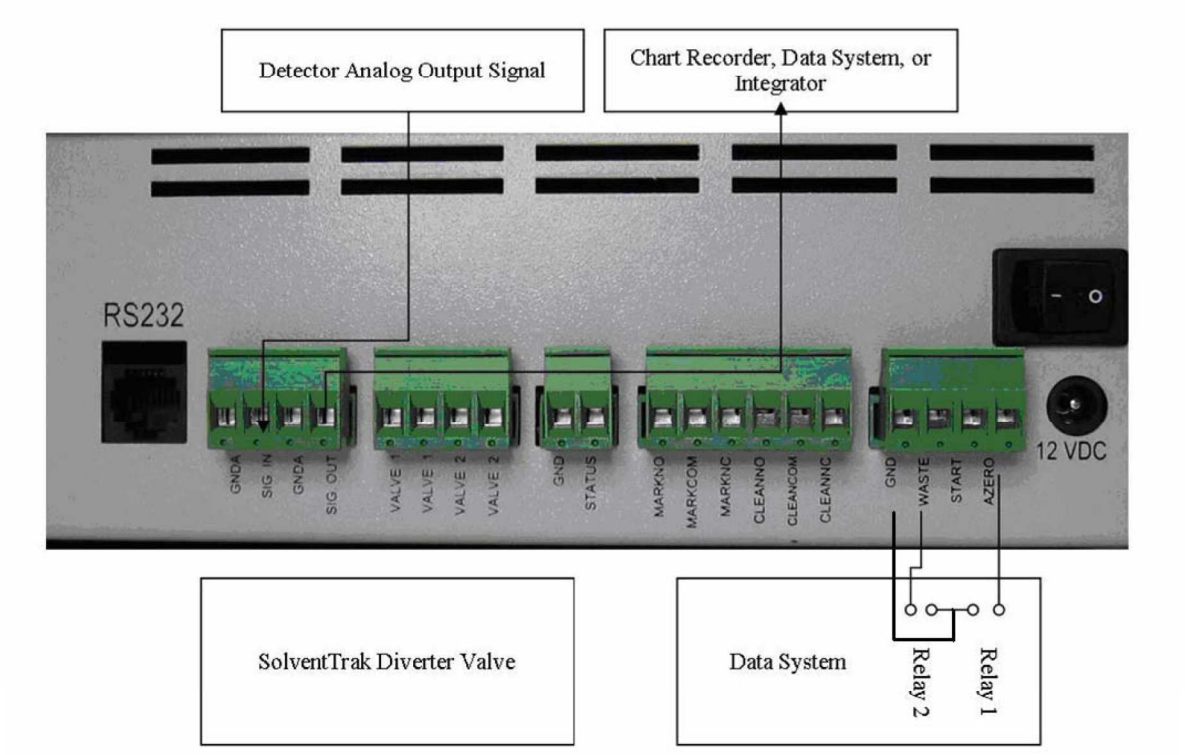


Figure 3.5 Remote Divert-to-Waste Override and Autozero By Data System

Cable lengths for contact closure connections can be as great as required to link all appropriate components.

When all wiring has been completed, connect the line power cord between its socket on the DC power supply module and a grounded wall outlet. Connect the 12VDC output to the 12VDC jack on the rear of the SolventTrak. SolventTrak is self switching for either 110V/60Hz or 220V/50Hz line power; no re-fusing or other adjustments are needed for operation at 220V/50Hz. Toggle the rear panel power switch to ON. The LCD Display will display the current detector output signal level in volts if the Signal In cable has been connected properly and the detector is powered. An automatic autozero of the outgoing display will be performed each time SolventTrak is powered up.

4.0. Operation

SolventTrak is designed for simple operation with only a few user settings and adjustments. Programming is via the keypad located on the front panel. Normally, once set, the instrument will properly handle a wide variety of applications having differing peak scaling and baseline profiles. New settings may be needed when the average or minimum peak width to be expected varies dramatically.

Front Panel

The front panel provides information on the status of the incoming signal and a keypad for setting detection slope thresholds, response delay, and autozero [Figure 4.1].

Figure 4.1. Front Panel Controls and Displays

LCD Input Signal Display

The LCD Signal Display displays the incoming signal level from the detector in use at the Signal Input jack. Under normal conditions the incoming and outgoing signals will be the same. If autozero is used the outgoing signal level at the time of autozeroing will be reset to 0.000 V and the incoming and outgoing signals will then differ by the amount of the autozero compensation. The signal display will show signal levels (autozero'd) between -1.000 and 1.000V.

Diverter Valve Status Field

The Valve Status field will indicate "WASTE" when a peak is sensed and the diverter valve moves to the waste position. It will indicate "Waste" when the remote over-ride input is active. It will display "WASTE" until the end of the waste cycle associated with that peak. It will indicate "RECYC" when a peak is not detected. It serves as a visual indicator of the diverter valve operation at all times in response to the presence of peaks.

NOTE THAT BOTH THE VALVE STATUS FIELD AND THE AUDIBLE INDICATOR ARE LINKED DIRECTLY TO VALVE OPERATION AND WILL REFLECT THE CURRENT "DELAY" SETTING AFTER EACH ACTUAL PEAK DETECTION EVENT AS WELL AS THE BUILT-IN TAILING SLOPE DELAY (SEE BELOW).

Programming Via the Keypad

Each setting may be accessed by first pressing the ENTER key once, and then pressing the Down Key until the parameter is displayed that you desire to change. Next press the enter key one more time. You are now in the EDIT Mode for that setting. Use the UP/Down keys to increase or decrease the setting. When the desired setting is displayed, simply press ENTER one final time to exit the EDIT mode and return to the RUNNING mode.

NOTE: As each setting is displayed, the current value of that setting will be shown.

Slope Parameter

The SLOPE parameter sets the limit threshold for baseline slope corresponding to the leading and trailing edges of peaks in the incoming detector signal. This parameter is set between 15 and 14,400, 15 being the lowest possible slope threshold value. [See the Adjusting Peak Detection Settings section below for a more complete explanation of how this setting should be determined.] The Slope setting can be modified by the user at any time during PeakTrak operation. [*See the Threshold Setting for information about Absolute Threshold override mode.*]

Width Parameter

The WIDTH parameter adjusts the peak width threshold for peak detection. The WIDTH parameter can be set from 1 through 300 seconds, 1 second being the narrowest possible peak width. [See the Adjusting Peak Detection settings section below for a more complete explanation of how this setting should be determined.] The WIDTH value can be modified by the user at any time during operation.

Delay Setting

The DELAY setting adjusts the delay in switching the diverter valve (and executing an associated remote contact closure), as well as the audible beep responses, to allow sufficient time for eluting

peak-containing fractions to reach the position of the diverter valve after they pass through the detector. This delay will normally take into account the length of tubing between the detector cell and the diverter valve, the diameter of the tubing, and the mobile phase flow rate through the column. This parameter can be set at values between 00 and 99, corresponding to an actual delay time of 0 to 99 seconds in one second increments. A delay setting of 0 will result in immediate contact closure response at the time of the actual peak detection. [See the Adjusting Delay Setting section below for a complete explanation of how this setting can be optimized.] The DELAY value can be changed at any time during PeakTrak operation. It has absolutely no effect on the sensitivity or response of peak detection, but of course is important in determining if peaks are completely diverted to waste by the valve.

Threshold Setting

When the Threshold setting is zero (recommended) this feature is inoperative. When the Threshold setting is any number other than zero the absolute baseline level will be monitored as well as the slope. Whenever the baseline exceeds the threshold setting the valve will be switched to waste until the baseline falls below the Threshold setting. When the baseline is below the Threshold setting normal "slope" peak detection is resumed automatically. *This additional peak sensing feature can be used in case a flat topped peak that exceeds the peak width setting is encountered unexpectedly.* The range of the Threshold setting is from 0 to 1000000 microvolts.

Zero Keybutton

The ZERO key allows manual autozeroing of the outgoing signal from SolventTrak. Pressing the key will cause the outgoing and displayed signal to have a value of 0.000V. The ZERO key can be used at any time during SolventTrak operation. Automatic autozero can be executed using the ZERO IN relay closure input at any time. The relay input will support use of a remote pushbutton for activating autozero from a distant location.

NOTE: ALTHOUGH USE OF THE MANUAL OR REMOTE AUTOZERO FUNCTION WILL NOT ADVERSELY AFFECT SOLVENTTRAK'S ABILITY TO DETECT PEAKS, IT IS STRONGLY RECOMMENDED THAT AUTOZERO BE USED ONLY WHEN NO PEAKS ARE ELUTING FROM THE COLUMN.

Alarm Mute Setting

The Alarm Mute setting toggles the audio buzzer ON or MUTE (OFF). If set to ON, the buzzer will generate an audible signal whenever peaks are detected. The audible buzzer will remain active for as long as the valve remains in the waste position. Setting the toggle to MUTE defeats the buzzer.

Tick Setting

The TICK Setting enables Tick Marking. It also determines the height of the Tick Mark itself. The range is from 0 to 6,500 units of height. If set to a value greater that 0, each time a peak beginning or end is detected, a "tick" will be inserted into the outgoing analog signal at that exact time to mark where the peak has been "cut" by SolventTrak.

When a tick occurs, the signal will be increased (to a value determined by the Tick setting value) above the last autozero baseline level for a duration of 0.2 seconds, and will then be restored to its current value. This will generate a "positive" tick on baseline, the magnitude of which will depend on the current TICK setting. Such ticks should not normally interfere with subsequent integration of the outgoing signal by an integrator or data system. If necessary, resetting your integrator's algorithm to

exclude narrow "peaks" at or below 0.2 sec will prevent the ticks from being treated as peaks. Setting the value to 0 (zero) defeats all tick marking of the signal baseline.

Valve Status Output

The Valve Status Output indicates when the Recycle Valve is in the Waste Position. The signal level is at zero when in Recycle position and positive when "cutting" a peak to waste.

Adjusting Peak Detection Settings

Determining Optimum Slope and Width Values

The peak slope and width settings programmed via SolventTrak's front panel determine the sensitivity and response of its peak detection algorithm and the subsequent performance in eliminating peaks from the mobile phase solvent to be recycled. Both values must be adjusted based on the size, sharpness, and width of peaks typical for the type of analyses being performed and the detector being used.

By definition, SolventTrak's SLOPE setting is equivalent to a baseline signal slope, or rate of change, which defines the start of a peak when exceeded (and the corresponding peak end when the rate of change drops below the threshold slope). As it collects and monitors incoming detector signal information, which consists of time-averaged "bunches" of 200 Hz signal data points according to the WIDTH parameter, SolventTrak determines that a peak has appeared if it finds one interval between two "bunches" which exceeds the signal slope limit followed by a second consecutive interval whose derivative (rate of change) equals or exceeds the first interval's.

It is assumed that 40 bunched data points are required across the full peak width at baseline for correct peak resolution.

Increasing the WIDTH averages more points for a larger effective slope measurement interval, reducing the effective "time slicing" performed during the search for peaks and causing smaller peaks to remain undetected.

Since the SLOPE and WIDTH values are interactive, the actual range of threshold slope limits runs from 122 microvolts per second (SLOPE = 15, WIDTH = 1) to 12.2 millivolts per second (SLOPE = 14,400, WIDTH = 1) for the fastest bunching and from 0.41 microvolts per second (SLOPE = 15, WIDTH = 300) to 40.8 microvolts per second (SLOPE = 14,400, WIDTH = 300) at the slowest bunching.

In order to handle peaks with extended tailing edges and the common situation of peaks with tailing shoulders, SolventTrak has two other special operational features:

First, each time an "end of peak" event is found (two consecutive derivative intervals at the current bunching rate drop below the set slope threshold), AN AUTOMATIC DELAY OF ONE-HALF OF THE CURRENT PEAK WIDTH SETTING IS APPLIED BEFORE THE DIVERTER VALVE IS RESTORED TO ITS "RECYCLE" POSITION. Increasing or decreasing the peak WIDTH parameter will change this delay in a corresponding manner, which can be observed via both the Peak Tick Marker function and the visual/audio peak divert indicators. If the built-in delay overlaps the start of a second peak, SolventTrak will automatically keep the valve in its "waste" position continuously across both peaks even though a new peak detection event occurred, to avoid returning even a small portion of the tailing edge of the first peak to the solvent reservoir.

Second, to prevent possible "synchronization" errors and to handle shouldered peaks in which the effective slope can drop below the threshold at the start of a tailing shoulder rather than at the true

end of the peak, SolventTrak constantly tests each segment of baseline to determine if the average rate of change OVER A TIME CORRESPONDING TO ONE-HALF OF THE CURRENT PEAK WIDTH SETTING remains below the current slope threshold. If this is true, SolventTrak determines that true baseline has again been found and resets its peak-finding logic accordingly. This "safety factor" can, in cases of very wide and flat peak crowns or wide shoulder peaks, sometimes cause SolventTrak to assume true baseline when a peak is actually present, if the WIDTH value is not set sufficiently high to allow for the maximum duration of a "flat" crown or shoulder.

NOTE: See the Threshold setting for details on how to override flat peaks and ensure that they are cut to waste.

Figure 4.2 shows which portions of eluate in a typical chromatogram would be cycled to waste versus back to the solvent reservoir, with SLOPE at 45 and WIDTH at 60 sec.

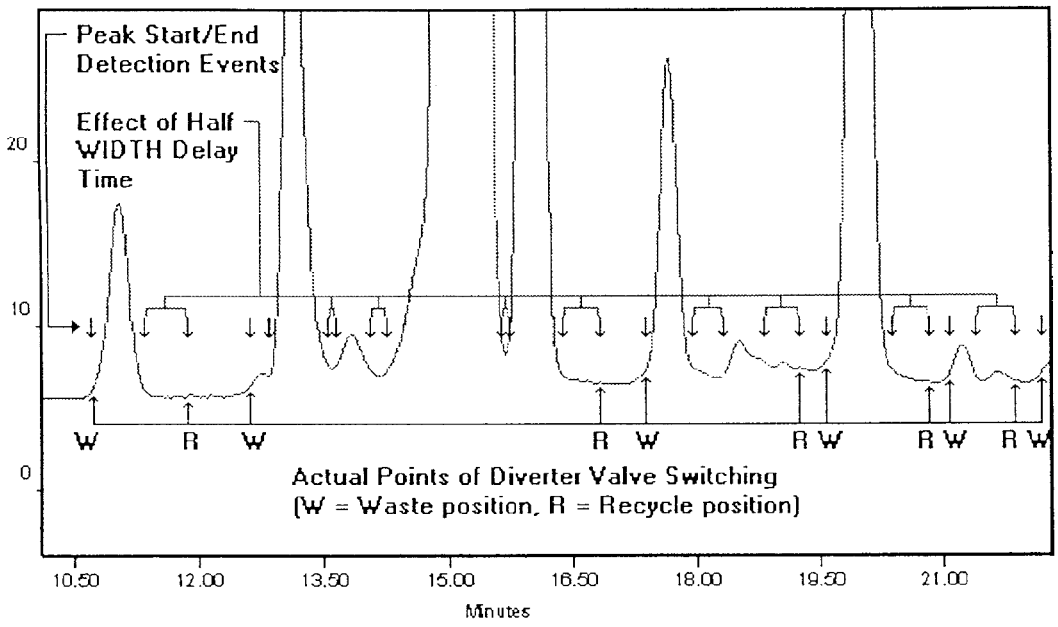


Figure 4.2 Example of Peak Detect/Divert Operation Showing Tick Marks

The basic rule for setting the SLOPE value is the higher the value, the later/higher level above baseline peak leading and trailing edge detection will occur, meaning less of the actual peak will be diverted to waste.

To guarantee maximum purity of recycled mobile phase, it is recommended that detection logic for a new application be set by first estimating the width at baseline of the WIDEST expected peak, and then setting the WIDTH at approximately that time value. Then set the SLOPE setting to 420. Run a typical sample with SolventTrak feeding its output signal to a chart recorder, integrator, or data system and with the Tick Marker enabled to visually indicate where peaks are cut, and observe whether the leading and trailing edges of peaks are being found properly and consistently.

Since it is usually more desirable to divert a little more solvent to waste in the interest of enhancing complete peak elimination, using the WIDTH associated with the widest expected peak will not only extend the built-in tailing edge delay but will also reduce the likelihood of flat-crowned peaks being recognized as baseline.

If noise is being detected as peaks due to the slope threshold being too low, raise the SLOPE value until only real peaks are found. If peaks are being cut too late, reduce SLOPE accordingly. It may be necessary to do several sample injections with typical peaks in order to optimize these settings.

Negative peaks will be detected and diverted to waste in exactly the same manner as positive peaks, using the same threshhold and peak width settings.

Adjusting the Delay Setting

The DELAY setting used in conjunction with the SLOPE AND WIDTH parameters can be a very effective means of "fine-tuning" the diversion of each peak-containing fraction. Basically, the DELAY setting should be estimated by computing the time required for the solvent front to move from the detector flow cell (where detection occurs) through the connecting tubing and into the diverter valve. This time depends on the size of the tubing used, the length of the tubing, and the flow rate of solvent from the column.

As an example of this calculation, if 0.020" inside diameter tubing connects the flow cell with the SolventTrak diverter valve over a length of 25 cm, and the HPLC pump flow rate is set at 1.0 ml/min for the column, the following analysis is made:

Contained volume in tubing = 2.03 ul/cm (from table below) x 25 cm = 50.75 ul

Flow rate = 1 ml/min = 16.7 ul/sec

Time required to clear contained volume = 50.75 ul/16.7 ul/sec = 3 sec

DELAY Setting Recommended = 3 (3 seconds delay)

Of course, more tubing or tubing with a larger inside diameter can be added to increase the effective time delay between detector and diverter valve. A shorter length of tubing (or tubing with a narrower inside diameter) can be used to reduce it.

You can easily use the DELAY factor to "shift" the peak cut window to account for inter-peak leading edge shape deviations by shortening the DELAY setting from its optimal value based on the contained volume calculation, moving the peak diversion point farther forward in time for each peak [Figure 4.3].

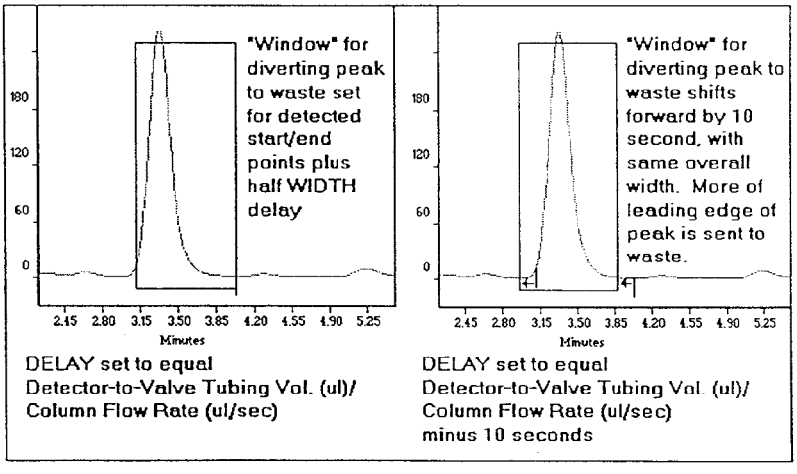


Figure 4.3 Using DELAY to Time-Shift Peak Diversion Windows

Note that a DELAY other than zero will cause the audible buzzer sound to be delayed, as well as a delay of the MARK relay closure. However, the Peak Tick Mark and "PEAK" LCD Display field will operate in real time regardless of the delay setting to tick-mark peaks as they appear at the detector, so a correct hard-copy record can be maintained. If SolventTrak's closure response is defeated by an external series relay during a delay cycle there will be no response to the delayed peak, except for an event mark on the detector signal output, if this feature is enabled.

One means of verifying that peak-containing eluate fractions are being consistently diverted to waste is to run the installed system with a second detector cell connected at the diverter valve "recycle" output port. If all peaks have been detected and diverted to waste, no indication of any residual contaminants (i.e. peaks) should appear in the output of the second detector.

The following table shows contained volumes in tubing of various inside diameters:

Tubing ID, In.	Tubing ID, mm	Volume, ul/in.	Volume, ul/cm
0.005	0.127	0.323ul/in.	0.127ul/cm
0.007	0.178	0.632ul/in.	0.249ul/cm
0.010	0.254	1.288ul/in.	0.507ul/cm
0.020	0.508	5.146ul/in.	2.026ul/cm
0.030	0.762	11.577ul/in.	4.558ul/cm
0.040	1.016	20.581ul/in.	8.103ul/cm
0.050	1.270	32.160ul/in.	12.660ul/cm

CleanUp (Shut Down) Setting

When the CleanUp option is ordered, a second valve is provided for switching over to a clean up or wash solvent at the end of operation. In addition, an external device can signal SolventTrak to start the clean up operation via the "Start" remote input. When the cycle is complete, SolventTrak will signal completion via the CleanUp relay contact closure.

The Cleanup mode is enabled by setting the Cleanup duration setting to a value above zero. The Cleanup time setting may be set in one minute increments from 0 to 990 minutes.

When the Cleanup mode is started via a remote signal. the LCD Display will indicate the Cleanup mode is active and normal peak detection will cease. **During clean up the Recycle valve will be held in the Waste position.** The time to completion will be displayed as it progresses.

Figure 4.4 Display showing that Cleanup Mode is running

5.0. Service and Maintenance

SolventTrak requires very little maintenance and should provide years of reliable operation.

Maintenance of Valve

The diverter valve is rated at 10 million cycles for a long service life. It can, however, become plugged or damaged if particulates are introduced. Replacement valves are available from Axxiom as part number 100-120.

To test a valve and verify complete cycling, disconnect the valve assembly from the detector and immerse the ends of both the waste and back-to-reservoir lines in compatible fluids. Fill a large syringe with air, connect it securely to the input tubing entering the valve, and force air through the valve with SolventTrak set in the "normal" ("recycle") position - air bubbles should appear only from the tubing line returning solvent to the reservoir. Use the manual override key to cycle the valve to the "waste" position and force air again through the input line. The air bubbles should now appear only at the tubing going into the waste container. If any air is seen escaping from the opposite tubing, the valve is faulty and should be replaced before using the system again.

Rear Panel Controls and Indicators

Power Switch

The main power switch turns line power to SolventTrak ON (1) or OFF (0). SolventTrak is continuously active while power is on, and will constantly detect peaks present in the input signal according to its current Slope/Width settings. Line power can be left ON over extended time periods, if desired.

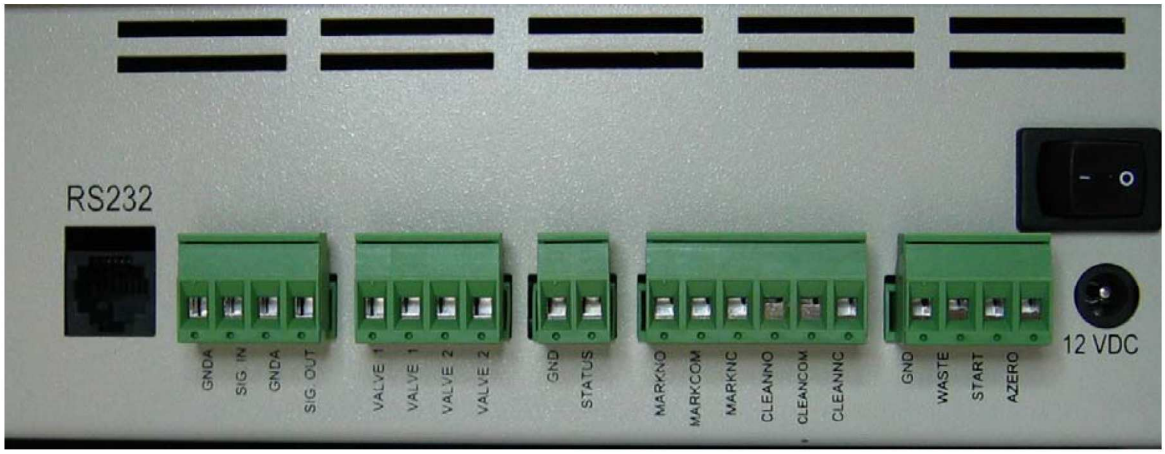


Figure 5.1. Rear Panel Controls

Corrosives

It is advisable to prevent corrosive solvents or other materials from coming into contact with the SolventTrak unit, any of its wiring connections and the diverter valve body. **Solvent leaks should be repaired as quickly as possible to prevent potential damage due to spillage.**

Safety

SolventTrak is not intended to be serviced by users.

DO NOT UNDER ANY CIRCUMSTANCES OPEN ITS CASE WHILE LINE VOLTAGE IS CONNECTED! Doing so may expose internal electrical connections inside and create an electrical shock hazard.

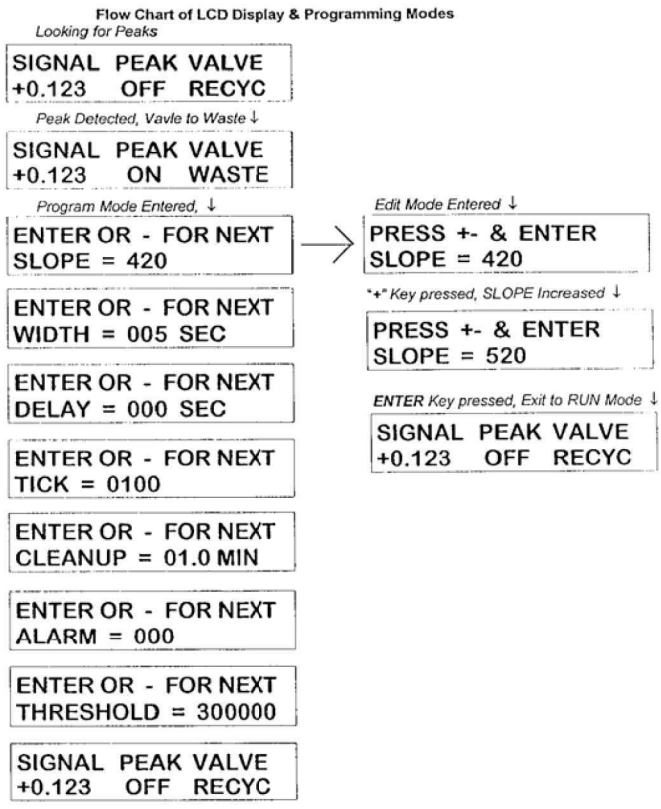
If a functional problem occurs, contact Axxiom Chromatography or your Axxiom dealer for service.

Appendix A Programming Steps and Procedures

To change a SolventTrak setting..

1. Press the "**ENTER/MENU**" Key once.
2. The first setting choice will be displayed, along with its current setting value. If it is not the setting you wish to change, press the "**-**" **(minus/down arrow)** Key in order to scroll down to the next setting. Repeat this until the desired setting is presented, then press the "**ENTER/MENU**" key. You will be placed in the EDIT mode for that setting.
3. Use the "**+**" & "**-**" **(plus/up arrow & minus/down arrow)** to increase or decrease the setting. *NOTE: Some settings have automatic step size increase/decrease where that amount of change will vary as the setting nears minimum or maximum.*
4. When the desired value has been set, press the "**ENTER/MENU**" key to make the setting permanent and return to the RUN mode.

NOTE: Normal Peak Detection will stop While in EDIT mode. It will automatically resume, using the new settings, after returning to RUN mode.



Examples of display screens during RUN, PEAK DETECTION & EDIT modes.