

Buck-Chrom
*BLC-20S PLUS*TM
HPLC Systems



with

*Buck-Chrom*TM [Iso/Bin]
HPLC Management System
for the
BLC-20S Series

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
PART 1 <i>Buck-Chrom</i> for the BLC-20S PLUS™ Introduction	
1.0 Introduction	1
1.1 General	1
1.2 Layout	1
1.3 Warranty	1
2.0 Installation.....	2
2.1 Packing List	2
2.2 Computer Requirements.....	2
2.3 Installation.....	2
2.3.1 General	2
2.3.2 Connecting the BLC-20S PLUS™	2
2.3.3 Pump Control.....	3
2.3.4 Pressure Monitor	3
2.3.5 Detector Control.....	3
2.3.5.1 Variable Wavelength Detector Control	3
2.3.5.2 Other Detector Control.....	3
2.3.5.3 External Data Output	4
2.3.5.4 Event Mark.....	4
2.3.6 BLC to Computer & DAS Cable Assembly	4
2.3.7 Installing the Software	5
Part 2 <i>Buck-Chrom PLUS™</i> Software	
1.0 Software.....	6
2.0 Installing the <i>Buck-Chrom PLUS™</i> Software and Manuals	6
2.1 General	6
2.2 Before Installing <i>Buck-Chrom</i>	6
2.3 Opening the <i>Buck-Chrom PLUS</i> CD	7
2.4 Manuals	7
2.5 Installing <i>Buck-Chrom</i> ™	8
2.6 Installing the <i>Buck-Chrom</i> ™ SUPPLEMENTAL DISK Files.....	9
3.0 Corrections and Errata.....	10
3.1 Regional Settings.....	10
3.2 Changes to Quick Guide to Set-Up and Start-Up	10
3.3 <i>Supplements</i> to the <i>Buck-Chrom</i> Manuals	10
4.0 Factory Test Record and .ini File	10

PART 1 *Buck-Chrom™* for the BLC-20S PLUS™ Introduction

1.0 INTRODUCTION

1.1 General - This manual contains information for the installation and operation of the *BLC-20S PLUS™ Buck-Chrom* HPLC Data System.

1.2 Layout - This manual is divided as follows:

Part 1 - Presents a general overview, system and installation information on the BLC-20S PLUS *Buck-Chrom™* HPLC Data System.

Part 2 - Contains the Instructions for preparing to install and installing the Software. The software is found on a diskette (the *Buck-Chrom™* supplemental files) and a CD that contains this manual in .pdf form and :

a *Quick Guide to Set-Up and Start-Up* [Quick Guide] of the *Buck-Chrom™* HPLC System.

Buck-Chrom installation software. This software automatically identifies and activates the pump control and wavelength features of the *PLUS* system. No other action is required.

the Guide to Using Buck-Chrom HPLC Management Software [Buck-Chrom HPLC Control Manual] – a complete operator's manual for the *Buck-Chrom™* HPLC Management Software - programming information for data acquisition, data processing, manipulation and reporting, and instrument control is in this section.

the Guide to using Buck-Chrom's Calibration Module [Calibrations] - the operator's manual for the *Buck-Chrom™* Calibration Module Program with programming information including standards development, curve processing, and calibration use.

1.3 Warranty - No other warranty exists, expressed or implied, except as shown here in, and in Buck Scientific's conditions of sale. Fitness to a particular application must be determined by the user. Parts and labor are warranted for one year, provided that the equipment is not damaged by improper use or by chemical spill.

The user must obtain a return authorization (RA) number from the factory in order to return goods for repair. This number should be marked on the outside of the shipping container. No parts will be accepted back which may represent a health hazard to service personnel. All repaired units will be returned via parcel post or mail unless we are authorized to ship by other means.

2.0 Installation

2.1 Packing List

BLC-20S PLUS™ Buck-Chrom™ HPLC Data System [DS015-0021-4 (115VAC) or 015-0021-5 (230VAC)] includes:

BLC-20S PLUS™ Buck-Chrom™ HPLC Management System Hardware Assembly (installed in Pump Module) [DS017-0063 series]...1 assembly

BLC-20S PLUS™ Buck-Chrom™ HPLC Management System Software [DS045-0022]...1 CD

BLC-20S PLUS™ Buck-Chrom™ Instrument.ini file and any updates [DS010-0196-1]...1 diskette

Cable Assembly, BLC-20S PLUS to Computer, High Density DB15M to Computer Serial DB9F [DS010-0272-1]...1 ea

Cable, Remote Control, Variable WL Detector [DS010-0028-3]...1 ea (Incl Recorder/Integrator)

BLC-20S PLUS™ Operator's Manual (this document) [DS 050-0007-5]...1 ea

Additional Special Order Items which may be included in the shipment:

Adapter, DB 9 Male to DB 25 Female [DS 232-0006]

Adapter Cable, DB 9 Male to USB Port w/ Driver Software [DS 025-0215]

If a Buck autosampler was also ordered with the system a multi-connector cable [DS010-0272 series] will be provided

If the Detector 2 option was ordered then a 5 pin DIN Male (Locking) to Wire Terminal (010-0288) will be provided

Contact your Dealer/Representative if you discover any items missing.

2.2 Computer Requirements - A minimum Pentium computer 166 MHz, one free COM port, and Windows 98/NT/XP/2000 operating system is required to run *Buck-Chrom™*. The COM port is required for the RS-232 serial connection to the BLC-20S PLUS rear connector.

2.3 Installation

2.3.1 General - After opening the shipping container, inspect the packaging for any damage. Record damage for making a claim. Check the packing list to ensure all items are included.

2.3.2 Connecting the BLC-20S PLUS - Identify compact/High Density (HD) DB 15F connector on the rear of the BLC-20S PLUS pump module. Connect the HDDB 15M pin shielded cable to the BLC-20S HDDB 15F connector and

then the DB9F plug on the cable to the DB 9 pin male receptacle on the COM port on your computer. Make sure that the connectors are pushed firmly into the receptacle on both ends of the cable and that the screws are tightened. A poor connection may result in lack of control of the system.



RS-232 Adapter (Special Order) - If the serial port on the computer is a 25 DB then you may need to order a DB 9 pin to DB 25 pin adapter which connects the RS-232 DB 9 Cable with the COM serial port.

If you only have USB ports on the computer, then you will need to order a Serial to USB Adapter Cable (025-0215).

2.3.3 Pump Control - The pump is controlled internally. There is no external control of the pump except from the pump controls on the front panel of the DVW-10. When the Buck-Chrom *PLUS* software is communicating with the pump, the pump flowrate display should change to 0.02 when the instrument is powered ON.

2.3.4 Pressure Monitor - The pressure monitor is connected internally. There is no external pressure output.

Setting of system pressure alarms in the method file of the software is part of the method development procedure.

2.3.5 Detector Control - The BLC-20S detector (DVW-10 series) is connected to the pump module with the Remote Control Cable [DS010-0028-3] to the Buck-Chrom *PLUS* PCB.

2.3.5.1 Variable Wavelength Detector Control

The BLC-20S's variable wavelength detector is controlled by the *Buck-Chrom* software via the remote cable above and on the front panel of the DVW. **You CANNOT** control the detector functions externally while the detector is attached to the pump module. Consult the factory if you desire to externally control parts of the system.

2.3.5.2 Other Detector Control

Follow the guidelines in the *Buck-Chrom* manuals for configuring the software for a non-variable wavelength detector.

If the BLC-20S was shipped with dual channel capability, then the second detector will be connected using the Detector 2 5 pin DIN to wire terminal cable [010-0288]. Wavelength cannot be controlled on Detector 2. The cable plug can be locked on to the panel connector.

2.3.5.3 External Data Output

Consult the factory.

2.3.5.4 Event Mark

The BLC-20S *PLUS* system is equipped with a position sensor on the injection valve (for event mark) and has a separate Event Mark button on the DVW-10 front panel.

Hint 1: It is a good idea to connect the RS 232 portion of the 010-0272 cable to your computer before power-on. Make sure you tighten the holding screws so that the plugs fit snugly to the 9 pin receptacles. An additional adapter may be required if the serial port is a DB 25 connector *or a USB port*.

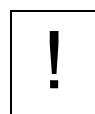
2.3.6 BLC Detector 2 Cable Assembly [010-0288]

BLC PLUS DETECTOR 1 & 2 CABLE ASSEMBLY CONNECTIONS

See the DVW-10 Manual for the 010-0028-3 Detector 1 Remote Cable connections. Pin connections are identical on both of the DB15M connectors.

The 010-0288 Detector 2 Cable Assembly supplied with the BLC-20S *PLUS* has a compact 5-position male DIN -type connector on one end and wires for a detector connection. The functions of the wires are listed in the table below.

Signal	5 Pin DIN Male Pin No	Logic or Analog	External Cable Wire Color
Channel 2 Integrator / Data (+)	5	Analog output	Red
Channel 2 Integrator / Data (-)	2	Analog output	Black
AutoZero	3	Digital input	Green
Digital Ground	4	Digital	White
Shield/Hood	None	Detector Chassis Ground	Green/Yellow



IMPORTANT FOR BEST OPERATION:

Connect the Green/Yellow wire of the Detector 2 cable to the other detector's ground terminal. Make sure that the Detector 2's power cord is plugged into the same outlet as the pump module. Isolate (cover) the tinned leads of unused wires so that they do not create ground loops and introduce noise into the system.

2.3.7 Installing the Software – The *Buck-Chrom*TM *PLUS* software is on the CD and diskette supplied. Follow the directions in Part 2, *Buck-Chrom PLUS*TM *Software*. Software manuals are on the disks and may be printed out.

PART 2 Buck-Chrom PLUS™ Software

1.0 SOFTWARE

The *Buck-Chrom PLUS* Software runs from *Buck-Chrom* Software and the supplemental *Buck-Chrom Supplemental* diskette that are in the License Agreement Envelope located with the *Factory Test Record*. The *Buck-Chrom* software enables the pump and wavelength control features of the BLC-20S PLUS / *Buck-Chrom PLUS*. Pump control is set on Pump A as an isocratic system. Wavelength control is accomplished on the General Page.

Opening the envelope constitutes agreement with the conditions of the License.

2.0 INSTALLING THE *Buck-Chrom PLUS*™ SOFTWARE AND MANUALS

2.1 General

The *Buck-Chrom PLUS* software and manuals are provided on a CD and a diskette in the License Agreement envelope.

The following files are on the *Buck-Chrom PLUS* CD:

<i>Buck_Chrom XXXX binary.EXE</i>	<i>Quick Guide Buck-Chrom -revX.pdf</i>
<i>Acrobat ReaderX Install.exe</i>	<i>Calibration Module.pdf</i>
<i>50-07-5RevX.pdf</i>	<i>Buck-Chrom HPLC Control.pdf</i>
<i>Read_Me_First-SCXXXXBinCDMsgX.doc</i>	

where XXXX is a version number

The following files are on the *Buck-Chrom* Supplemental diskette:

BuckSciMicro.ini updated files [see note with the License Agreement envelope]

2.2 Before Installing *Buck-Chrom*

Before Installing *Buck-Chrom* software, there are two options to choose from to prepare the hard disk of your computer.

Un-install Option [Recommended for Demo Versions]

NO ACTION IS REQUIRED IF THERE WAS NO PREVIOUSLY INSTALLED *BUCK-CHROM* PROGRAM.

Prior to loading the software, it is advisable to "un-install" any previous version of *Buck-Chrom*.

Using the *Start...Run* function or *Windows™ Explorer*, locate the *Unwise.exe* or *Unwise32.exe* file in the Buck Chrom folder/directory and select or double click on it. This should remove all previously installed *Buck-Chrom* program files. It will not remove data files or added user named files. These may be deleted or moved to another folder. You may choose to leave them without affecting the installation of the new version of *Buck-Chrom*.

2.3 Opening the *Buck-Chrom PLUS* CD

Before starting, identify the drive names/letters of the hard disk drive, the CD drive and the 3-1/2" diskette drive.

We recommend that you read the Quick Guide manual on the CD prior to installing *Buck-Chrom*. It requires *Acrobat Reader*. If you do not have *Acrobat Reader X.0* installed, you will need it to view all the manuals. Earlier versions of the *Reader* may not work as well as 4.0 or later for displaying graphics. Use the instructions described below to locate the *Acrobat ReaderX Install.exe* on the CD. Install the *Version X.0* by double clicking on the *Acrobat ReaderX Install.exe* and following the installation instructions. If *Acrobat Reader X.0* is already installed, open the *Quick Guide Buck-Chrom-revX.pdf* on the CD (or update on the diskette) and review them.

Put the CD into the CD drive. Normally Windows will automatically access a CD. If Windows is configured so that the CD does not automatically start, use one of the following methods to access the CD files:

Click on *Start...Run* and use the *Browse* to select the CD drive.

Double click on the *My Computer* icon then click on the CD drive.

Open *Windows Explorer*, then go to the CD drive.

Again, we recommend that you read the Quick Guide manual on the CD prior to installing *Buck-Chrom PLUS*. Install *Acrobat Reader*, then open the *Quick Guide Buck-Chrom revxxx.pdf*.

2.4 Manuals

Create a folder called *manuals* and then copy the *Buck-Chrom PLUS* and *Buck-Chrom* manuals into it. We recommend that this be done after you have installed *Buck-Chrom*. The manuals are in *.pdf* format.

The manuals are entitled:

Quick Guide to Buck-Chrom Set-Up and Start-Up

Guide to Using Buck-Chrom HPLC Management Software [Buck-Chrom PLUS HPLC Control Manual]

Guide to using Buck-Chrom's Calibration Module [Calibration instructions]

If you desire to review the manuals before you exit the *CD*, double-click on any of the manual icons.

After the *CD* is removed you may access the manuals from:

Windows Explorer: *Buck-Chrom XXXX FOLDER...manuals FOLDER... manual .pdf* files;

Desktop Shortcut: Create shortcuts from each of the *.pdf* files to put on the Desktop;
or

CD: Reinsert the *CD*. Follow your original steps to find the *CD* and to open the *CD* files directory. Double-click on the *.pdf* file icon to open a manual.

You may also print the manuals from *Acrobat Reader*.

2.5 Installing *Buck-Chrom*[™]

Using *Windows Explorer*, locate:

Read_Me_First-SCXXXXMsg.doc
Buck_Chrom XXXXX Binary.EXE

Open the *Read_Me_First* document file (if present) to review any pre-installation information/notices.

Install *Buck-Chrom* by double clicking on the *Buck_Chrom XXXXX Binary.EXE* file, then follow the steps using the guidance below:

Buck-Chrom FOLDER

During the installation process you will be asked where to locate *Buck-Chrom*. We recommend that it be installed in *C:\Buck-Chrom XXXX* as suggested. If it is necessary to locate it in another folder/directory or drive (not *C:*), then you will need

to make adjustments when you first open the Buck-Chrom program in the .wav folder box on the *Scales & Zeros* page, then save the .ini file. Also save the solvents, columns and method files to ensure the new Buck-Chrom location is recorded for these Startup files. Data Logging may also need to be adjusted.

BACKUP & RE-INSTALLATION

The backup option (as described above) is recommended for previous version Buck-Chrom files. Backing up files is especially critical should it become necessary to re-install the same version of the software. Although the install program will use the original folder and will replace only files with the same name and the same date/time (original installation), we recommend that you create a sub-folder called *Backup* and copy all the data, startup, .ini and configuration files to it.

After the *Buck-Chrom* installation is completed:

Create a *Manuals* folder and copy the .pdf manuals to it.
Compare the files listed below with the files installed on the hard drive.
Create desktop shortcuts for the *Calibrations*, *Buck_Chrom*, and *Viewer (.exe)* programs.

Buck-Chrom Installed Files:

Directory of C:\Buck-Chrom **FOLDER**

DataFiles [**Folder**] wav files [**Folder**] Manuals [**Folder**] *installed by user*

Calibrations.exe	Pslcrun.nbr	StartUpPeakIDs.pip
Columns.clm	ReportIcon.BMP	StartUpZeros.zer
BuckSci.ICO	Solvents.slv	StartUp.lcm
BuckSciMicro.ini	Buck_Chrom XXXX.exe	StartUp.pod
Install.log	StartupCalibrDB.CDX	Unwise.exe
Loading Study	StartUpCalibParam.dat	Unwise32.exe
verXXXX.spm	Startupcalibrdb.dbf	Viewer.exe
PeakPickStartup.val	Startupcalibrdb.fpt	
PeakPick.rtf		

where XXXX is a version number

2.6 Installing the *Buck-Chrom* SUPPLEMENTAL DISK Files

Locate the 3.5" SUPPLEMENTAL DISK diskette. One file needs to be copied into the C:\Buck-Chrom folder or the folder where you designated *Buck-Chrom* to be installed. This file is:

BuckSciMicro.ini - An updated version of the *Buck-Chrom* configuration file which contains settings specific to the tested unit (the auto-installed *BuckSciMicro.ini* file is generic and does not match with the shipped units)

Other *update* files may need to be copied in to the *Buck-Chrom* folder or one of the sub-folders (see specific instructions with the license agreement envelope).

Insert the diskette into your computer's 3.5" drive and then, using *Windows Explorer*, copy the files to the *Buck-Chrom XXXX* directory folders.

3.0 Corrections and Errata

3.1 Regional Settings **CORRECTION TO PAGE 7 - QUICK START GUIDE**

For applications (countries) using the " , " (comma) in place of the " . " (period) decimal point, it is **NOT NECESSARY** to change the decimal point parameter from *comma* to *period* in the *Regional...Numbers* box. Buck-Chrom will interpret the regional options found in the *Start...Settings* menu.

3.2 Changes to Quick Guide to Set-Up and Start-Up

The Buck-Chrom Interface Box is not used in a BLC-20S PLUS system. The Buck-Chrom PLUS PCB is installed inside the BLC-20S pump module.

3.3 Supplements to Buck-Chrom Manuals

Other changes to Buck-Chrom may be contained in *Supplements* immediately following this section, on the CD, and/or on the 3.5" Diskette.

4.0 Factory Test Record and .ini File

A system *Factory Test Record* and a printout of the *BuckSciMicro.ini* file are enclosed with the License Agreement Envelope. These documents should be kept as they contain information concerning the system components, software, and offsets.

The *.ini* information should be used to restore the Buck-Chrom settings should the diskette or hard-drive *BuckSciMicro.ini* files become corrupted. If it becomes necessary to change some of the settings, record the new values on the printout sheet.

You may view and printout the *BuckSciMicro.ini* file in *Notepad* (or other text editor) by double clicking on it from *Window Explorer*. Always save the changes you make to the settings in the Buck-Chrom program by selecting the Save *Ini File* option from the *I*ni File menu bar.

If you need to make changes to the *.ini* file from the editor, you must exit *Buck-Chrom* for the changes to take effect (e.g. changing COM ports).

Supplement to Operator's Manuals

BLC-20S AUTOMATED ISOCRATIC STACK HPLC SYSTEMS

BLC-20S PLUS Systems

015-0030 / 015-0030-1 SS
015-0030-2 / 015-0030-3 PEEK
015-0030-4 / 015-0030-5 HIGH PRESSURE SS

015-0050 / 015-0050-1 SS
015-0050-2 / 015-0050-3 PEEK
015-0050-4 / 015-0050-5 HIGH PRESSURE SS

Supplement to
Operator's Manuals

BLC-20S SERIES AUTOMATED (PLUS) ISOCRATIC HPLC SYSTEMS

015-0030 BLC-20S PLUS, SS, 115VAC
015-0030-1 BLC-20S PLUS, SS, 230VAC
015-0030-2 BLC-20S PLUS, PEEK, 115VAC
015-0030-3 BLC-20S PLUS, PEEK, 230VAC
015-0030-4 BLC-20SP PLUS, HIGH PRESSURE, SS, 115VAC
015-0030-5 BLC-20SP PLUS, HIGH PRESSURE, SS, 230VAC

&

015-0050 BLC-20S PLUS 2 CHANNEL, SS, 115VAC
015-0050-1 BLC-20S PLUS 2 CHANNEL, SS, 230VAC
015-0050-2 BLC-20S PLUS 2 CHANNEL, PEEK, 115VAC
015-0050-3 BLC-20S PLUS 2 CHANNEL, PEEK, 230VAC
015-0050-4 BLC-20SP PLUS 2 CHANNEL, HIGH PRESSURE, SS, 115VAC
015-0050-5 BLC-20SP PLUS 2 CHANNEL, HIGH PRESSURE, SS, 230VAC

Contents

<u>Section</u>	<u>Contents</u>
1.0	Introduction and Specifications
2.0	Installation
3.0	Operation
4.0	Theory of Operation
5.0	Maintenance
6.0	Troubleshooting
7.0	Replacement Parts

Section 1.0 Introduction

This supplement to the BPM-200 Series and detector Operator's Manuals contains additional information needed to install, operate, and perform user maintenance on the Series I Isocratic HPLC Pump, and operate it with the *Buck-Chrom PLUS* system. Compare this to the standard BPM Operator's Manual and other instructions provided with the system.

1.1 Description of the BLC-20S PLUS SYSTEM

The principal differences between the BLC-20S *PLUS* system and the standard binary versions are:

The BPM-20S *PLUS* is a single pump - isocratic only version.

The BPM-20S has a two-way prime/purge valve (PPV) that is plumbed internally.

The injector valve is installed internally. Units may have a through-the-handle (T-t-H), C1 model, or a sample injection port version (C2). If it has a C2 valve, it will have a "sample injection" port above the valve.

The front panel has two outlet ports: a bulkhead out to the column, and a waste tubing outlet.

There is no external pump hand-held controller.

There is no external RS232 pump control connector.

There is a detector connection (*Detector 1*) for input to the automation hardware. On the two channel version there is a second detector connector (*Detector 2*).

There is a *Computer/Aux* connector in place of a *Pressure/Aux* connector.

Pump control functions are added to the detector's display (see below).

If the unit is a high pressure version, then the detector's front panel will also display Pressure and Limits.

The *PLUS* versions have built in HPLC management hardware (printed circuit board - PCB) and software - *Buck-Chrom* single or dual channel.

Only Pump A is available in the control software.

The pressure transducer installed in the pulse damper is connected directly to the *Buck-Chrom PLUS* PCB. Pressure is displayed and pressure limits are set in the software.

Additional control/communication with an Autosampler can be accomplished through contact closure and special cables.

The flow rate of the Series I pump fitted with a standard 10 mL pump head can be set in 0.01 mL increments from 0.01 to 10.00 mL/min for operating pressures to 2500 psi. It is available in either type 316 stainless steel or bio-compatible PEEK. Higher pressure can be achieved with a stainless steel "P" version. The high pressure P version must be ordered if pressure display on the front panel is needed.

1.2 Pressure Conversion Factors: psi & bar

To convert from psi to bar, multiply *psi* by 0.0689 (1000 psi x 0.0689 = 68.9 bar)

To convert from bar to psi, divide *bar* by .0689 (400 bar / 0.0689 = 5805.5 psi)

Section 2.0 Installation

2.1 Solvent Preparation

Proper solvent preparation will prevent a great number of pumping problems. The most common problem is bubble formation, which may affect the flow rate consistency. Aside from leaky fittings, the problem of bubble formation arises from two sources: solvent out-gassing and cavitation. Filtration of HPLC solvents is also required. See the pump section of the Operator's Manual for further details on Solvent Out-gassing and Sparging, Cavitation, and Filtration.

2.2 Solvents With Harmful Effects

All portions of the BLC Series pump, injection valve, column, and fluid path that contact the mobile phase are manufactured of stainless steel, PEEK, sapphire, ruby, or fluorocarbon polymer. Observe caution when using solvents that have extreme pH values. See the operator's manuals for detailed information.

2.3 PACKING LIST: BLC-20S and BLC-20SP Plus Versions

BLC-20S PLUS, SS, 015-0030, 115VAC; or BLC-20S PLUS, SS, 015-0030-1 230VAC
BLC-20S PLUS, PEEK, 015-0030-21, 115VAC; or BLC-20S PLUS, PEEK, 015-0030-3,
230VAC

BLC-20SP PLUS, SS, 015-0030-4, 115VAC; or BLC-20SP PLUS, SS, 015-0030-5,
230VAC

BLC-20S PLUS 2CH, SS, 015-0050 115VAC; or BLC-20S PLUS 2CH, SS, 015-0050-1
230VAC

BLC-20S PLUS 2CH, PEEK, 015-0050-2 115VAC; or BLC-20S PLUS 2CH, PEEK, 015-
0050-3 230VAC

BLC-20SP PLUS 2CH, SS, 015-0050-4 115VAC; or BLC-20SP PLUS 2CH, SS, 015-0050-
5 230VAC

BLC-20S Pump Module, with Injection Valve and Installed 1 or 2 Channel *Buck-Chrom*
PLUS PCB

Column Holder (Channel)

Isocratic Pump Accessory Kit

DVW-10 with UV Lamp - BLC-20S and Analytical Flowcell, and detector Accessory Kit

Cables for detector(s), computer, and auxillary devices

Injection Valve supplemental instructions (C1 or C2)

Supplement to Operator's Manuals (this document) [DS090-0056]

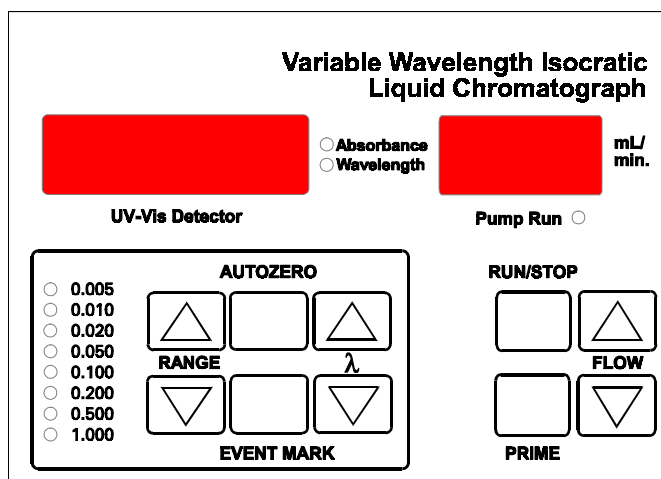
Section 3.0 OPERATION

3.1 Front Panel Components, Indicators, and Controls



The front panel of the BLC-20S pump module (BPM-20S) is shown at the left.

It has two indicator LEDs, the pumphead and inlet port, a prime/purge valve, an injection valve with a 20uL sample loop (see separate instructions), an outlet to the column, and a waste tubing outlet port.



DVW-10 Front Panel

Components

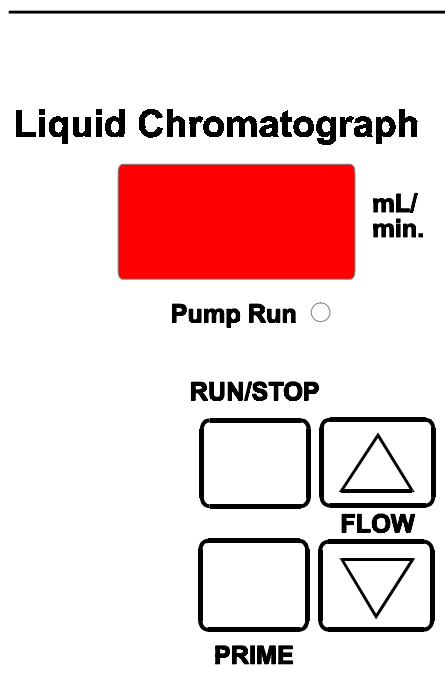
The prime purge valve (at right) is plumbed internally. Use the large syringe provided to prime the pump as described in the operator's manual.



The *Outlet to Column* bulkhead is connected to the outlet port of the injection valve. Connect the tubing provided in the accessory kit from the bulkhead to your column. Use the bulkhead fittings on the bulkhead end and the one-piece fittings at the column end.

One waste tube is from the sample loop overflow. If the unit is shipped with a T-t-H valve, then a second waste tube is from the injection port 5 for the sample port backflush option (see valve instructions).

3.1.1 Pump Controls (Detector Display Panel)

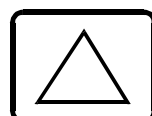


BLC-20S Series Pump Keypad

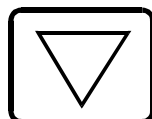
3.1.1.1 Digital Display

The 3-digit display shows the pump flow rate (mL/min).

3.1.1.2 UP and DOWN Arrow Control Buttons



When pressed, this **UP** arrow button increases the flow rate, or increases the upper and lower pressure limits.



When pressed, this **DOWN** arrow button decreases the flow rate, or decreases the upper and lower pressure limits.

Fast And Slow Button Repeat

If the UP arrow or DOWN arrow button is held down for more than approximately one half of a second, the button press will repeat at a slow rate of approximately 10 times a second. Once slow button repeat has begun, fast button repeat can be initiated by using a second finger to press down the opposite arrow button.

During fast button repeat, the button press will repeat at a rate of approximately 100 times a second. Switching back and forth between repeat speeds can be accomplished by pressing and releasing the second arrow button while keeping the first arrow button held down.

RUN/STOP



3.1.1.3 RUN/STOP and PRIME Buttons

When pressed, the **RUN/STOP** button alternately starts and stops the pump. It also resets the pump after a fault has occurred.



When the **PRIME** button is pressed, the pump runs at the maximum flow rate for the pump head until a pressure of 200 psi is achieved. The pump stops and the fault LED lights indicating that the pump is primed. Reset the fault light by pressing the

RUN/STOP button. The prime function will also stop when any button is pressed.

3.1.1.4 Status LEDs

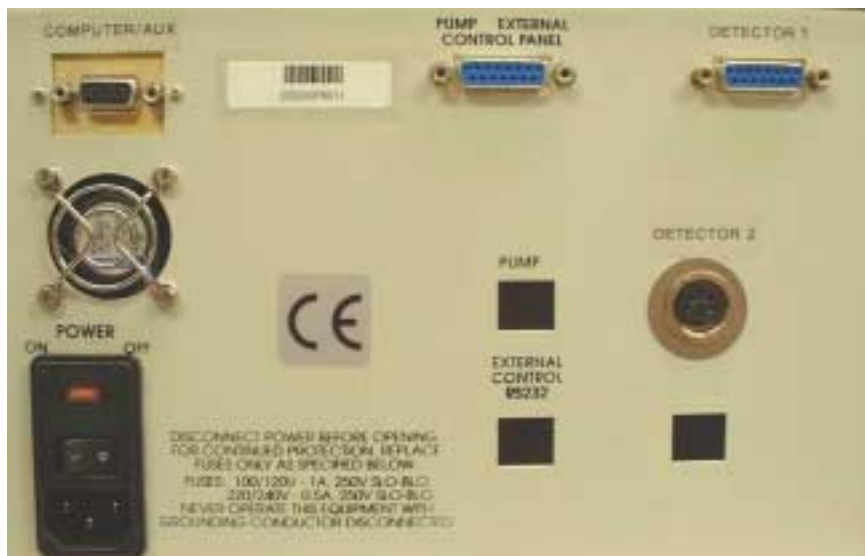
3.1.1.4.1 BPM Status LEDs

The green LED lights to indicate that the BPM module is powered.
The yellow LED lights to indicate that the pump is ON and is being controlled by the internal hardware/software or from the front panel of the detector.

3.1.1.4.2 DVW Status LED

The green LED lights to indicate that the pump is running.

3.2 Rear Panel Connections



The BPM-20S rear panel is shown at the left.

Rear Panel of BLC-20S Pump Module (BPM-20S)

3.2.1 External Pump Control

Locate the *PUMP EXTERNAL CONTROL PANEL* DB15F receptacle on the BPM-20S shown at the right.

Locate the DB15M connector of the cable on the rear of the detector.



Attach the cable plug to the DB15F receptacle and secure the two screws on the plug to the hex nuts on the panel as shown on the right.

NOTE: If the pump cable from the rear of the detector is not attached to the rear of the pump module connector, the pump-side display on the detector front panel will not be powered.

! **CAUTION** DO NOT connect any other cable to the *PUMP EXTERNAL CONTROL PANEL* receptacle. You may cause damage to the pump PCB and/or to the instrument connected to the other end of the cable.



An external Hand-held Controller (see operator's Manual) may be ordered separately. Consult your Distributor/Sales Representative.

3.2.2 Detector Control

3.2.2.1 Detector 1 Control

Locate the detector DB15M double ended Remote Cable (010-0221) in the Accessory Kit. Attach and secure (to the hex nuts) one end of the cable to the variable wavelength detector's *RECORDER/INTEGRATOR OUPUT* DB15F receptacle. Attach and secure the other end to the *DETECTOR 1* DB15F receptacle shown at the right.



NOTE: Wavelength may only be set from/on the *DETECTOR 1* channel. See the *Buck-Chrom* instructions for settings when other types of detectors are to be used on/as *DETECTOR 1*.

3.2.2.2 Detector 2 Control (for 2 Channel PLUS Systems)

Units with a second channel (*DETECTOR 2*) have a special DIN connector for this cable. Analog integrator and autozero functions are available on this receptacle (shown at right).

Locate the *DETECTOR 2* cable (010-0288). Attach the 5-pin DIN male plug to the 5-pin DIN female panel receptacle marked *DETECTOR 2*. Turn the outer ring of the plug shield clock-wise to lock the plug in to the receptacle.



Attach the tinned wire leads of the cable to the second detector. Functions and wire colors are on the label at the wire-end of the cable.

3.2.3 Computer Connection



Locate the *COMPUTER/AUX* high density/compact (HD) DB15F connector on the panel (shown to the left).

Locate the BPM (HDDDB15M) to Computer (DB9F) Cable (010-0272-1). Attach and secure the HDDDB15M end to the rear panel.

Attach and secure the other end to the serial port (DB15M) of the computer.



CAUTION DO NOT attach any other cable to this receptacle or the *Buck-Chrom PLUS* PCB may be damaged. Consult your Distributor/Sales Representative or the factory for wiring information.

NOTE 1: Connect the computer cable prior to powering the system ON.

NOTE 2: See the *Buck-Chrom* instructions for using/ordering adaptor cables if your computer does not have a DB9M serial port.

3.2.3 Auxillary Connection

The HDDDB15F *COMPUTER/AUX* receptacle is configured for autosampler and other functions. Consult your Distributor/Sales Representative or the factory for requirements and to order optional cables.

3.3 Power-up Configurations

3.2.1 Pressure Compensation

On power-up, press the PRIME button on the front panel while pressing the Power On switch of the CORCOM on the rear of the BLC. The pump will display a number from 0 to 25 for the 10 ml SS and PEEK heads (or 0 to XX for other pump heads). This represents the running pressure of the pump from 0 psi to 2500 psi. Each digit represents 100 psi.

Pressure compensation is used to maintain flow accuracy by compensating for the compressibility of the fluid. To make a preliminary setting, enter the compensation value at a factor of 100 lower than the pressure set point (e.g. if the pump is running at 2000 psi, then the pressure compensation value may need to be set at 19). The actual pressure compensation needed depends on the solvent(s) being used. To ensure accuracy in the method, a flow test for the particular condition(s) of the application should be conducted. This is accomplished by measuring the graduated flow over a 1-2 minute period.

To change the pressure compensation number use the UP arrow and DOWN arrow buttons. When you have selected the correct pressure compensation press the RUN/STOP button to return to normal operation of the pump.

3.2.2 Non-volatile Memory Reset

If the pump is operating erratically, there is the possibility that the memory has been corrupted. To reset the memory and restore the pump to its default parameters, turn the power off at the CORCOM, then press and hold the UP arrow button when the power is switched on. Release the button when the display reads “rES”. The parameters stored in non-volatile memory, i.e., the flowrate, the pressure compensation, the lower pressure limit, and the upper pressure limit will be set to the factory default values. The head type setting is the only parameter not changed by the non-volatile memory reset function. If the firmware is upgraded to a newer version, a non-volatile memory reset will automatically occur the first time the power is switched on.

3.2.3 Pump Head Setting Change

ONLY OPERATE A PUMP AT ITS DESIGNATED HEAD SETTING. Incorrect setting of the pump head may result in substandard performance or may damage the pump components. The pump parameters and drivers for flowrate, pressure and power compensation, and pressure limits for each type of pump head are stored in the firmware.

Your pump(s) has been programmed, tested, and shipped with the correct pump head setting.

If for some reason you are changing the pump head to a different size, or you are observing different values than those described in the specifications for the upper flowrate or pressure compensation of the installed pump head, then the pump head setting may need to be changed.

Display the pump head setting mode on power-up by pressing the RUN/STOP button. The default setting of “**5 10**” (S 10) should appear for a stainless steel 10mL/min pump head. If it is not showing, use the UP arrow button to cycle to it. For example, If you are replacing the SS head with a PEEK 10 mL/min head, push the DOWN button until you reach “**P 10**” (P 10). Push the RUN/STOP button to store the setting and to reset the display.

If the EPROM is removed or the battery is replaced, the default pump head setting on first power-up will be for a stainless steel 10 mL/min pump.

3.3 Power-Up Tests

3.3.1 Display Firmware Version Mode

The firmware version can be displayed during power-up by pressing and holding the RUN/STOP and the UP arrow buttons on the pump side of the DVW display panel when the power is switched on. Release the buttons when the display reads “**UEr**”.

The decimal point number shown on the display is the firmware version. To exit this mode, press the RUN/STOP button.

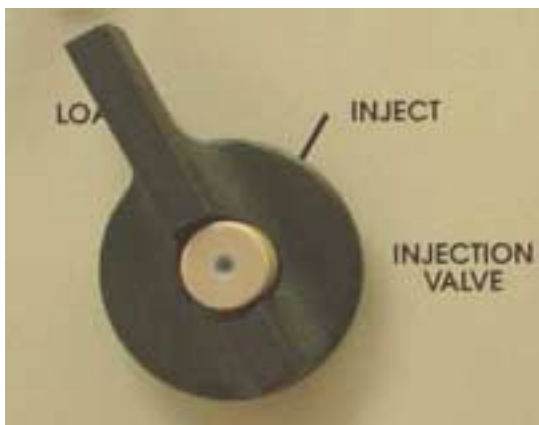
3.3.2 Align Refill Switch Mode. The signal that initiates the refill phase can be displayed during power-up by pressing and holding the PRIME and the UP arrow buttons when the power is switched on. Release the buttons when the display reads "rFL". When the slotted disk allows the light beam to pass from the emitter to the detector on the slotted optical switch a pulse will be generated which signals the beginning of refill. When this pulse occurs the three horizontal segments shown at the top of the display will turn off and the three horizontal segments at the bottom of the display will turn on. To exit this mode, press the RUN/STOP button.

3.3.3 Serial Port Loopback Test Mode. *Buck-Chrom PLUS* communicates with the pump using an internal serial connection. If *Buck-Chrom PLUS* cannot communicate with the pump, the serial port loopback test can be used to verify that the pump's serial port is functioning properly. During power-up press and hold the UP arrow and the DOWN arrow buttons when the power is switched on and then release the buttons. The display must show "C00" for the first half of the test to pass. If not, call your Service Representative. To exit this mode, press the RUN/STOP button.

Section 4.0 THEORY OF OPERATION

4.1 Mechanical Operation

4.1.1 Injection Valve Position Sensor/Event Mark



The BLC-20S *PLUS* unit is equipped with an Injection Valve Position Sensor as standard. A black handle indicates that there is a position sensor on the valve (C1 and C2 valves) and by its ring marked with the white letters "0" (for LOAD) and "1" (for INJECT) (C2 valve). The sensor is attached directly to the *Buck-Chrom PLUS* PCB.

The sensor provides continuous closure when the valve is in the INJECT ("1") position. The contact is +5V in the LOAD position and 9V in the INJECT position.

The closure signal is used to activate a relay in the chromatography system software/hardware and is used to signal that the injection has occurred and to start a chromatography run in the data system.

When used with the detector's Remote Control and Recorder/Integrator Cable, the closure signal is sent via the cable to activate the relay in the chromatography system software/hardware when the *EVENT MARK* button on the front panel is pressed. See the Remote Control Section of the Detector for additional information.

4.1.2 Pressure Sensing

The pulse damper has a built-in pressure transducer that senses fluid pressure. The output is sent to the transducer amplifier on the *Buck-Chrom PLUS* PCB. It provides the information presented on the software display.

4.1.3 Pressure Output

The pressure output from the transducer is sent directly to the *PLUS* PCB. There is no other external pressure output.

4.1.4 Pulse Damping

In the diaphragm-type pulse damper configured with the pressure transducer the amount of mobile phase in contact with the pulse damper is small, only 1.2 mL at 6,000 psi, and the geometry used insures that the flow path is completely swept, so solvent "memory effects" are virtually eliminated.

4.2 Remote Interfacing

4.2.1 General

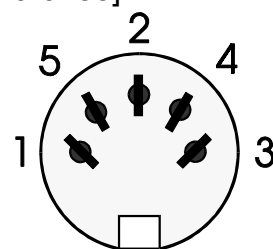
All communication and control between the pump and the *Buck-Chrom PLUS* hardware/software is wired internally. If a chromatography run is NOT in process, the pump may be controlled from the front panel of the detector (as in section 3 above).

DO NOT attempt to control the pump or the detector when an automated run is in progress.

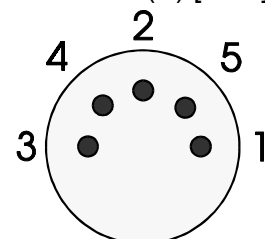
4.2.2 Receptacle/Cable Configurations

CABLE ASSEMBLY - INTEGRATOR DETECTOR 2 (WIRE END) [010-0288]

	PUMP MODULE DETECTOR 2 5 Pin DIN F- Locking (A)	5 Pin DIN M - WIRE TERMINALS (B)
Function	Pin	Color
Channel 2 Analog Out Inegrator /Data (+)	5	Red
Channel 2 Analog Out Inegrator /Data (-)	2	Black
AutoZero	3	Green
Digital Ground	4	White
Detector or Chassis Ground	Shield/Hood	Green/Yellow



Rear Panel Detector 2
Connector (A) [front]



Detector 2 Cable (B) [front]

CABLE ASSEMBLY - RS232 OUTPUT; PUMP MODULE TO COMPUTER [010-0272-1]

	PUMP MODULE COMPUTER/AUX DB15M HI DENSITY	COMPUTER SERIAL RS-232 DB9F
Function	PIN	PIN
RS 232 Transmit (TX)	3	2
RS 232 Receive (RCV)	4	3
Ground	7	5

Do not use any other pins. They may be connected to other functions. Applying power or other signals may cause damage to the *PLUS* circuits.

CABLE ASSEMBLY UNIVERSAL REMOTE [010-0221]

See the DVW-10 manual.

CABLE ASSEMBLY, DVW-10 TO PUMP MODULE EXTERNAL CONTROL

Consult Factory.

Section 5.0 Maintenance

Cleaning and minor repairs of the BLC Series I pump can be performed as described in the Operator's Manual and as outlined below.

NOTE: Lower than normal pressure, pressure variations, or leaks in the pumping system can all indicate possible problems with the piston seal, piston, or check valves.

5.1 Pulse Damper Replacement

5.1.1 Removing the Pulse Damper Assembly

WARNING: There are potentially lethal voltages inside the BLC case. Disconnect the line cord before removing the cover. Never bypass the power grounds.

1. Make certain that the system has been depressurized. Unplug the power cord and remove the cover.
2. Disconnect the tubing from the pulse damper.
3. Disconnect the green and white transducer wires on the blue 2 pin MTA from the *PLUS* circuit board and disconnect the red and black wires at the 2 pin in-line connector between the transducer and the pump PCB. Do NOT remove the pressure transducer.
4. Remove the four screws that secure the pulse damper to the underside of the chassis cabinet.
5. Remove the pulse damper.

5.1.2 Pulse Damper Refurbishing

Refurbishing the pulse damper is a time-consuming procedure. You may want to return the pulse damper to have it rebuilt. Do NOT remove the pressure transducer from the pulse damper until you have received written instructions. Do NOT attempt to refill or refurbish the pulse damper until you have a refurbishing kit. Instructions are furnished with the kit. Be sure to specify the diameter of the pulse damper.

5.1.3 Pulse Damper Installation

1. Position the pulse damper, aligning it with the four mounting holes in the bottom of the chassis cabinet. The pressure transducer should be pointed toward the rear of the chassis (as it was configured before removal).
2. From the underside of the BPM chassis, tighten the four screws to hold the pulse damper assembly in place.
3. Connect the tubing from the prime-purge valve to the port on the front of the pulse damper. Connect the line to the injection valve to the rear port.
4. Re-connect the transducer's wire harness connector to *PLUS* and pump PCBs as above.

5. Replace the cover.

5.2 Battery Replacement (if present)

The battery provides power for the memory that holds the current pump configuration. If the pump is set at a flowrate other than 1.00 or 10.0 and the power is turned off, when the power is turned back on the flowrate should appear as it was set. If this flowrate does not appear the battery will need to be replaced.

1. Unplug the unit.
2. Remove the cover.
3. Tilt the unit so that the Control PCB is facing up (with RS-232 connection). The battery can be seen in the near left corner of the circuit board. The battery is silver, circular, and has a positive pole mark (+) on the top. Gently pull it from its socket.
4. With the positive mark (+) up, gently slide the new battery into the battery socket. Be sure the battery is all the way into place. It must contact the base of the battery socket.
5. Replace the cover to the unit.
6. Reconnect the power line cord to the CORCOM.

See the Replacement Parts Section for the new battery part number.

Section 6.0 Troubleshooting

You Notice	This May Mean	Possible Cause	You Should
<ol style="list-style-type: none"> 1. Uneven pressure trace. 2. Pressure drops. 	<ol style="list-style-type: none"> 1. Bubble in check valve. 2. Leaks in system. 	<ol style="list-style-type: none"> 1. Solvent not properly degassed. 2. Fittings are not tight. 	<ol style="list-style-type: none"> 1. Check to be certain that mobile phase is properly degassed. 2. Check connections for leaks by tightening fittings.
<ol style="list-style-type: none"> 1. Fluid between the pump head and the chassis. 	<ol style="list-style-type: none"> 1. The piston seal(s) are worn. 	<ol style="list-style-type: none"> 1. Long usage time since last seal change. 2. Salt deposits on seal (especially if buffered aqueous mobile phases are used without the self-flush head.) 	<ol style="list-style-type: none"> 1. Replace piston seal. See Sections in Operator's Manual (O.M.). 3. Check the piston for salt deposits. Clean as necessary. See Section s in Operator's Manual.
<p>Pump makes a loud clanging or slapping noise (intermittent contact with cam).</p>	<p>Piston carrier is catching in piston guide.</p>	<ol style="list-style-type: none"> 1. Cap nut screws on the pump head are loose. 2. Seal(s) are worn. 3. Piston guide is worn. 4. Salt build-up on piston carrier from use of buffers. 5. Excess lubricant on piston carrier. 	<ol style="list-style-type: none"> 1. Check cap nut screws on pump head. Tighten if necessary. 2. Replace seals. 3. Replace piston guide and seals. See O.M. 4. Consider changing to a self-flushing pump head if using buffers. 5. Clean excess lubricant and dirt off piston carrier. See O.M.
<p>Blue dye in mobile phase.</p>	<p>Pulse damper diaphragm has burst.</p>	<p>Sudden pressure drop when purging system.</p>	<p>Replace or refurbish pulse damper. See Section 5.</p>
<p>Pump runs for 50 pump strokes, then shuts down.</p>	<p>Lower pressure limit is activating.</p>	<ol style="list-style-type: none"> 1. Mobile phase is not properly filtered. 2. Particles from worn seal trapped in the system (e.g., tubing, filters, injection valve, column inlet). 	<ol style="list-style-type: none"> 1. Check to be certain the low pressure limit is set to 0 psi. 2. Only increase the low pressure limit after the pump attains operating pressure. 3. Contact service technician.
<ol style="list-style-type: none"> 1. Pump shuts down after run is initiated even with no column connected. 2. Pump runs to maximum pressure and shuts down. 	<p>Clog in fluid system.</p>		<ol style="list-style-type: none"> 1. Remove and clean the inlet filter and/or column frits. See O.M. 2. If the problem persists, remove tubing from system one piece at a time until you find the clogged piece. Most clogs occur outside the pump itself.
<p>No pump display on detector front panel</p>	<p>No power</p>	<ol style="list-style-type: none"> 1. Detector to BPM cable not connected. 2. BPM not powered ON. 3. Pump PCB has lost power. 	<ol style="list-style-type: none"> 1. Attach cable securely. Consult Factory. 2. Power BPM ON. Check fuses. 3. Consult Factory.

See the Operator's Manual for detailed pump maintenance instructions.

Section 7.0 Replacement Parts

Part No.	Description
025-0186	PULSE DAMPER WITH PRESSURE TRANSDUCER, SS
025-0187	REPLACEMENT BATTERY FOR HIGH PRESSURE CONTROL PCB
025-0266-1	CABLE, REMOTE RC/INT, UNIVERSAL, DBL ENDED DB15M (RFSP)
025-0308-1	CABLE ASSEMBLY, BLC-20S PLUS to COMPUTER, HDDB15 - DB9F
025-0309	CABLE ASSEMBLY, BLC-20S PLUS 2CH DIN 5M - WIRE TERM

Quick Guide to Star-Chrom Set-Up and Start-Up



Star-ChromTM

HPLC Management System

D-Star Instruments
8424 Quarry Rd.
Manassas, VA 20110-5326
Phone: 703-335-0770
Fax: 703-335-9952
Toll- Free: 800- dstar12
E-Mail: dstarinstr@aol.com
Web Site: www.d-star.com

Created May 21 2000
Revised August, 2003

Contents

Specifications	3
Diagram of Interconnecting Cables	4
Installing the Star-Chrom Software and Manuals	5
Troubleshooting Control Via RS-232	8
Instructions on how to correct the wavelength display	9
Instructions on how to turn off wavelength control for fixed wavelength detectors ..	10
Instructions on how to zero and scale the pressure reading	11
Rear Panel of the Interface Box	12
Map and Description of Screen	13
Menu Items	14
Important Note: Starting up the system	16
Tabbed Pages:	
General	18
Samples	19
Comments	20
Data Logging	21
Col & Sol IDs	22
Solvent Prg	23
Peak Picking	24
Alarms	25
History	26
Plot1	27
Small Plot	28
Plot2	29
Scales & Zeros	30
Hardware	31
Peak IDs	32
Peak Results	33
Calibr	35
Report	36
Solvent Recycle Valve	38
Default parameters for the DStarLCMicro.ini file	39
Files that are installed on the hard disk	39



Star-Chrom HPLC Management System:

Windows 95/98/NT software for analytical HPLC data acquisition/control

Includes: Software, External interface box power supply and connecting cable

Specifications:

Control via RS-232 port; can be used with Laptop or Desktop computer 24 bit resolution; +/- 2.5 VDC input, dual channel

Pressure input (0-1VDC 10 bit)

User set frequency of acquisition up to 5 data points / second, all channels (maximum number of data points per run 40,000)

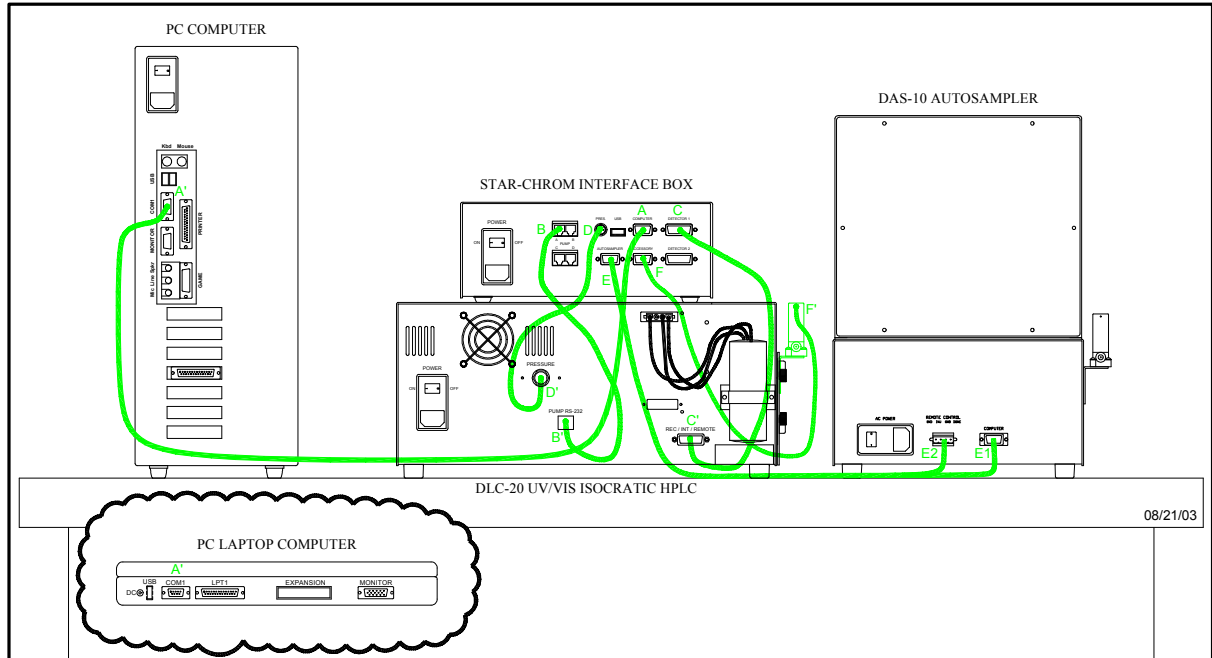
Contact closure for control of autosampler or injection valve; senses contact closure for start of data acquisition; control pump on/off and flow control via RS-232 signal; set and document D-Star variable wavelength detector

If a pressure signal is available, control of high and low pump pressure alarms

Software will control optional valve for recycling mobile phase using the Solvent Saver™ feature

Windows 95/98/NT compatible; software makes documentation of conditions and experiments easy; verbal feedback is available in the following languages: English, German, French; more languages to follow

Diagram of Interconnecting Cables



- A - Computer cable, Serial RS-232, DB9-M to DB9-F
- B - Pump control cable, RJ-11/6-plug to RJ-11/6-plug
- C - Remote control / signal cable for D-Star HPLC System, DB15-M to DB15-M
- D - Pressure cable, modular Hex5-M (or MiniDIN6-plug) to Hex5-M (or MiniDIN6-plug)
- E - Autosampler remote control cable, DB9-F to DB9-M & Molex5-M (Hydra cable)
- F - Accessory cable for recycle valve DBC15 (optional)

IMPORTANT! READ THIS FIRST

Installing the Star-Chrom Software and Manuals

The Star-Chrom software and manuals are provided on a CD and a diskette in the License Agreement envelope.

The following files are on the CD:

StarChrom_CD_Setup.EXE

CheckStarChromInstalled.exe

Autorun.inf

DStar.ICO

Quick Guide StarChrom.pdf

StarChrom HPLC Control.pdf

Calibration Module.pdf

ar405eng.exe

Before Installing Star-Chrom

Before Installing Star-Chrom, there are two options to choose in preparing the hard disk of your computer.

Uninstall Option [Recommended for Demo Versions]

Prior to loading the software, it is advisable to “uninstall” any previous version of Star-Chrom. This is especially true for demonstration versions of software with StarChrom.exe file dates prior to February 2000. Using the Start...Run function or Windows® Explorer, locate the Unwise.exe or Unwise32.exe file in the StarChrom folder/directory and select or double click on it. This should remove all Star-Chrom program files. It will not remove data files or added user named files. These may be deleted or moved to another folder. You may choose to leave them without affecting the installation of the new version of Star-Chrom.

Backup Option [recommended for RS232 Non-demo Versions]

During the installation process, the install program you will be asked verify that you want to backup files and to name a folder where to save backup files. Normally Install will move all files with the same name, but which have different date/times as those being installed, to a backup folder. This includes all the Startup, solvent, column, and configuration files you may have updated with your own parameters. After installation you may rename these files or replace the newly installed equivalent files after you have reviewed the new Star-Chrom program initial parameters. Old method files should be copied to another directory as a further safety backup.

No action is required if there was no previously installed Star-Chrom program.

Installing the Star-Chrom CD

Before starting, identify the drive names/letters of the hard disk drive, the CD drive and the 3-1/2" diskette drive. Put the CD into the CD drive. Normally Windows will automatically start the CD. The Install StarChrom CD dialog box will open.

If Windows is configured so that the CD does not automatically start, use one of the following methods to open the Install dialog box:

Click on Start...Run and use the Browse to select the CD drive. Select CheckStarChromInstalled.exe to open.

Double click on the My Computer icon, click on the CD drive and the double click on the CheckStarChromInstalled.exe file.

Open Windows Explorer to go to the CD drive, then open (double click on) the file named CheckStarChromInstalled.exe.

Install StarChrom CD Dialog Box:

When the Install StarChrom CD dialog box opens, there will be six option buttons:

Quick Guide Manual, Calibration Manual, HPLC Control Manual, Install/Re-Install, Install Acrobat Reader, Exit

Select the Install option (Re-Install if a StarChrom folder exists). Follow the steps using the guidance below.

StarChrom folder: During the installation process, you will be asked where to locate Star-Chrom. We recommend that it be installed in C:\StarChrom. If it is necessary to locate it in another folder or a drive letter other than C:, you may use the browse function to change to the drive and folder you desire. However, you will have to edit the DStarLCMicro.ini file (see below in the section titled "Installing the Star-Chrom Diskette Files"). In addition, after opening them for the first time, save the solvents, columns and method files to ensure the new Star-Chrom location is recorded for these Startup files. Data Logging may also need to be adjusted in a similar manner.

The backup option (as described above) is recommended.

After the Star-Chrom installation is completed, the Install dialog box will reappear.

Manuals: Installation saves the Star-Chrom manuals into a folder called manuals. The manuals are in .pdf format. If you do not have Acrobat Reader 4.0 installed, you will need it to view the manuals. Earlier versions of the Reader may not work well as 4.0 for displaying graphics. Install the Version 4.0 by pressing on the Install Acrobat Reader button.

The manuals are entitled:

Quick Guide to Set-Up and Start-Up

Guide to Using Star-Chrom HPLC Management Software

Guide to using Star-Chrom's Calibration Module

If you desire to review the manuals before you exit the *Install* dialog box, click on any of the manual buttons. We recommend that you review the *Quick Start Guide* immediately.

After the CD is removed, you may access the manuals from:

The Desktop: Click on Start...Programs...StarChrom then any of the manuals;

Windows Explorer: StarChrom FOLDER...manuals FOLDER... manual .pdf files;

Desktop Shortcut: Create shortcuts from each of the .pdf files to put on the Desktop; or

CD: Reinsert the CD. Follow your original steps to find the CD and to open the Install CD dialog box. Press the buttons to access the manuals.

Installing the Star-Chrom Diskette Files

Locate the 3.5" Diskette sent with the Star-Chrom CD. This disk contains a file which needs to be loaded into the folder where you designated Star-Chrom to be installed (e.g. C:\StarChrom). This file is:

DStarLCMicro.ini: An updated version of the Star-Chrom configuration file that contains settings specific for your instrument system.

Insert the diskette into your computer's 3.5" drive and then using Windows Explorer, copy the two files to the Star-Chrom folder.

Regional Settings

For applications (countries) using the “ , “ (comma) in place of the “ . “ (period) decimal point, it will be necessary to change the decimal point parameter from comma to period in the Regional...Numbers box. The regional options are found in the Windows Control panel which may be accessed via the Start...Settings menu.

Troubleshooting Control Via RS-232

Your system is controlled via an RS-232 (Serial) port from the computer. There are usually two serial ports on most computers. These are DB-9 connectors. Older computers may have DB-25 connectors and require an adapter (not supplied). Your Star-Chrom is configured when initially installed to use the COM 1 serial communications port. Sometimes computers use COM 1 for some other purpose. For example, a mouse may be connected to COM 1.

If this is the case then you will need to change the DStarLC.ini file using either the Notepad or Wordpad file editor (supplied with Windows).

Find the following line in the .ini file:

1 is the comm port for hardware control

Change it to read:

2 is the comm port for hardware control

Note: When a USB to serial port adapter is used to provide an RS-232 serial port on a computer that does not come with one, the COM port assignment may turn out to be another number such as COM 3 or higher. Use the Windows "Start ... Settings ... Control Panel ... System ... Device Manager ... Ports" technique to determine the port number assigned to the adapter.

Save the file and close the editing program.

Close the Star-Chrom program and re-open it. Go to the Hardware page and make sure that your change is seen there.

Instructions on how to correct the wavelength display

NOTE: If the main detector (detector 1) is a fixed wavelength detector skip this page.

If the “measured wavelength” on the “General Conditions” page is not the same (+/- 2nm) as the wavelength on the front panel of the instrument, continue:

On the “Scales & Zeros” page of Star-Chrom make sure that the value entered for “D-Star detector zero” is 0.

Go to “General” conditions page.

From the front panel of D-Star detector (detector 1 only), change the wavelength display to read 000 nm using the detector’s up/down arrow keys.

On the “General” conditions page read the measured wavelength.

Enter this new value on the “Scales & Zeros” page in the “D-star detector zero” box.

Press the “**Click here to read...**” button at the bottom of the page.

Save the ini file by using the “INI File” menu item.

Select the “Save File” option.

Go to “General” page and enter the desired wavelength. Press **enter** on the keyboard. The detector wavelength should gradually change to the wavelength to within 2 nm

Instructions on how to turn off wavelength control for fixed wavelength detectors

You will need to change the DStarLC.ini file using Notepad, Wordpad or another text editor.

Open the “DStarLCmicro.ini” file .

Find the following lines in the .ini file:

y use dstar detector

Change it to

n use dstar detector

Save the changed file.

This will change the “General...” page in the Star-Chrom software and will prevent the software from trying to control the wavelength which is only possible if the D-Star variable wavelength detector is used as detector 1.

Instructions on how to zero and scale the pressure reading

Steps to set Pressure Scale and Pressure Zero

Pressure Scale and Zero settings for the D-Star pressure transducer in the HPLC system shipped with this instruction/CD are already entered in the DStarLCMicro.ini file on the 3-1/2" diskette. You should have copied this file to the StarChrom folder on the hard drive during the Install process. Use the following procedure to fine tune the settings or change the scale factor. It is also prudent to check the pressure zero periodically, and to occasionally compare the transducer against a calibrated gauge.

Setting the Pressure Zero (Zero Offset)

=On the Scales & Zeros page, enter 1.000 into the pressure scale - applied to readings box and the =0.000 into the pressure zero - applied to readings box. Press the "Click here to read..." button.

Check the Direct Mode Bar (yellow printing on the green space above the page tabs) reading of pressure (Press=xx) and enter that amount into the pressure zero box and press the "Click here to read... button". Usually the zero offset is 197 - 200.

Adjust the zero offset until the display centers/averages around "0" [for example the pressure may fluctuate from -5 to 4].

Setting Pressure Scale Factors for PSI (Pounds per Square Inch) Units

=Enter 7.46 into the "pressure scale - applied to readings" box and press the "Click here to read..." button.

The Direct Mode Bar display should read higher than a 1.000 Scale factor when pressure is applied.

Ensure that the maximum pressure reading for pressure transducer on the Hardware page is 7500.

Press the "Click here to read... button". This sets the % scale factor for PSI pressure on the Plot 2 page.

Setting Pressure Scale Factors for BAR Units

=Enter .508 into the pressure scale - applied to readings box and press the "Click here to read..." button.

The Direct Mode Bar display should read lower than the PSI or 1.000 Scale when pressure is applied.

Ensure that the maximum pressure reading for pressure transducer on the Hardware page is 510. Press the "Click here to read... button". This sets the % scale factor for BAR pressure on the Plot 2 page.

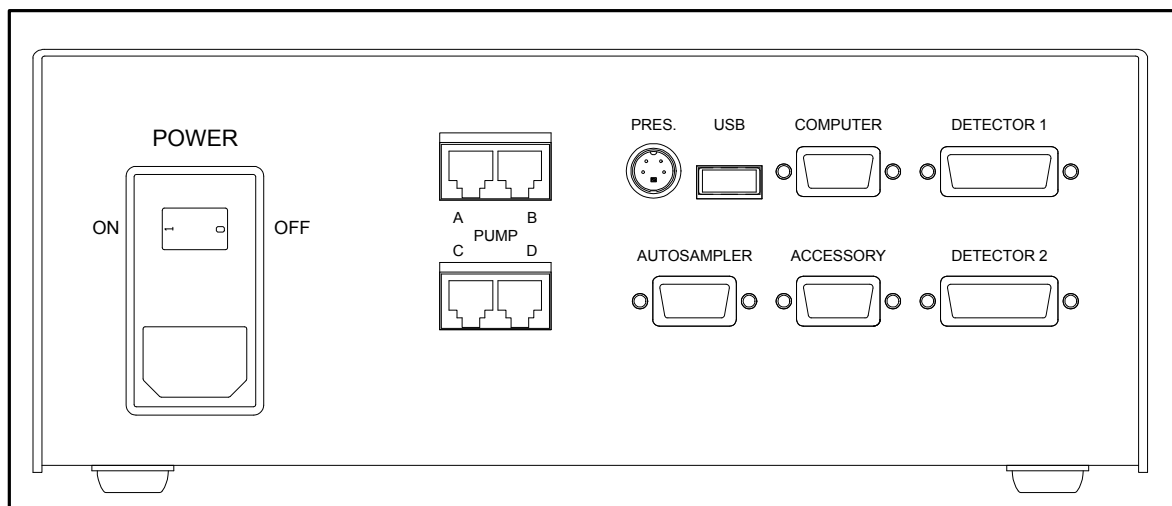
REMEMBER to save the new parameters in the .ini file [Ini File menu]

Pressure Alarms

The Pump Pressure Alarm Limits on the Alarms page may be set in PSI or BAR depending on which of these scale factors used.

NOTE: For OTHER pressure transducers, a scale factor must be computed using a calibrated gauge.

Rear Panel of the Star-Chrom Interface Box



Power Input

Corcom power receptacle

Accepts detachable IEC cords

Pump A & B

(Isocratic & Binary versions)

6 pin RJ-11 Receptacle

Pin # Description

1 ground
2 signal
3 signal
6 ground

Pump C & D

(Quaternary versions)

6 pin RJ-11 Receptacle

Pin # Description

1 ground
2 signal
3 signal
6 ground

Pressure

5 pin HEX Female Receptacle

Pin # Description
B signal +
E signal -

USB

(not on this version)

Type A USB Receptacle

Pin # Description

1 +5V
2 DM
3 DP
4 ground

AutoSampler

DB-9 Male Receptacle

Pin # Description

1 start_data (in)
2 digital ground

5 start_inj (out)
6 digital ground
7 RS-232 Rx
8 RS-232 Tx
9 RS-232 ground

Computer

DB-9 Female Receptacle

Pin # Description

1-9 RS-232 connections to COM or Serial I/O port on computer.

Accessory

DB-9 Female Receptacle

Pin # Description

3 recycle +12VDC*
4 recycle contact closure to ground

Detector 1

DB-15 Female Receptacle

Pin # Description

9 signal +
3 wavelength +
8 autozero
6 wavelength up
12 analog ground
14 wavelength down
2 digital ground
10 event / inject

Detector 2

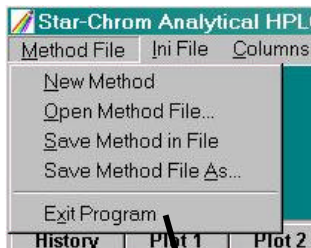
DB-15 Female Receptacle

Pin # Description

9 signal +
8 autozero
12 analog ground
2 digital ground

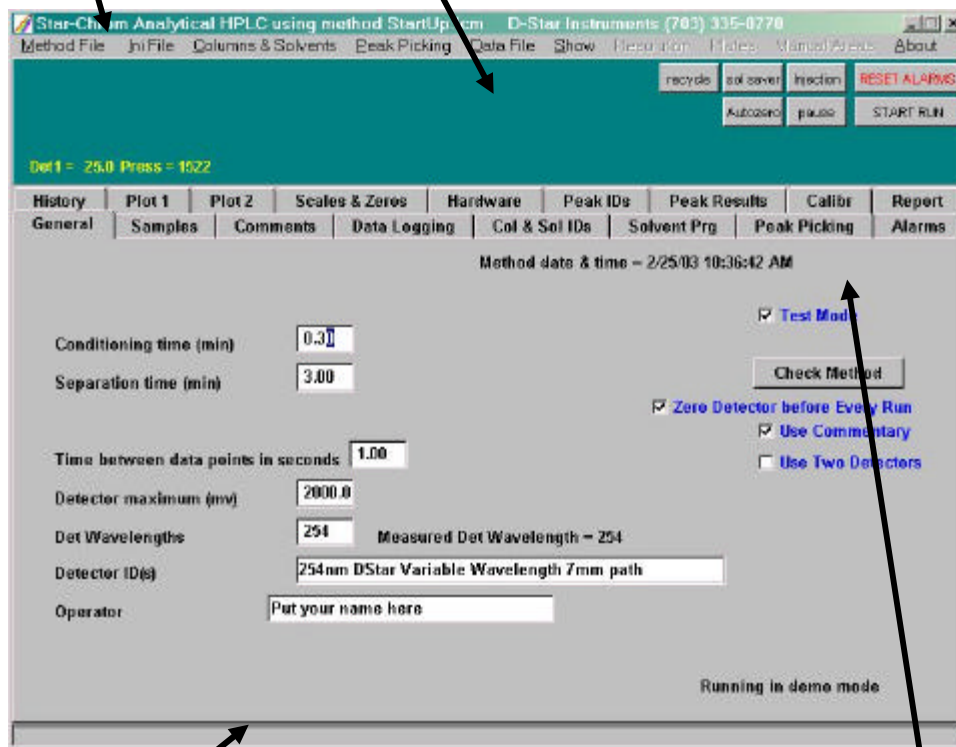
* Maximum recycle valve current is 300mA (40 ohm minimum solenoid resistance), it is switched by software, and is normally open.

Map and Description of Screen.



The **Menu bar** will reveal drop down lists when the cursor is placed on the menu name and the left mouse button is clicked.

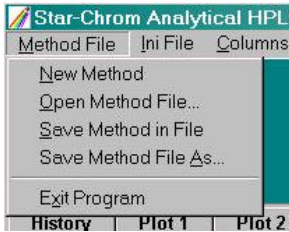
The **Direct Mode bar** displays current information such as the signal from the detector, pump pressure etc. Also, there are buttons which start/stop or pause the run, start data acquisition (Injection), turn on/off the solvent recycler and reset the alarms.



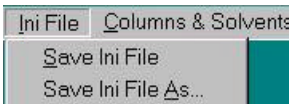
Hints line. This line will give hints based on where the cursor is placed. If this line is not visible because of the Windows "Task bar" you can change the task bar properties to "Auto hide and Remain on top". This is done by putting the cursor on the Task Bar and clicking the right mouse button and selecting "Properties".

The bottom half of the screen contains a series of **Tabbed Pages**. When the cursor is placed on the desired tab and the left mouse button is clicked the desired page comes to the front.

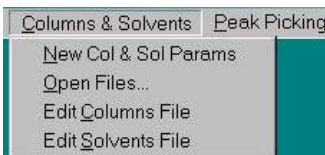
Menu Items



Method files contain information such as the gradient program and flow rate as well as the information on the "General" page. The file "StartUp.lcm" is automatically loaded when the program opens.



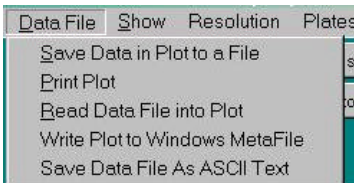
The "ini" file saves information about the hardware and is used when the program opens to initialize the hardware parameters.



These files are used for documentation of the run conditions. You should make a list of the columns you use as well as the solvents. The files "Columns.clm" and "Solvents.slv" are the default files.



The peak picking files store peak picking parameters. You may find that you will have different parameters for different conditions. The file "PeakPickStartup.val" is the default.



This menu lets you save the current plot including the method to a data file. It also lets you read in a previous data file. Additionally you can export the plot as a ".wmf" Windows Metafile image file which can be inserted into a word processor document. You can also export the data to an ASCII text file which can be read by many other programs.



The “Show” menu item has several interesting options. The “Show Chromatogram in Small Plot” will let you show a previous run superimposed on the current plot. You need to read in the previous data file in order to see the plot.



This item lets you calculate the resolution between pairs of peaks. You can enter the value for Tzero or use an unretained peak as the void volume marker.

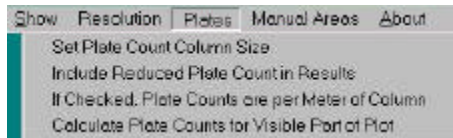


Plate counts are important to evaluate the “goodness” of the column. The results can be reported in plates per column or plates per meter. Reduced plate counts is useful to compare columns of different size particles.



This item lets you calculate the areas of the peaks using a manual method. The results can either replace the current “peak Results” table or can be added to results that are already there.



The “About” box show the current version number.

Important Note: Starting up the system

The correct order for starting up the system is as follows:

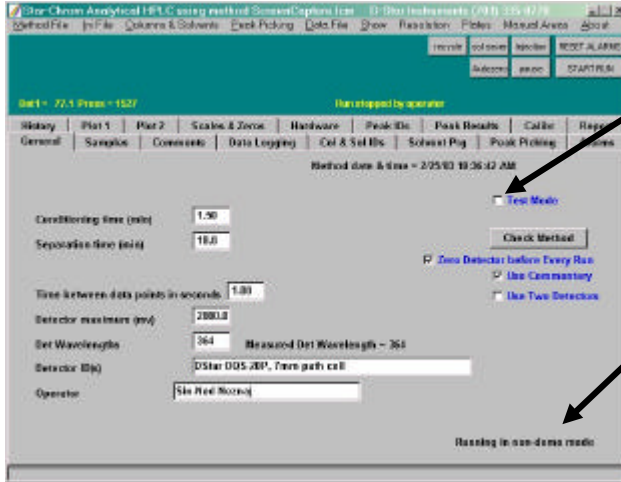
1. Turn on the Pump/Detector (HPLC).
2. Plug in the Star-Chrom interface box and turn on the power switch. You will see a pump flowrate value of 0.02. This indicates that the interface box is on and is communicating with the pump.
3. Start the Star-Chrom program. In the lower right corner of the screen you should see “Running in non-demo mode”. It may take a minute for the software to connect to the box.

If the interface box is in “Demo mode” the software will not control the HPLC. You should make sure the interface box is powered (see #2) then make sure the RS-232 serial cable is connected.

4. If you disconnect the power to the interface box you will need to restart the Star-Chrom software.

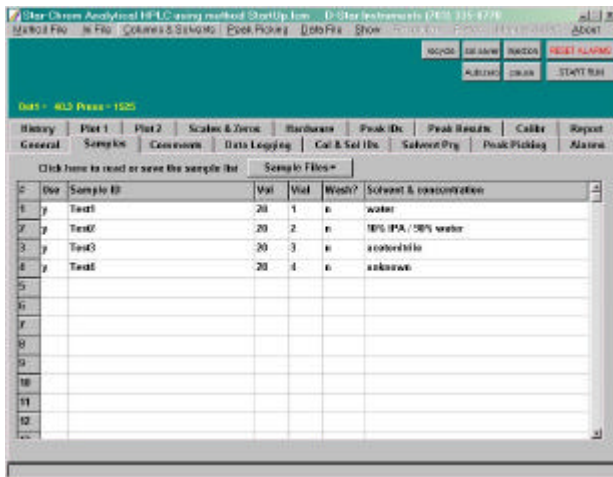
OK, you have plugged everything in and turned everything on. You have connected a column and have primed the pump with your mobile phase. Now you want to start a run. You still need to do a few things.

1. Fill in the “General ” page with the “separation time” etc. Make sure it is not in “Test Mode” and that it is “running in non-demo mode”.

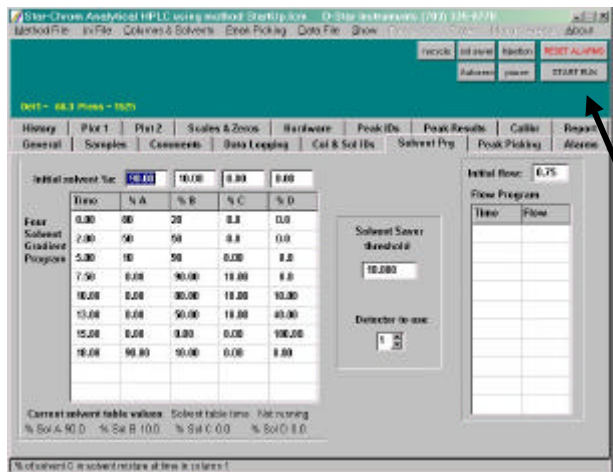


Test Mode box (shown ON) should be unchecked.

Message should say “Running in non-demo mode” (shown “Running in demo mode”)*



2. Fill in the “Sample” page. The “sample id” is used as part of the file name so be descriptive. This will make it easier to identify the file later.

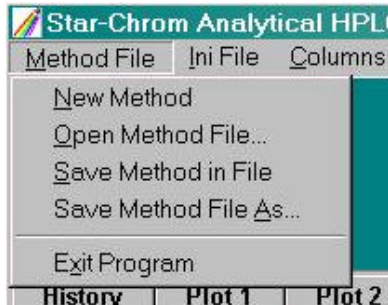
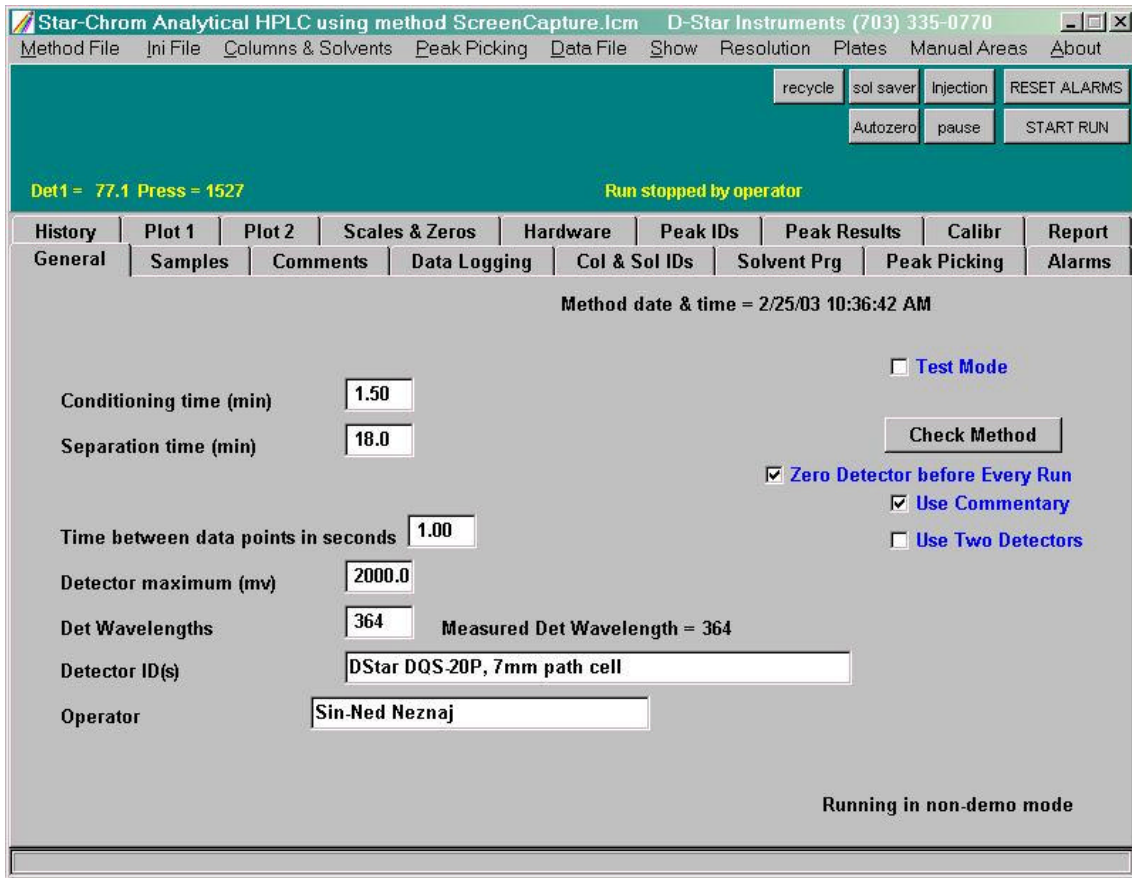


3. Fill in the “Solvent Prg” page. Enter the initial solvent composition and flow rate.

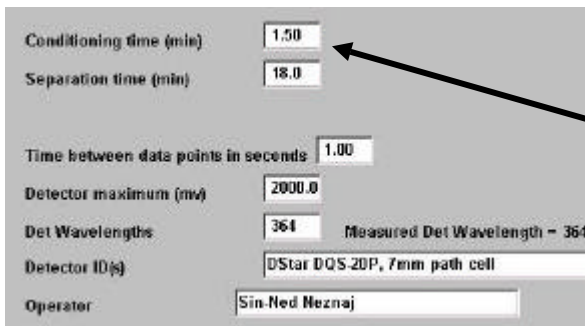
4. Press the “Start Run” button.
5. Inject the sample when prompted.

* **Note:** If the program shows that it is in the demo mode there is no communication with the interface box. **Check cables and power.**

General Page



When you start the Star-Chrom program this screen will appear. The method called “startup.lcm” is automatically loaded. You can change any value in any of the data boxes. You make changes in any data box by highlighting the value with the cursor and typing in the new value. Save the method using the “Method File” menu item and you can save it to the same or different method name.



Remember that there are hints at the bottom of the window. For example if the mouse cursor is in the box after “Conditioning time” then the hint at the bottom is “The column will be conditioned for this time before the injection.”

Samples Page

Star-Chrom Analytical HPLC using method StartUp.lcm D-Star Instruments (703) 335-0770

Method File | Ini File | Columns & Solvents | Peak Picking | Data File | Show | Resolution | Plates | Manual Areas | About

recycle | sol saver | Injection | RESET ALARMS
Autozero | pause | START RUN

Det1 = 40.3 Press = 1525

History | Plot 1 | Plot 2 | Scales & Zeros | Hardware | Peak IDs | Peak Results | Calibr | Report
General | Samples | Comments | Data Logging | Col & Sol IDs | Solvent Prg | Peak Picking | Alarms

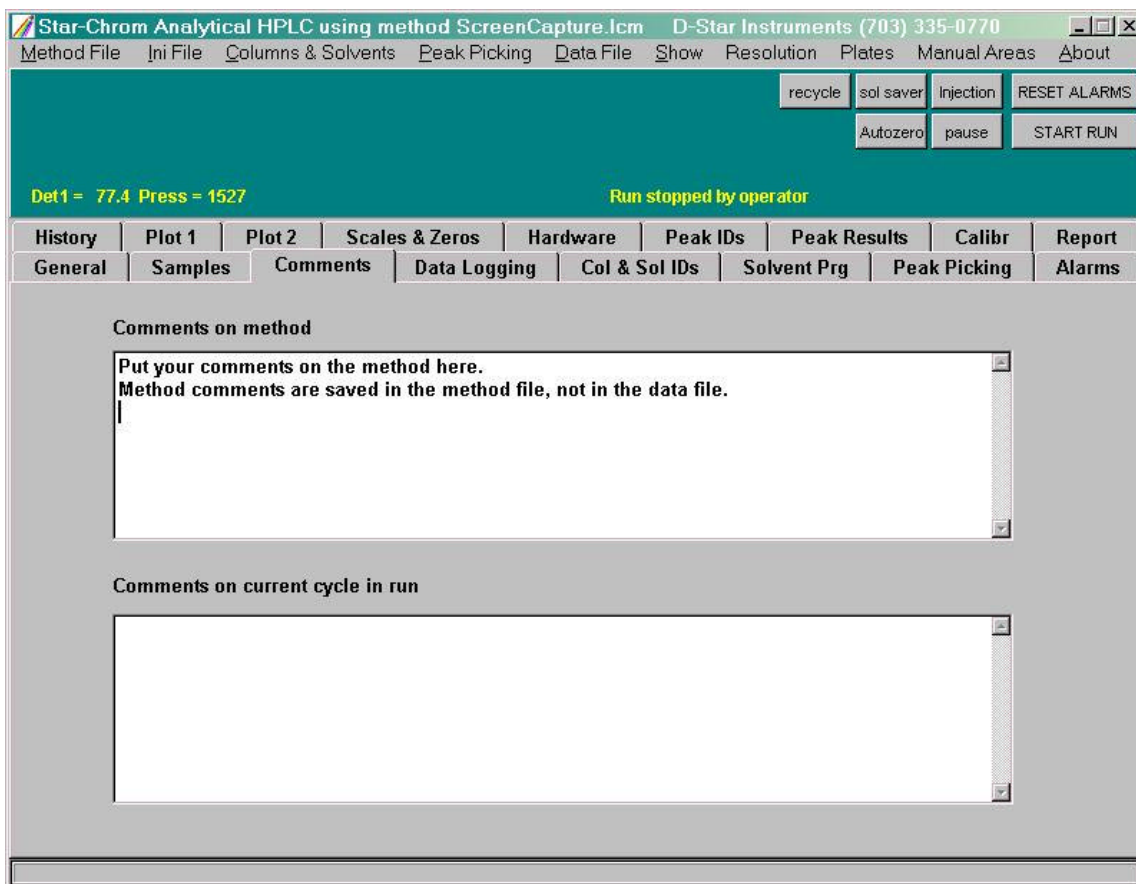
Click here to read or save the sample list | Sample Files ▾

#	Use	Sample ID	Vol	Vial	Wash?	Solvent & concentration
1	y	Test1	20	1	n	water
2	y	Test2	20	2	n	10% IPA / 90% water
3	y	Test3	20	3	n	acetonitrile
4	y	Test4	20	4	n	unknown
5						
6						
7						
8						
9						
10						
11						
12						

You must fill in information in the “Sample ID” column. When you press the “sample files” button you can open a previously saved sample description. The “Sample ID” is used in the creation of the file name and long file names are used so be descriptive.

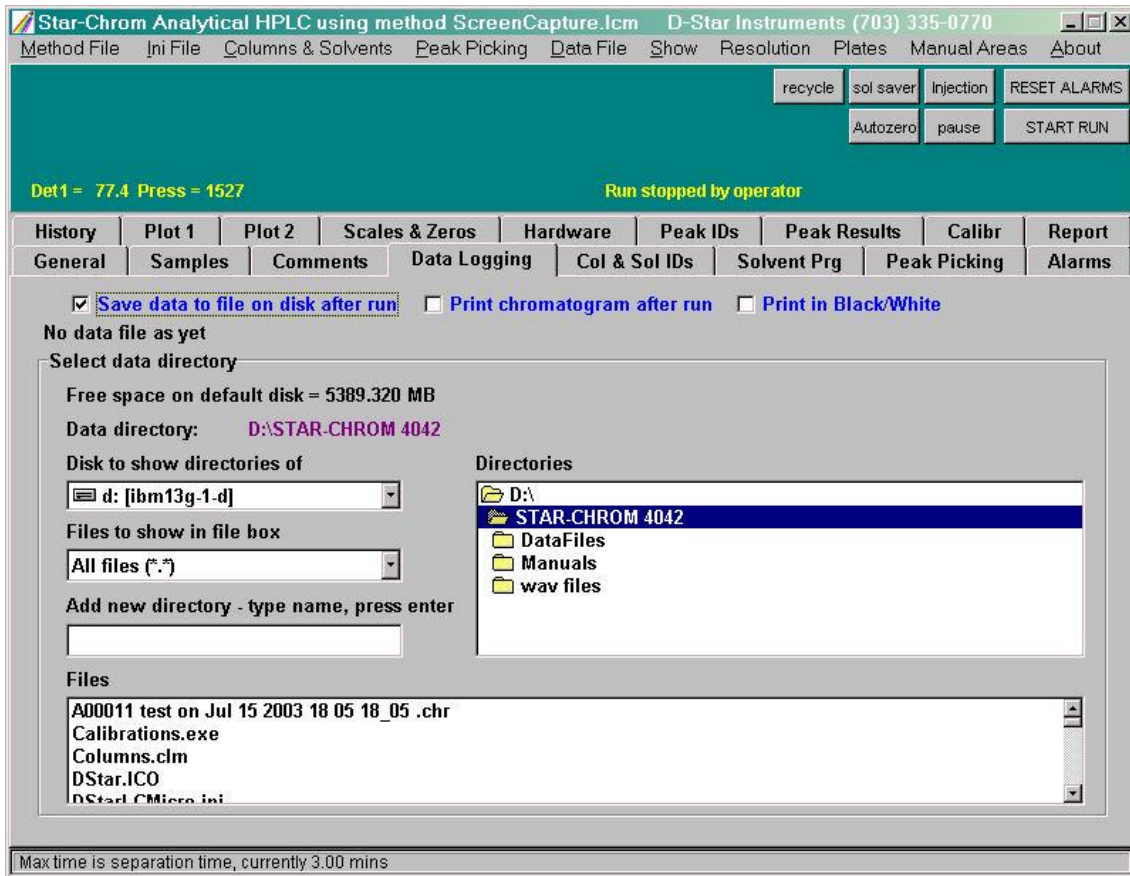
The “Use” box is used to identify which samples are going to be injected. If you use an autosampler and you need to re-inject one of the samples in the middle just enter “n” in the samples that will not be injected.

Comments Page

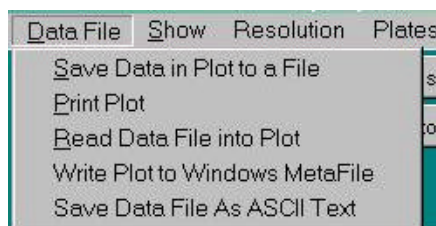


You may place comments that describe the method and/or current run. The method comments are saved with the method file (when saved) and the run comments are automatically saved at the end of the current run with the data file.

Data Logging Page



You need to select and create a directory where your data file will be saved. Make sure that the line titled “Data Directory:” has the proper path where you want the data stored. You open the directory by double clicking on the file folder. Check the “Data Directory:” line to make sure the correct directory is opened. This info is not saved with the method file. The default directory is the directory where “StarChrom.exe is stored. Therefore if you forget to select a directory your data will be stored in the default directory. You can use Windows Explorer to create directories and move files.



You may also save data at the end of a run and before starting a new run using the “Data File” menu item.

You can look at previously run data by selecting “Read Data into Plot”. You can also save data as an ASCII text file for importing into other programs such as a spreadsheet program. You may save the plot as a .wmf image file which can be used in a word processor document.

Col & Sol IDs Page

Star-Chrom Analytical HPLC using method ScreenCapture.Icm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver injection RESET ALARMS
Autozero pause START RUN

Det1 = 77.5 Press = 1527 Run stopped by operator

History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	

column 1 Nucleosil C18 5u 4.6mm x 25cm

Dead volume of column 1 = 3.2

Solvent Bottle A Water

Solvent Bottle B 90/10 IPA/H2O

Solvent Bottle C Methanol

Solvent Bottle D Acetonitrile

This page is used for documentation of column and solvent information. The button at the end of the description box will open a list of available options. You will need to create your own column & solvent lists using the "Column & Solvents" menu. Then select "Edit Columns File" or "Edit Solvents File"

Columns & Solvents Peak Picking

- New Col & Sol Params
- Open Files...
- Edit Columns File
- Edit Solvents File

Solvent Prg Page

Star-Chrom Analytical HPLC using method StartUp.lcm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver injection RESET ALARMS

Autozero pause START RUN

Det1 = 68.3 Press = 1525

History Plot 1 Plot 2 Scales & Zeros Hardware Peak IDs Peak Results Calibr Report

General Samples Comments Data Logging Col & Sol IDs Solvent Prg Peak Picking Alarms

Initial solvent %s:

Four Solvent Gradient Program

Time	% A	% B	% C	% D
0.00	80	20	0.0	0.0
2.00	50	50	0.0	0.0
5.00	10	90	0.00	0.0
7.50	0.00	90.00	10.00	0.0
10.00	0.00	80.00	10.00	10.00
13.00	0.00	50.00	10.00	40.00
15.00	0.00	0.00	0.00	100.00
18.00	90.00	10.00	0.00	0.00

Solvent Saver threshold:

Detector to use:

Initial flow:

Flow Program

Time	Flow

Current solvent table values Solvent table time Not running
 % Sol A 90.0 % Sol B 10.0 % Sol C 0.0 % Sol D 0.0

% of solvent C in solvent mixture at time in column 1

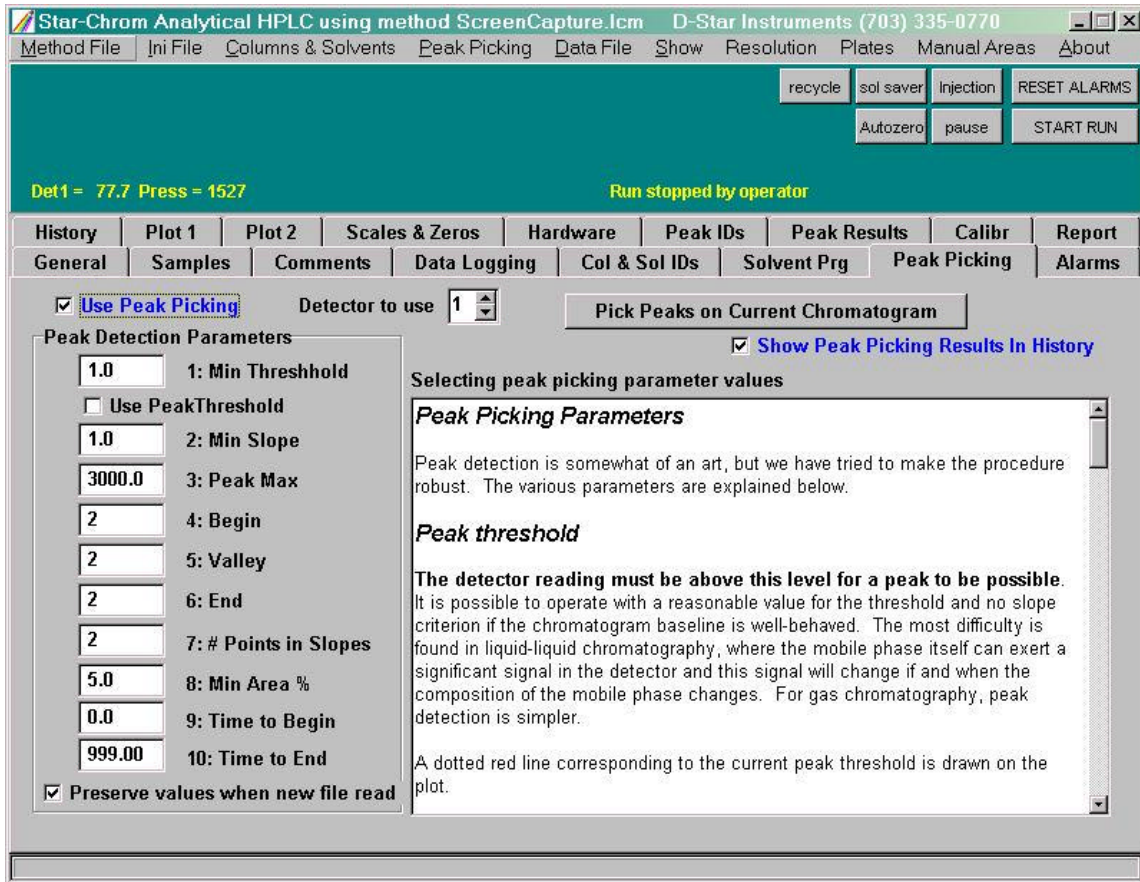
The “Solvent Prg” page allows you to enter a gradient profile, a flow rate and flow program.

A recycle valve (12 VDC three-way) can be installed at the output of the flow cell. The valve is connected to the interface box expansion port as seen in the previous page describing the back panel of the interface box. This valve will allow you to set a threshold +/- value around zero. The solvent would be recycled if the detector signal is between the +/- of the value in the “Solvent Saver threshold” box.

The initial solvent composition (if it is a gradient system) and flow are set on this page. During the run if you want to change conditions you would change the values in the initial boxes. A change in the flow occurs immediately. A change in solvent composition occurs when you leave this page.

Linear gradients segments are programmed by entering the desired composition and at what time in the run you want that composition.

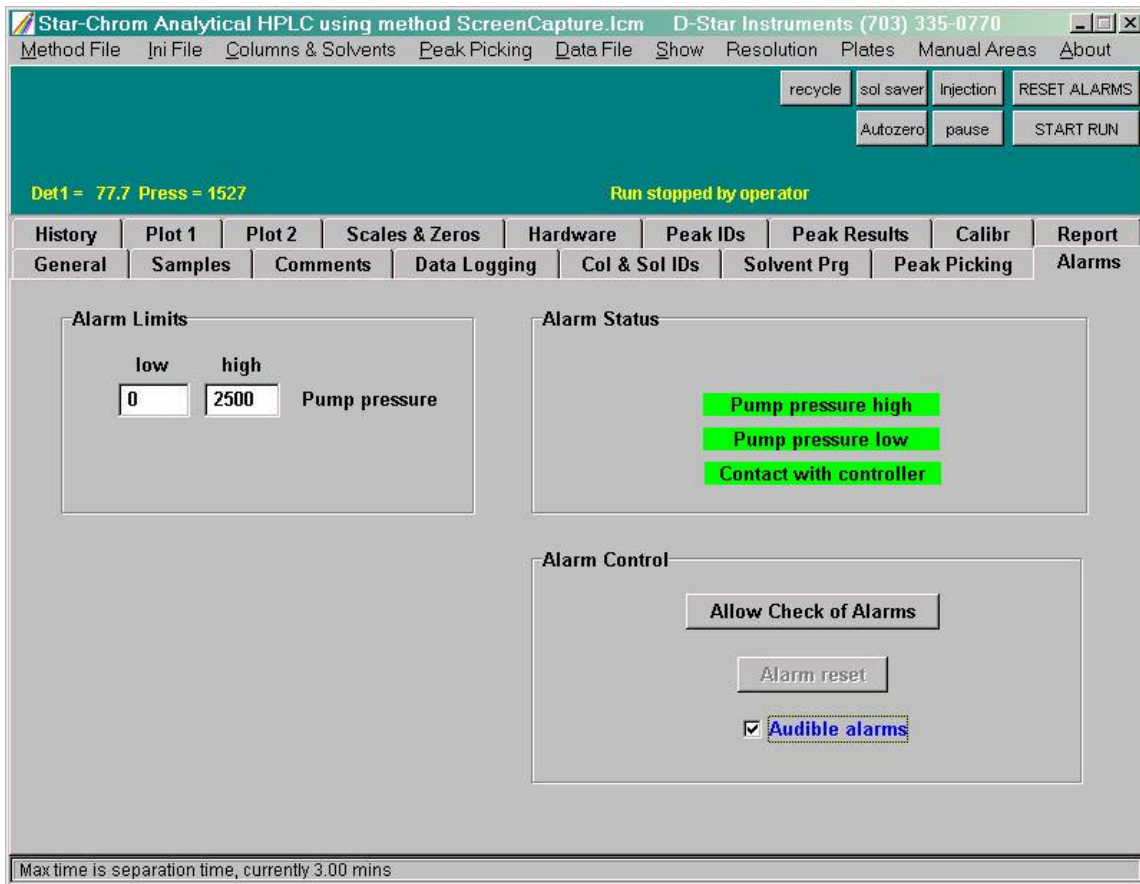
Peak Picking Page



These “Peak Detection Parameters” are used to integrate the peak areas. They affect the data found on the “Peak Results” page. When you press the button labeled “Pick Peaks on Current Chromatogram” the chromatogram will be re-integrated using the current parameters. If this button is grayed out then there is no chromatogram in the plot window. Use the “Data File” menu item and select “Read Data File into Plot” to load a previous run.

The “Use peak picking” box must be checked

Alarms Page



If a pressure signal is available you can set the high / low alarm values. You can defeat the alarm by pressing the “Alarm Reset” button and then you do not press the “Allow Check of Alarms” button. If the “Reset Alarms” button is flashing red and blue during the run then the alarms are not active. Go to the “Alarms” page and press the “Allow check of alarms” button. The “Reset Alarms” button should stop flashing.

When an alarm occurs this screen will be visible and the run will be paused. If you correct the problem and then press the “Alarm Reset” button the run will continue starting at the time the alarm occurred. Remember to re-activate the alarms by pressing the “Allow Check of Alarms” button.



History Page

Star-Chrom Analytical HPLC using method ScreenCapture.lcm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver Injection RESET ALARMS

Autozero pause START RUN

Det1 = 77.7 Press = 1527 Run stopped by operator

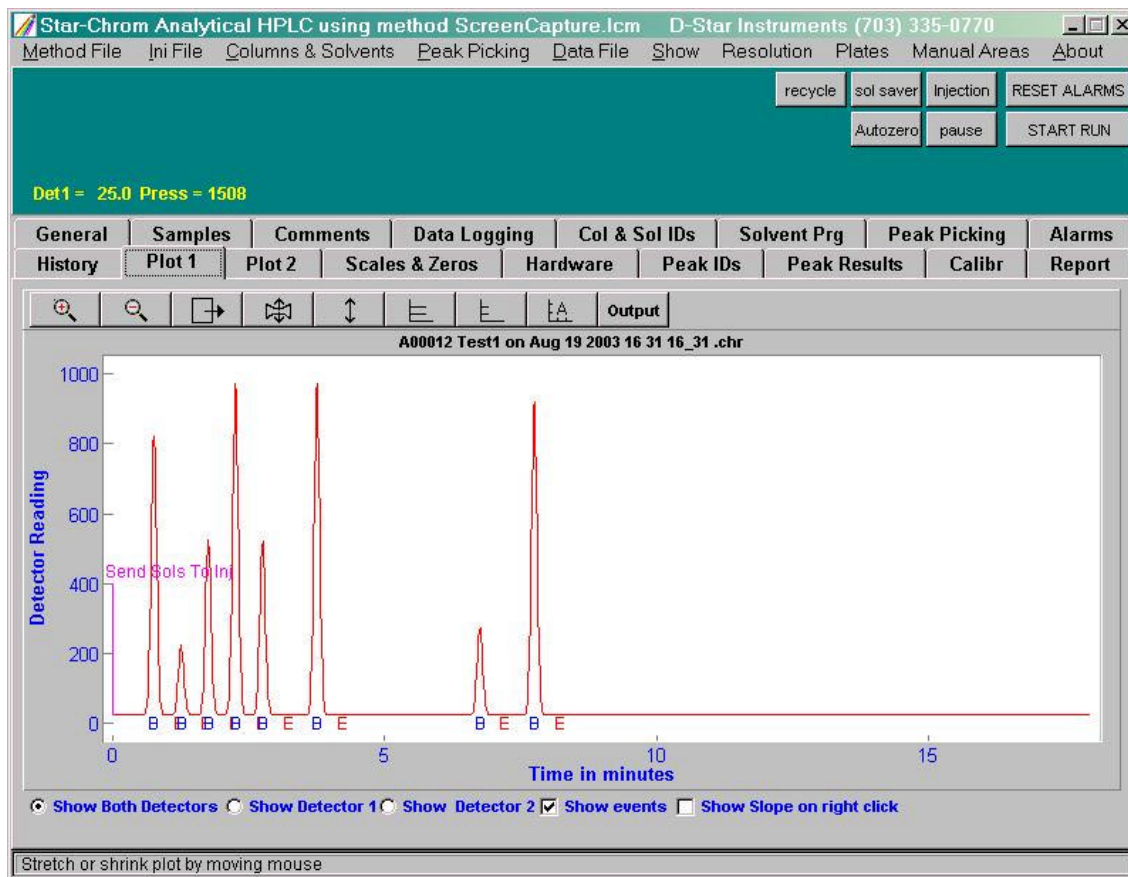
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms
History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr Report

Today's date = 08/19/03 Time of day = 16:10:21 Refresh History Print History

```
Run started 08/19/03 15:47:22
08/19/03 15:47:22 Sample ID is: Test1
08/19/03 15:47:22 Operator ID is: Put your name here
08/19/03 15:47:22 Volume injected: 20
08/19/03 15:47:22 Vial #: 1
08/19/03 15:47:22 Needle not washed after injection
08/19/03 15:47:22 Solvent, concentration: water
08/19/03 15:47:22 Detector ID: DStar Variable Wavelength, 7mm path
08/19/03 15:47:22 Detector wavelength is 364
08/19/03 15:47:22 Column 1 is Wazzatstoff C18 3um, 1.0 mm x 40 mm
08/19/03 15:47:22 Dead volume of column is 2.00 mLs
08/19/03 15:47:22 Starting programmed flow rate is 0.75
08/19/03 15:47:22 Contents of Sol Bottle A: Water
08/19/03 15:47:22 Contents of Sol Bottle B: 90/10 IPA/H2O
08/19/03 15:47:22 Contents of Sol Bottle C: Methanol
08/19/03 15:47:22 Contents of Sol Bottle D: Acetonitrile
08/19/03 15:47:22 Changed from Demo to non-Demo mode
08/19/03 15:47:24 Detector set to 364
08/19/03 15:47:26 New cycle
08/19/03 15:47:26 Equilibration begun at 0.00 minutes
```

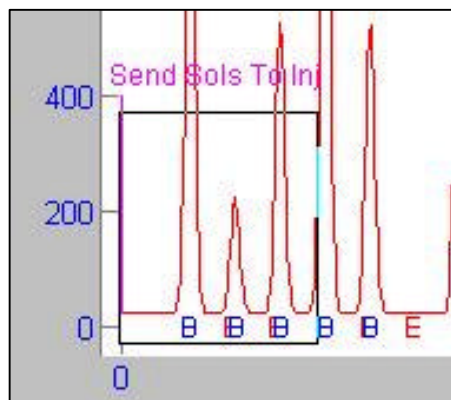
You can not enter anything on this page. This page shows a log of events and is saved with the data file.

Plot1 Page

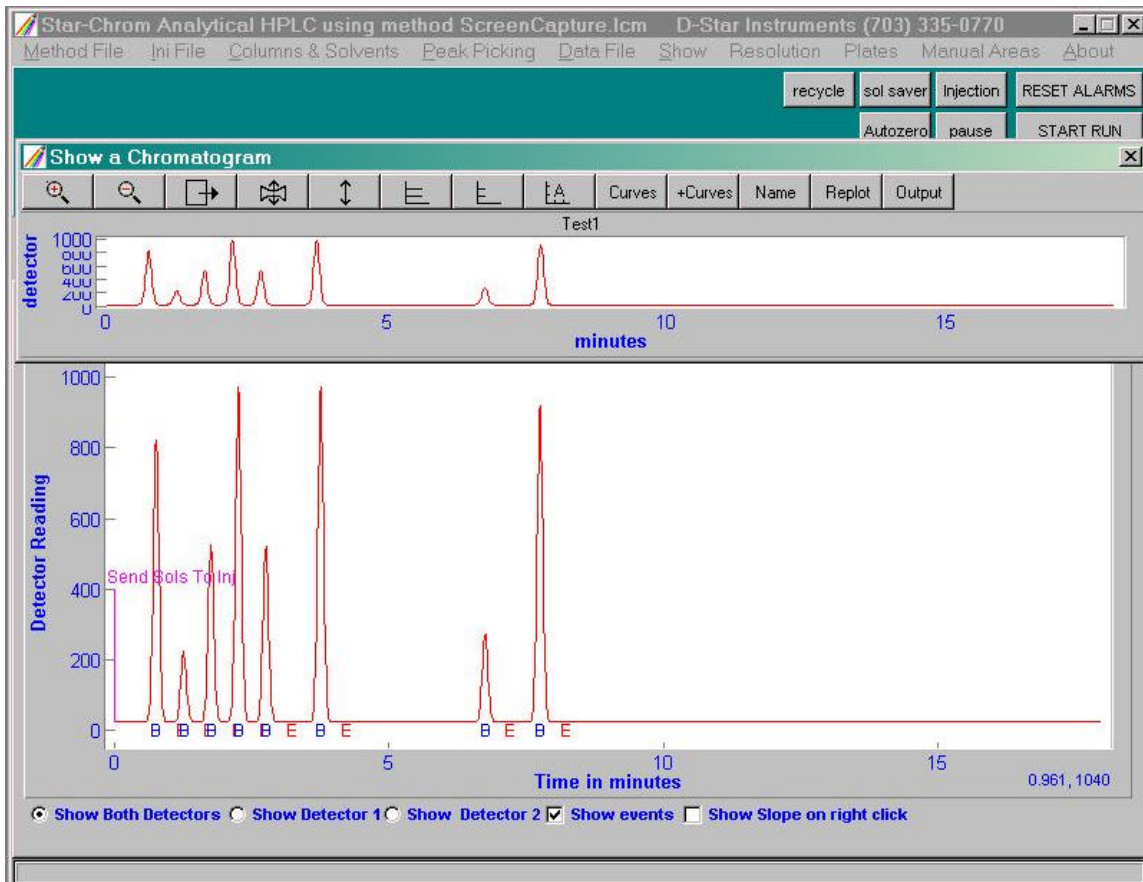


The "Plot 1" page shows the detector plot including any events. The plot can be zoomed in/out using the appropriate buttons. If you have integrated the chromatogram and have set up a calibration curve then the peaks will be identified. Use the button on the "Peak Picking" page to integrate.

To zoom the plot left click on the magnifying lens with the "+" in it. Put the cursor in the lower left corner of the area you want to enlarge. Press and hold the left mouse button. Drag the cursor to the upper right corner of the area to be enlarged and release the mouse button. The "-" button will unzoom the plot. The up/down arrow button will make the largest peak that is visible in the plot window fill the plot window.



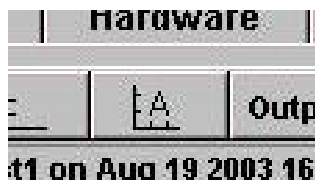
Small Plot (floating window)



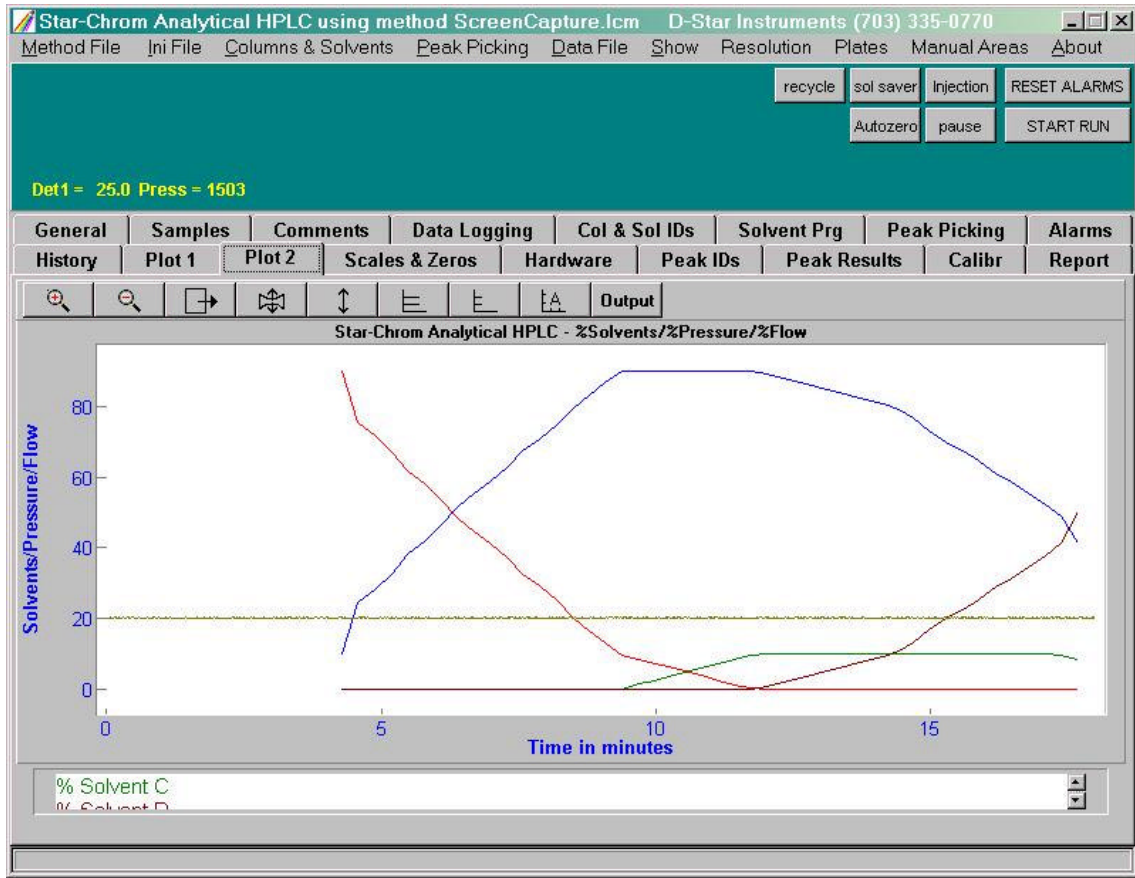
Using the “Show” menu you can read a previous chromatogram and display it on a small plot. First select “Show chromatogram in small plot” then “Read new file into small plot”.



Make sure to adjust the time axis if necessary using the button shown.



Plot2 Page



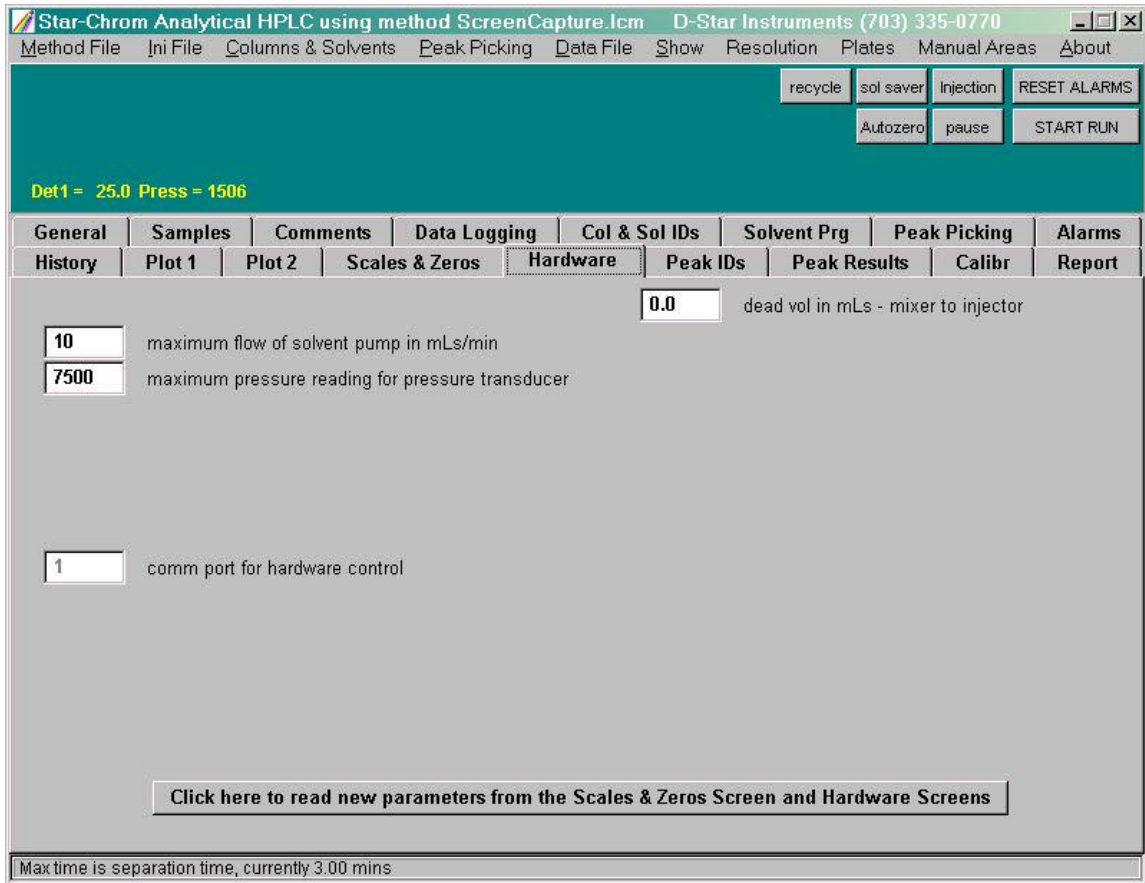
The "Plot 2" page shows the pressure plot as well as the programmed gradient that was run. The plot can be zoomed in/out using the appropriate buttons. The plots are shown in percent of full scale.

Scales & Zeros Page

You can change the scales and zeros using this page. If you make a change you must press the “Click here to read ...” button at the bottom of the page. If these new parameters check out to be OK. You then would open the “INI file” menu and select “save”.

NOTE: Change the zero first and press the “Click here...” button and verify that you have zeroed the value. Then you can apply the scale factor.

Hardware Page



The “Hardware” page has some information about the hardware being used. You should not change information on this page.

Peak IDs Page

Star-Chrom Analytical HPLC using method StartUp.lcm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver injection RESET ALARMS

Autozero pause START RUN

Det1 = 78.7 Press = 1525

Use y/n?	Begin	End	B/W	Class	Cmpd ID	Detector	Det number
y	2.24	2.60	B	Put class of compound	Uracil	Var UV 7mm path	254.0 nm
y	3.26	3.66	B	Put class of compound	Phenol	Var UV 7mm path	254.0 nm
y	4.39	4.90	B	Put class of compound	Toluene	Var UV 7mm path	254.0 nm

Currently selected calibration is: Q.C. Test Mix

Identify Peaks in Screen Plot Right Clicks on Plot Set Limits in Peak ID Table Auto Get Amounts at End of Run

Note: The peak-picking on this page needs to know where the baseline is and uses "zeros"

Reprocess Chromatograms in Plot Use Current DB To Transform Peak Area to Quantity

Peak ID Parameters ▾

Zeroing the Plot ▾

The "Peak ID's" page is used to identify peaks on the plot. Normally this information is transferred from the calibration database. However, you can enter the begin and end times for a window to look for a peak. Then enter a name in the "Cmpd ID" column. You can clear the information on this page using the "Peak ID Parameters" button.

The identification process occurs when the "Pick peaks..." button on the "Peak Picking" page is pressed. There are times when you want to integrate everything in a particular time window the "Reprocess Chromatogram plot" button and "Zeroing the Plot" buttons are used for this. Otherwise the peak area is based on the peak identified using the "Peak Picking" parameters.

Peak Results Page

Star-Chrom Analytical HPLC using method StartUp.lcm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver injection RESET ALARMS

Autozero pause START RUN

Det1 = 78.7 Press = 1525

General Samples Comments Data Logging Col & Sol IDs Solvent Prg Peak Picking Alarms

History Plot 1 Plot 2 Scales & Zeros Hardware Peak IDs Peak Results Calibr Report

Peak areas for the 4 biggest peaks of 37 total Peak Areas Plate Counts Peak Resolutions

Peak #	Begin	End	Peak Area	Maximum	Time of max	Area %	Begins as	Quantity	Name
1	1.36	1.64	1121	220.44	1.44	6.6	Valley		
2	2.25	2.67	4202	584.55	2.38	24.7	Valley	14.952	Uracil
3	3.22	3.94	5440	679.75	3.46	31.9	Valley	15.888	Phenol
4	4.41	5.25	5455	548.14	4.69	32.0	Valley	15.931	Toluene

Area of all peaks found = 17030.79, 95.22% kept as peaks above minimum area % of 5.00

Peak Options... ▼

The “Peak Results” page is where you will find the results of an integration performed on the current chromatogram. This integration occurs immediately after a run or when the “Pick Peaks..” button on “Pick Peaks” page is pressed. In addition you can view “Plate Counts” or “Peak Resolution” information by clicking on the appropriate radio button. The information that is displayed is calculated based on the parameters entered on the “Peak Picking” page.

NOTE: not all peak areas are visible in this table. The “minimum area %” on the “Peak picking” page will determine which areas are seen.

Plate counts and peak resolutions are calculated by using the appropriate menu item.

Show Resolution Plates Manual Areas About

Use First Peak as TZero

Set TZero

Calculate Resolutions of Pairs of Nearest Big Peaks

Show Resolution Plates Manual Areas About

Set Plate Count Column Size

Include Reduced Plate Count in Results

If Checked, Plate Counts are per Meter of Column

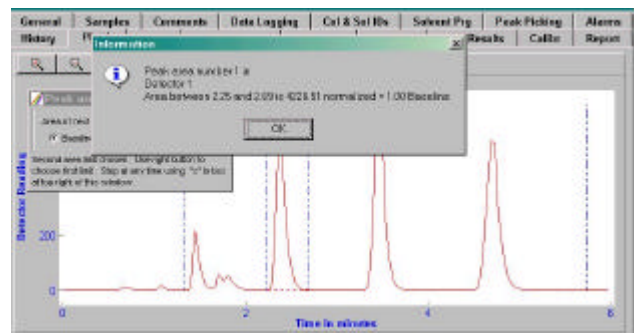
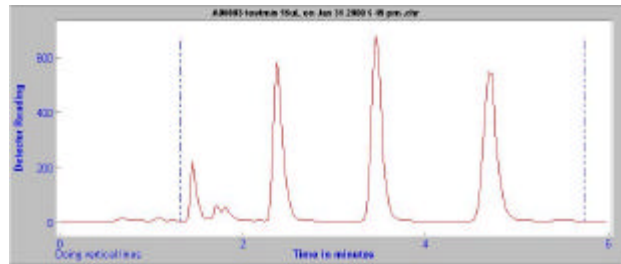
Calculate Plate Counts for Visible Part of Plot

You may want to manually integrate the plot. To do this you would use the menu item “Manual Areas”.

First you have to zero the baseline by selecting “Choose where to set the baseline to zero”. Then you will follow the directions and right click on the curve where the end points are.

You will see vertical lines indicating where you selected to zero the baseline. If the chromatogram is flat then you would only need to mark the begin and end. If you have a sloping baseline you will need to mark more zeroing points. When you are done, select “Make Chromatogram Zero ...” from the “manual areas” menu.

Select “Begin Peak Area Measurement” from the “Manual Area” menu. Right click on the curve where you want to begin and end an integration area. To finish select “End peak area..” The Results will replace the “Peak results” table or will be added to it depending on the item you selected in the “Manual Area” menu list.



Calibr Page

Star-Chrom Analytical HPLC using method StartUp.lcm D-Star Instruments (703) 335-0770

Method File Ini File Columns & Solvents Peak Picking Data File Show Resolution Plates Manual Areas About

recycle sol saver Injection RESET ALARMS

Autozero pause START RUN

Det1 = 79.0 Press = 1518

General Samples Comments Data Logging Col & Sol IDs Solvent Prg Peak Picking Alarms

History Plot 1 Plot 2 Scales & Zeros Hardware Peak IDs Peak Results Calibr Report

Calib DB table is D:\Star-Chrom 4042\Startupcalibrdb.dbf

Q.C. Test Mix

Calibration DB Files

Send Record to Peak IDs

Coeffs: 1.213584E-15 3.558265E-3 0.000000E+0
0.000000E+0 0.000000E+0 0.000000E+0

Offsets: 3446.42 12.50 Range: 1236.36 to 5479.15

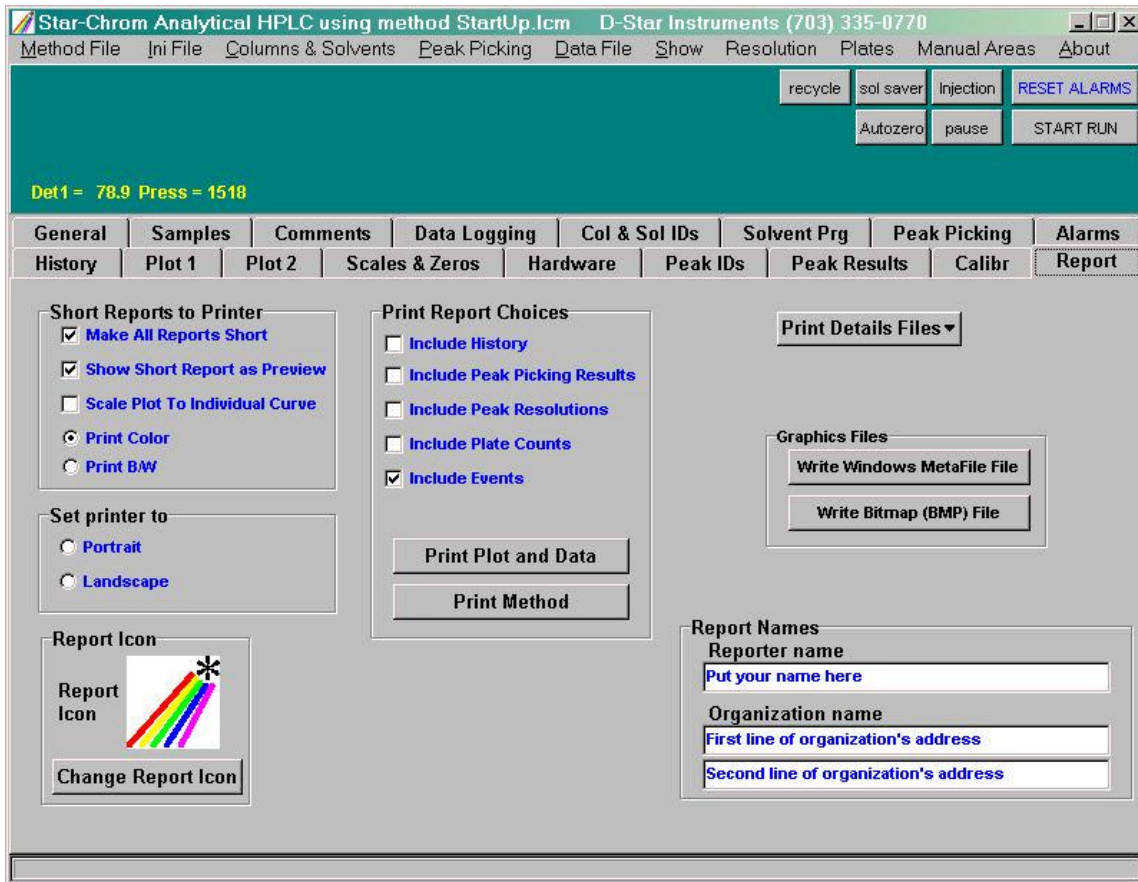
Sort on... Class, Cmpd ID, Detector, Detector number, Date Show All Records Filter records according to line last clicked in Peak ID Info table

Date, Class, Cmpd ID 01/30/00 18:37:17 Operator's name goes here

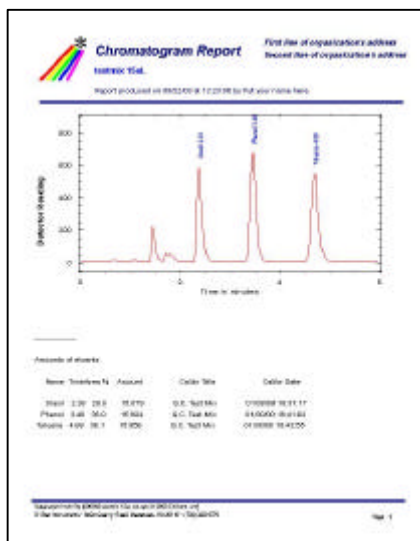
CLASS	COMPOUND	DETECT_ID	DET_NUM
Put class of compound here	Uracil	Var UV 7mm path	254.0 nm
Put class of compound here	Phenol	Var UV 7mm path	254.0 nm
Put class of compound here	Toluene	Var UV 7mm path	254.0 nm

The “Calibr” page is used to open a calibration database and then select the records for the peaks of interest that are to be quantified. The record of interest is selected by clicking on it and then press the button “Send Record to Peak IDs”. This will transfer the peak id info to the “Peak IDs” page.

Report Page



The “Report” page is used to set up the type of report that will be printed. The default is “short report” which shows a chromatogram at the top of the page, the operating conditions are below the chromatogram and any reports are printed below that. A preview of the report is shown before printing. Pressing the “Print Plot and Data” button will show you the preview.

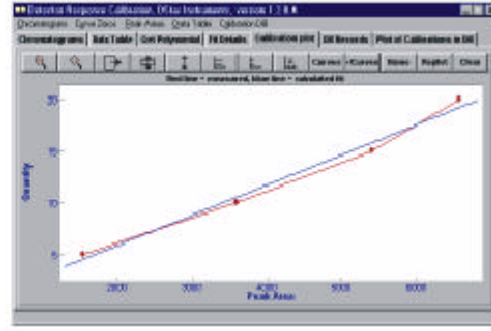


You can also save image files such as “.wmf” (Windows MetaFiles) or .bmp (bit-mapped) which can be used in a document that is created by a word processor program.

Note: you can scan your logo and save it as a .bmp file. This logo can be loaded and printed at the top left of your report. If you name your logo “ReportIcon.BMP” then it will load automatically.

If you uncheck “make all reports short” then print you will get a full page print of the plot.

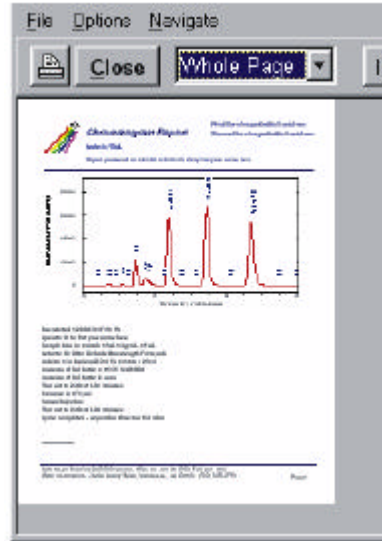
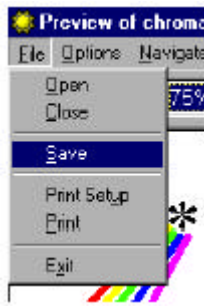
There are two other programs that are included with the Star-Chrom software. The “Calibrations.exe” program is



used to create a calibration curve and save it to a calibration database.

The other program is called “Viewer.exe” this is a freely distributable program that will allow you to send reports generated by the Star-Chrom program via e-mail. Once the recipient has the viewer program they can see and print the report sent.

The .ace file is created when you select save from the “file” menu.



Solvent Recycle Valve

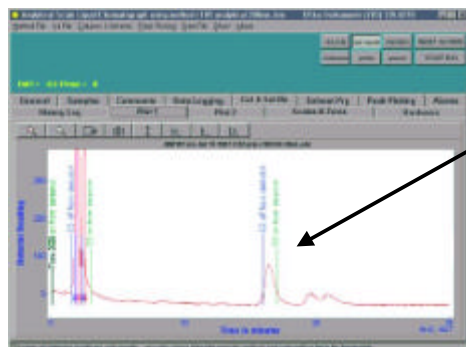
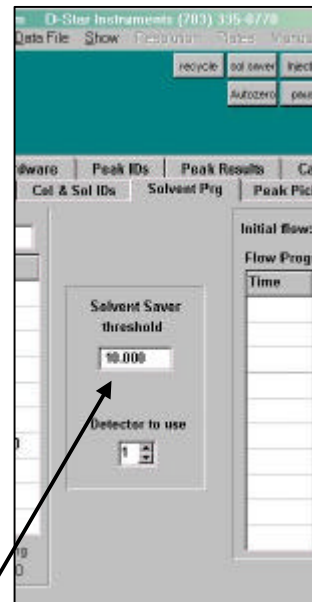
(three-way valve, 12VDC 300mA max.)



Normally closed, connects to waste bottle
 The valve is actuated when the baseline is outside of the threshold value

From Detector

Normally Open to solvent bottle



The opening and closing of the valve is automatically documented (see the history page). Solvent savings can be as much as 70%.

Default parameters for the DStarLCMicro.ini file:

10 maximum flow of solvent pump in mLs/min
7500 maximum pressure reading for pressure transducer
1 is the comm port for hardware control
1.000 detector 1 scale
0.000 detector 1 zero
1.000 detector 2 scale
0.000 detector 2 zero
1.000 pressure scale
0.000 pressure zero
0 limit for pump pressure too low
2500 limit for pump pressure too high
0.0 dead vol in mLs from mixer to injector
y use dstar detector
1.000 detector wavelength scale
0.000 detector wavelength zero
\\star-chrom 4042\wav files\ directory for wav files for notifying operator using sound

Files that are installed on the hard disk:

Directory of the C:\StarChrom folder and file descriptions

DataFiles	folder containing <i>sample</i> chr files (chromatograms)
Manuals	folder containing three Star-Chrom manuals pdf files)
wav files	folder containing numerous wav files (audible messages)
backup	folder containing any files saved from install/reinstall, optional
Calibrations.exe	Calibrations application
Columns.clm	initial columns file
DStar.ico	icon file with Star-Chrom logo
DStarLCMicro.ini	user replaced from diskette
Install.log	installation log
Loading Study verxxx.spm	example of a typical sample file
PeakPick.rtf	help documentation for peak picking
PeakPickStartup.val	initial values for peak picking
Pslcrun.nbr	index number file for auto-naming of chr files
ReportIcon.bmp	sample icon for use in reports (user configurable)
Solvents.slv	initial solvents file
Star_Chrom 4042.exe	Star-Chrom application
StartUp.lcm	initial column file
StartUp.pod	initial personalized information for reports
StartUpCalibParam.dat	initial calibration parameters
StartupCalibrdb.CDX	initial calibration database support
Startupcalibrdb.dbf	initial calibration database
Startupcalibrdb.fpt	initial calibration database support
StartUpPeakIDs.pip	initial peak identification parameters
StartUpZeros.zer	initial constants
SysAGT	hidden file containing system conformation information
Unwise.exe	uninstallation utility
Unwise32.exe	uninstallation utility
Viewer.exe	Viewer application

DPM-200 10 ML STACK PUMP MODULE



010-0266-6 (SS)

010-0266-7 (PEEK)

010-0266-8 (SS HI PRESS)

Operator's Manual

TABLE OF CONTENTS

1. INTRODUCTION.....	1-4
1.1 Description of the DPM-200 Pumps.....	1-4
1.1.1 Module Features.....	1-4
1.1.2 Wetted Materials.....	1-4
1.1.3 Self-Flushing Pump Heads.....	1-5
1.2 Specifications for the DPM-200 Pumps.....	1-6
1.3 Warranty.....	1-6
2. INSTALLATION.....	2-1
2.1 Unpacking and Inspection (Packing List).....	2-1
2.2 Location/Environment.....	2-2
2.3 Electrical Connections.....	2-2
2.3.1 Converting the Corcom from 115Vac to 230Vac.....	2-2
2.3.2 Converting from 230Vac to 115Vac.....	2-3
2.4 Solvent Preparation.....	2-4
2.4.1 Solvent Out-gassing and Sparging.....	2-4
2.4.2 Cavitation.....	2-5
2.4.3 Filtration.....	2-5
2.4.4 Solvents With Harmful Effects / Chemical Incompatibility.....	2-5
2.5 Instrument Installation.....	2-6
2.5.1 Mobile Phase Reservoirs.....	2-6
2.5.2 Self-Flush Solution.....	2-6
2.5.3 Inlet Tubing and Filters.....	2-7
2.5.4 Outlet Tubing.....	2-7
2.5.5 Priming the Pump and the Flushing Lines.....	2-8
2.5.6 Long Term Pressure Calibration Accuracy.....	2-8
2.6 Preparation for Storage or Shipping.....	2-8
2.6.1 Isopropanol Flush.....	2-8
2.6.2 Packaging for Shipping.....	2-8
2.7 Gradient Mixer (Option).....	2-9
2.8 Injection Valve.....	2-9
2.9 Column Holder.....	2-9
3. OPERATION.....	3-1
3.1 Front Panel and Indicators.....	3-1
3.1.1 Prime/Purge Valve.....	3-1
3.1.2 Hand Held Controller Panel.....	3-2
3.1.2.1 Digital Display.....	3-2
3.1.2.2 Keypad.....	3-2
3.1.2.3 Status LEDs.....	3-2
3.1.2.4 Power-up Configuration.....	3-2
3.1.3.5 Power-Up Tests.....	3-3
3.2 Rear Panel, Remote Inputs, and Output.....	3-4
3.2.1 Hardware Implementation.....	3-4
3.2.2 Hand-Shaking.....	3-5

3.2.3 Command Interpreter	3-5
4. THEORY OF OPERATION.....	4-1
4.1 Mechanical Operation.....	4-1
4.1.1 Liquid System Flow Path.....	4-1
4.1.2 Pump Cycle.....	4-1
4.1.3 Pulse Damping.....	4-1
4.2 Electronic Control.....	4-1
4.2.1 Microprocessor Control.....	4-1
4.2.2 DC Power Supply.....	4-2
4.2.3 Remote Interfacing.....	4-2
4.2.4 Motor Stall Detector	4-2
4.3 Gradient Operations.....	4-4
5. MAINTENANCE	5-1
5.1 Filter Replacement.....	5-1
5.1.1 Inlet Filters.....	5-1
5.2 Changing Pump Heads.....	5-1
5.2.1 Removing the Pump Head.....	5-1
5.2.2 Cleaning the Pump Head Assembly.....	5-2
5.2.3 Replacing Piston Seals.....	5-4
5.2.3.1 Removing the Seals.....	5-4
5.2.3.2 Cleaning the Piston.....	5-5
5.2.3.3 Replacing the Seals.....	5-5
5.2.4 Changing the Piston.....	5-5
5.2.5 Replacing the Pump Head.....	5-6
5.3 Conditioning New Seals	5-6
5.4 Check Valve Cleaning and Replacement.....	5-6
5.5 Pulse Damper Replacement.....	5-7
5.5.1 Removing the Pulse Damper	5-7
5.5.2 Pulse Damper Refurbishing.....	5-7
5.5.3 Pulse Damper Installation	5-7
5.6 Cleaning the Pump.....	5-8
5.7 Lubrication.....	5-8
5.8 Fuse Replacement.....	5-8
5.9 Battery Replacement.....	5-9
6. TROUBLESHOOTING.....	6-1
7. ACCESSORIES, REPLACEMENT AND SPARE PARTS	7-1
APPENDIX A	
A.1 Rear Panel Serial Communications Port.....	A-1
A.1.1 Hardware Implementation.....	A-1
A.1.2 Hand-Shaking	A-1
A.1.3 Command Interpreter	A-2

1. INTRODUCTION

This operator's manual contains information needed to install, operate, and perform user maintenance on the DPM-200 Stack HPLC Pump Module.

1.1 Description of the DPM-200 Pumps

The DPM-200 high performance liquid chromatography (HPLC) pump is designed to be a reliable component within a basic analytical or sophisticated research instruments. While ideal for HPLC applications, the DPM-200 pumps are also useful as metering pumps for general laboratory or industrial use.

The flow rate of the DPM-200 pump fitted with a standard 10 mL pump head can be set in 0.01 mL increments from 0.01 to 10.00 mL/min. The pump heads are available in type 316 stainless steel or in PEEK.

The low pulsation flow produced by the reciprocating, single-piston pump is achieved by using an advanced rapid-refill cam design, programmed stepper motor acceleration, and an internal pulse damper.

1.1.1 Module Features

The DPM-200 Pumps:

- Primarily used for analytical techniques. Contact your Dealer/Representative for preparative options.
- Incorporates a diaphragm-type pulse damper which reduces pulsation in the system by as much as 90% and includes an isolated pressure transducer (i.e., the transducer adds no dead volume).
- Integrated prime/purge and mixing valve.
- Self-flushing pump heads.
- Hand held controller (remote) LED readout shows the flow rate; and status LEDs.
- Microprocessor advanced control.
- Digital stepper motor design prevents flowrate drift over time and temperature, which is a common problem found in analog design.
- Back panel RS232 serial communications port for complete Star-Chrom HPLC Management software control.

1.1.2 Wetted Materials

Pump heads, check valve bodies, and tubing are made out of type 316 stainless steel or bio-compatible PEEK. Other materials are synthetic ruby and sapphire (check valve internals and piston) and fluorocarbon damper diaphragm.

1.1.3 Self-Flushing Pump Heads

Self-flushing pump heads provide continuous washing of the piston surface without the inconvenience of a manual flush or gravity feed arrangement. The self-flushing pump head uses a secondary seal and set of check valves to create a continuous and positive flow in the area behind the high pressure pump seal. The flushing solution washes away any buffer salts that have precipitated onto the piston. If not removed, these precipitates can abrade the high pressure seal and cause premature seal failure, leakage, and can possibly damage the pump.

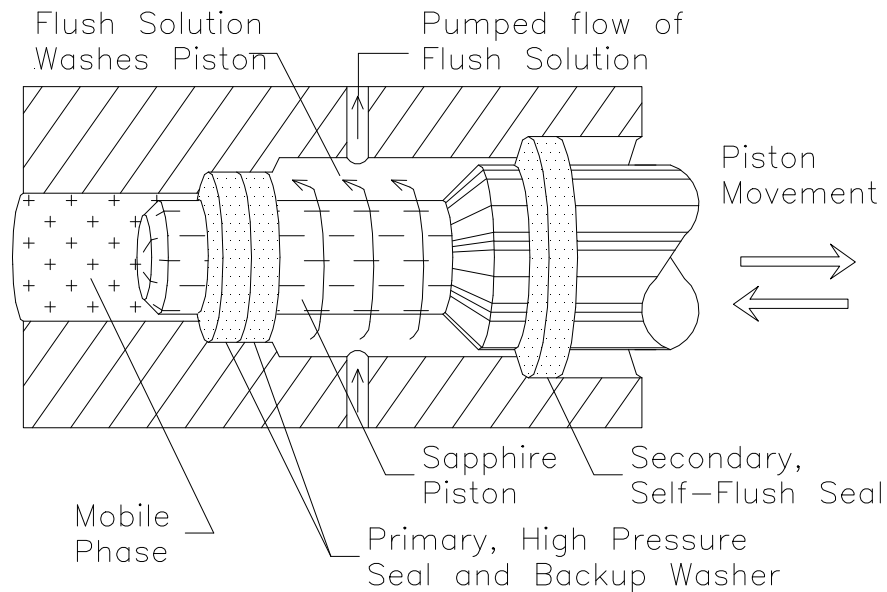


Figure 1-1. Self-Flushing Pump Head

1.2 Specifications for the DPM-200 Pumps

Flow Rates	0.01 to 10.00 mL/min for 10 mL/min head
Pressure	0 to 2,500 psi for 10 mL/min pump heads 0 to 5,000 psi for 10 mL/min high-pressure SS pump head
Pressure Accuracy	$\pm 1\%$ of full scale pressure
Pressure Zero Offset	± 2 psi
Flow Accuracy	3% for flowrates of 0.33 ml/min and above; 0.01 ml/min for flowrates below 0.33 ml/min
Flow Precision	0.5% RSD
Power	110-120 Vac, 50-60 Hz; or 220-240 Vac, 50-60 Hz
Features	Three-way prime purge valve Pulse damper with pressure transducer Prime purging
Remote Inputs	RS-232
Remote Control	External hand-held control panel for diagnostics and monitoring single pump performance

1.3 Warranty

No other warranty exists, expressed or implied, except as shown here and in D-Star Instruments' Conditions of Sale.

Fitness to a particular application must be determined by the user.

Parts & labor are warranted for one (1) year, provided the unit is not damaged by improper use, abuse or by chemical spill.

Request a Return Authorization (RA) before returning any parts.

Return defective unit to Dealer for repair. Clean/sterilize all components prior to shipment. No parts will be accepted which present a health or safety hazard to service personnel. Repaired unit will be returned via parcel service or mail.

The flowcell and all tubing supplied with the instrument are warranted at the time of installation only.

2. INSTALLATION

2.1 Unpacking and Inspection

Prior to opening the shipping container, inspect it for damage or evidence of mishandling. If it has been damaged or mishandled, notify the carrier before opening the container. Once the container is opened, inspect the contents for damage. Any damage should be reported to the carrier immediately. Save the shipping container. Check the contents against the packing list.

PACKING LIST

DPM-200 Binary Pump Module [for Gradient system]:

110/115V SS [DS015-0032], 220/240V [DS015-0032-1]

110/115V PEEK [DS015-0032-2], 220/240V [DS015-0032-3]

110/115V SS High Press [DS015-0032-4], 220/240V [DS015-0032-5]

with Accessory Package - DPM-200 [DS010-0085-2] that includes:

Fuses:

Spare Fuses-Corcom (115V): 5 x 20 mm, SLOW, 250V, 1.0A [DS430-0007]...2 ea

Spare Fuses-Corcom (230V): 5 x 20 mm, SLOW, 250V, 0.5A [DS430-0008]...2 ea

Spare Fuse-Pump: 5 x 20 mm, FAST, 250V, 5A [DS430-0006]...2 ea

Tubing:

Inlet Flush Assembly [DS010-0069]...2 ea

Outlet Flush Assembly [DS010-0070]...2 ea

Solvent Inlet Filter Assembly [DS010-0072]...2 ea

Syringe, Luer Lok, 30 cc [DS443-0001]...1 ea

Column Plug, Outlet Check Valve and Pulse Damper, 1/16" [DS250-0028]...3 ea

DIV-1, Through-the-Handle Injection Valve Assy w/ 20uL Sample Loop & tubing...1 ea

Column Holder assembly...1 ea

Bulkhead to DIV-1 tubing assembly (SS)...1 ea

Column to Detector flowcell tubing assembly (SS) & 1-piece Column Fittings (2)...1 set

Detector to Waste tubing assembly (CTFE)...1 set

Hand-held control panel...1 ea

Power Cord 115V [DS610-0004] or 230V [DS610-0005]...1 ea

Operating Manual (this document) [DS050-0021] ...1 ea

Operating Instructions, DIV-1 Injection Valve [DS090-0021] ...1 ea

NOTES:



1. Part numbers shown above are for shipped kits. Parts numbers shown elsewhere in the manual are for replacement/spare part ordering.
2. Items indicated in the manual with the word "optional" are not supplied unless specifically ordered (additional cost).

2.2 Location/Environment

The preferred environment for the DPM-200 pump is normal laboratory conditions. The area should be clean and have a stable temperature and humidity. The specific temperature and humidity conditions are 10 to 30 °C and 20% to 90% relative humidity. The instrument should be located on a stable flat surface with surrounding space for ventilation and the necessary electrical and fluid connections.

2.3 Electrical Connections

Unpack the DPM-200 pump module and check the voltage setting on the Power Entry Module (Corcom) on the back panel (see Figure 2-1). Make certain the voltage setting agrees with the power to be supplied to the unit. A module which is connected to a 100-120Vac voltage source should have a voltage setting of "115V", and a module connected to a 220-240Vac voltage source should have a voltage setting of "230V". The pump can be ordered with either of the two voltage configurations.

If the voltage setting is correct, position the pump module so that there is at least a one inch clearance on all sides of the module to permit proper ventilation. Then plug the pump into a properly grounded electrical outlet.

WARNING: Do not bypass the safety ground connection as a serious shock hazard could result.

If the module does not have the correct voltage configuration, notify your representative, to obtain the correct power cord and fuses for your installation.

To change the voltage setting, a small flat-blade screwdriver is the only tool required; proceed as follows:

2.3.1 *Converting the Corcom from 115Vac to 230Vac*

1. Remove the power cord from the power entry module (see Figure 2-1) located in the rear of the pump.
2. Using a small, flat-blade screwdriver, carefully pry out the power entry module cover at point A, Figure 2-1. The hinged cover will swing down as shown in Figure 2-2.

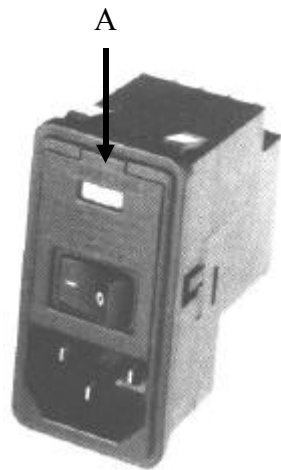


Figure 2-1

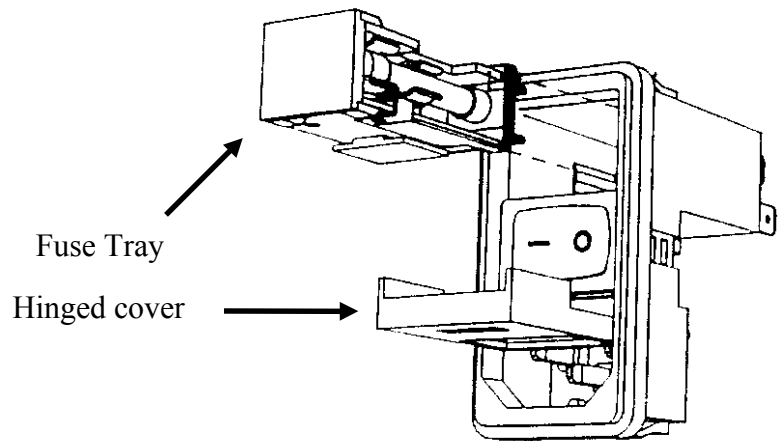


Figure 2-2

3. Using a small, flat-blade screwdriver, carefully pry out at the top of the fuse tray and carefully remove the tray as illustrated in Figure 2-2.
4. Remove the fuses present in the fuse tray by gently lifting it up at the rearmost end of the tray (where the metal tabs extend) and removing the fuse from the retaining clip.
5. Place two 230V fuses into the two compartments in the fuse tray. The fuse ratings **must be as shown on the back of the chassis**. The 230V 5x20 mm long fuses **must** be placed fully to the rearmost end of the tray (closest to the metal tabs extending from the tray), or contact will not be made and the unit will not operate. **Both** fuses must be present to complete the circuit.
6. With the two proper fuses in the tray, orient the tray with the two fuses at the sides and the **230V marking at the top**, as shown in Figure 2-2, and insert it with the metal tabs first into the top of the power entry module until it is firmly seated in place. It may be necessary to squeeze the two fuses at the rear to permit them to enter the power module housing. Close the power entry module cover by pressing at the top center until it snaps into place. Insert a suitable 230V power cord.

2.3.2 Converting from 230Vac to 115Vac

1. Remove the power cord from the power entry module (see Figure 2-1) located in the rear of the pump; pry out the power entry module cover at point A, Figure 2-1; pry out at the top of the fuse tray and carefully remove the tray as illustrated in Figure 2-2.
2. Exchange the 230V fuses in the fuse tray with the 115V fuses.
3. Orient the tray with the fuse at the side and the **115V marking at the top**, as shown in Figure 2-2, and insert it with the metal tabs first into the top of the power entry

module until it is firmly seated in place. It may be necessary to squeeze the fuse at the rear to permit it to enter the power module housing. (If the clip and fuse are on the incorrect sides of the tray, it will not be possible to place the tray into the power entry module). Close the power entry module cover by pressing at the top center until it snaps into place. Insert a suitable 115V power cord.

2.4 Solvent Preparation

Proper solvent preparation will prevent a great number of pumping problems. The most common problem is bubble formation, which may affect the flow rate consistency. Aside from leaky fittings, the problem of bubble formation arises from two sources: solvent out-gassing and cavitation. Filtration of HPLC solvents is also required.

2.4.1 Solvent Out-gassing and Sparging

Solvent out-gassing occurs because the mobile phase contains dissolved atmospheric gases, primarily N₂ and O₂. These dissolved gases may lead to bubble formation and should be removed by degassing the mobile phase before or during use. The best practical technique for degassing is to sparge the solvent with standard laboratory grade (99.9+%) helium. Helium is only sparingly soluble in HPLC solvents, so other gases dissolved in the solvent diffuse into the helium bubbles and are swept from the system. Solvent filtration is not an effective alternative to helium degassing.

It is recommended that you sparge the solvent vigorously for 10 to 15 minutes before using it. Then maintain a trickle sparge during use to keep atmospheric gases from dissolving back into the mobile phase. The sparged solvent must be continually blanketed with helium at 2 to 3 psi. Non-blanketed sparged solvents will have atmospheric gases dissolved back into the mobile phase within four hours.

Solvent mixtures using water and organic solvents (like methanol or acetonitrile) hold less dissolved gas than pure solvents. Sparging to reduce the amount of dissolved gas is therefore particularly important when utilizing solvent mixture.

Even with sparging some out-gassing may occur. A back pressure regulator installed after the detector flow cell will help prevent bubbles from forming and thus limit baseline noise.

WARNING: Always release pressure from the pump slowly. A rapid pressure release could cause the pulse damper diaphragm to rupture.

2.4.2 Cavitation

Cavitation occurs when inlet conditions restrict the flow of solvent and vapor bubbles are formed during the inlet stroke. The key to preventing cavitation is to reduce inlet restrictions. The most common causes of inlet restrictions are crimped inlet lines and plugged inlet filters.

Inlet lines with tubing longer than 48" (120 cm) or with tubing of less than 0.085" (2 mm) ID may also cause cavitation.

Placing the solvent reservoirs below the pump level also promotes cavitation. The optimal location of the reservoirs is slightly above the pump level, but it is adequate to have them on the same level as the pump.

2.4.3 Filtration

Solvent filtration is good practice for the reliability of the DPM-200 pumps and other components in a HPLC system. Solvents should always be filtered with a 0.5 micron filter prior to use. This ensures that no particles will interfere with the reliable operation of the piston seals and check valves. Solvents in which buffers or other salts readily precipitate out will need to be filtered more often. After filtration, the solvents should be stored in a closed, particulate-free bottle.

2.4.4 Solvents With Harmful Effects / **Chemical Incompatibility**

It is the user's responsibility to determine which solvents are compatible with the materials of construction listed above. Please note that the following advisories have been provided by suppliers of parts and materials:

PEEK - *Concentrated Nitric Acid and concentrated Sulfuric Acid* attack PEEK chemically and will cause severe effects. **NOT RECOMMENDED.**

Methylene Chloride (MeCl), DMSO, and THF cause PEEK to swell. *MeCl* has been observed to cause flaking. **NOT RECOMMENDED.**

FUSED SILICA - Avoid high pH solvents (8.5+) which will etch the window (synthetic quartz). High pH solutions may be used for short periods of time (one minute) to clean protein buildups.

TEFZEL - Some *chlorinated chemicals* may cause swelling.

DELTRIN - Not suitable for use with *acids, bases, or oxidizing agents* [This is usually a non-wetted part in normal operation].

STAINLESS STEEL - Stainless steel components that contact the mobile phase are extremely sensitive to acids (including some Lewis acids). Avoid using solvents that contain any amount of hydrochloric acid.

Some solvents you should specifically avoid are:

Aqua Regia	Hydrobromic Acid
Bromine	Hydrochloric Acid
Aqueous Chlorine	Hydrofluoric Acid

Aqueous Copper Chloride Ferric Chloride Solution Ferrous Chloride Solution Freon 12 (wet)	Hydrofluorosilicic Acid Hydrogen Peroxide Aqueous Iodine Mercuric Chloride Solution
--	--

In addition, some users of HPLC systems have observed that chloroform, carbon tetrachloride, and methylene chloride slowly decompose/hydrolyze to liberate hydrochloric acid, which, as noted above, attacks stainless steel. Do not leave these solvents in the systems for a prolonged period. Flushing daily is recommended.

It is also recommended that you avoid ammonium hydroxide. Although ammonium hydroxide will not harm the pump itself, it may damage the stator and rotor in the Injection Valve.

Contact your Dealer/Representative or D-Star Instruments for information on other flowcells that may be better suited for your application.

2.5 Instrument Installation

2.5.1 Mobile Phase Reservoirs

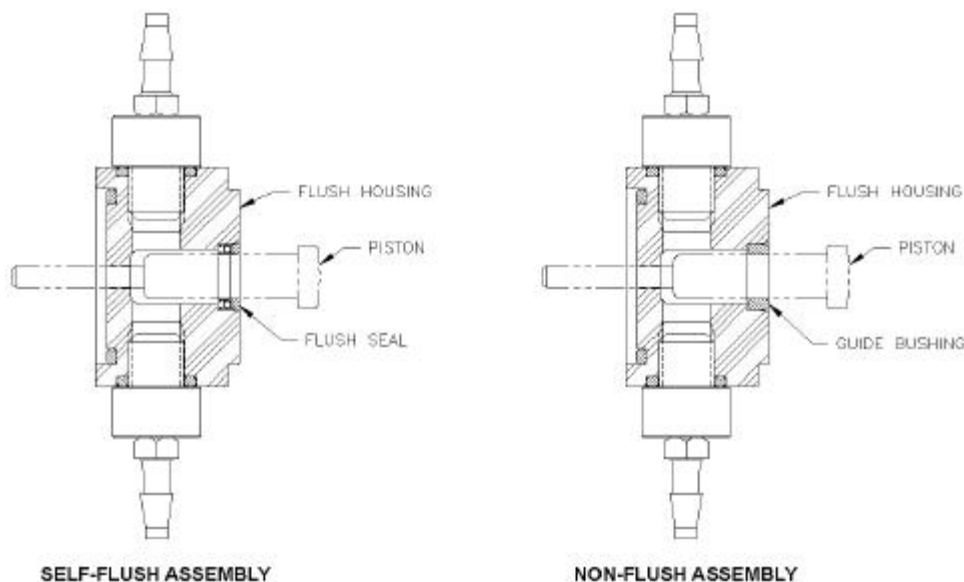
The mobile phase reservoir should be placed at the same level or slightly higher than the pump, never below the pump, and the inlet tubing should be as short as practical. These steps minimize pressure losses on the inlet side of the pump during refill and help to avoid bubble formation. These steps are particularly important when using high vapor pressure solvents (hexane, methylene chloride, etc.). Mobile phases should be degassed, filtered and covered. (See Section 2.4.)

2.5.2 Self-Flush Solution

Self-flush heads require 250-500 mL of 20% methanol in water as a flushing solution. A pH indicator that will indicate the concentration of salts in the solution is recommended as a reminder to change the solution. This flush solution should be replaced with a fresh solution weekly to avoid frequent pump maintenance.

WARNING: If you do not use the self-flush feature of this pump, you must carefully remove the self-flush seal with the seal tool provided and place the seal into the guide bushing bag. Replace the flush seal with the guide bushing provided (See illustration below). If this is not done; low flow rates, excessive noise and shortened pump life will result.

THIS IS NOT RECOMMENDED



2.5.3 Inlet Tubing and Filters

The following table shows the inlet tubing used in the DPM-200 pumps. All inlet lines are supplied in a 36" (91 cm) length, and are made of a Teflon-based material. Use a 0.5 micron slip-on inlet filter.

Pump Head Type	Inlet Tubing Size
All	0.085" ID x 1/8" OD

2.5.4 Outlet Tubing

Outlet tubing is a 0.030" ID, 1/16" outer diameter. Tubing with a 0.030" inner diameter is from the pumphead to the prime-purge valve (PPV). Tubing with a 0.010" inner diameter is used after the PPV in the 10mL analytical system. Any tubing must be cut squarely with no burrs. The tube itself should not be crimped and the center hole must be open. A tubing cutter is recommended for cutting stainless steel tubing.

2.5.5 Priming the Pump and the Flushing Lines

Be sure all of the connections downstream of the prime/purge valve are closed. Connect a syringe to the prime/purge valve. Open the prime/purge valve 1 to 2 turns (counter-clockwise). Using the Hand Held Controller, run each pump at a flowrate of 3 to 5 mL/min. Prime the pump by pulling mobile phase and any air bubbles through the system and into the syringe (a minimum of 20 mL). Close the prime/purge valve and stop the pump.

To prime the flush lines for a self-flush head, connect one of the small Luer-to-barb fittings to a syringe and pull 10-20 mL of flush solution through the outlet line (at the top of the pump head).

2.5.6 Long Term Pressure Calibration Accuracy

This note applies if your pump is equipped with an electronic pressure transducer. The transducer has been zeroed and calibrated at the factory. Over the life of the pump, some drift may occur. For example, it is typical for the zero to drift < 10 p.s.i. after about 1 year of operation (i.e., with no back pressure on the pump a reading of 1-9 p.s.i. may be displayed). A similar drift may also occur at higher pressures, and are typically less than 1% (e.g. <50 p.s.i. at 5,000 p.s.i. back pressure).

If pressure calibration and/or drift is a concern, consult the factory. The pump can be shipped back to the factory for recalibration. Alternatively, written calibration and zero-reset procedures are available. Consult the factory to receive these instructions.

2.6 Preparation for Storage or Shipping

2.6.1 Isopropanol Flush

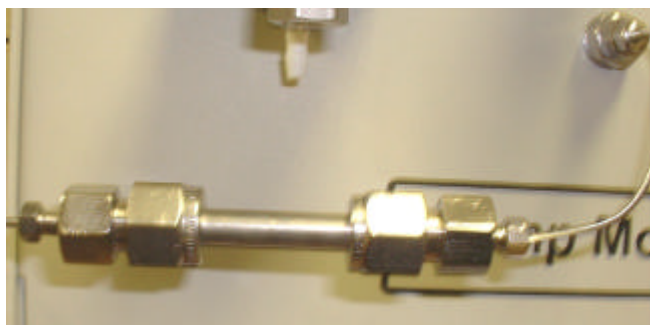
Disconnect the outlet tubing from the pump. Insert the inlet filter in isopropanol. Open the prime/purge valve and use a syringe to draw a minimum of 50 mL. Close the prime/purge valve and pump a minimum of 5 mL of isopropanol to exit. Leave the inlet tubing connected to the pump. Place the inlet filter in a small plastic bag and attach it to the tubing with a rubber band. Plug the outlet port with the shipping plug, leave a length of outlet tubing on the pump, or cover the outlet port with plastic film.

2.6.2 Packaging for Shipping

CAUTION: Re-package the DPM in the original carton, if possible. If the original carton is not available, wrap the module in several layers of bubble wrap and cushion the bottom, top, and all four sides with 2" of packaging foam. Although heavy, HPLC pumps are delicate instruments and must be carefully packaged to withstand the shocks and vibration of shipment.

2.7 Gradient Mixer (Option) [See section 4.3 for detailed discussion of mixer options]

Locate the gradient mixer. Remove the bulkhead to injector valve line at the bulkhead and insert it into the open end of the mixer. Install the tubing end to the bulkhead.



2.8 Injection Valve (See the Valve Instructions for additional information)

Locate the valve and install it on the left side of the chassis as shown in the photos. Use the screw holding the cover and the second screw in the cover to fasten the valve to the unit. Use the lower holes as shown. Although the valve may be used on either side, closest to the column and flowcell is recommended.

Connect the DB9 plug to the autosampler connector on Sta-Chrom interface box.

Do not ship the DPM with the DIV-1 Injector valve attached to the cover.

2.9 Column Holder

Locate the column holder. Slip the screws on the side of the cover through the holder and allow the holder to drop in to the slots. The holder has been tested with a one inch column.

Do not ship the unit with the column holder attached.



3. OPERATION

3.1 Front Panel and Indicators



Figure 3-1. DPM-200 Pump Front Panel

3.1.1 Prime/Purge Valve

CAUTION: When you press the PRIME key on the hand held controller, the pump will run at the maximum flow rate. Be sure the prime/purge valve is open.

The prime/purge valve vents the flow to atmosphere and permits efficient priming of the DPM-200 pump. When the valve is closed (fully clock-wise) firmly, high-pressure flow is directed to the bulkhead Outlet port. When the valve is opened (counter clock-wise) one-half to one full turn, pressure is vented and flow exits through the front drain port in the prime/purge valve stem assembly. Suction with a Luer tip syringe at the drain port will purge air bubbles from the pump and reservoir lines (provided there are no open valves to lines downstream at the injector/column interface). To prime the pump, draw about 20 to 30 mL of mobile phase.

3.1.2 Hand Held Controller Panel

3.1.2.1 Digital Display

The 3-digit display shows the pump flow rate (mL/min).

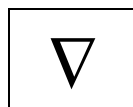
3.1.2.2 Keypad



When pressed, this button alternately starts and stops the pump.



When pressed, this button increases the flow rate.



When pressed, this button decreases the flow rate.



When the PRIME button is pressed, the pump runs at the maximum flow rate for the pump head. It will stop when any button is pressed.

Fast And Slow Button Repeat On The Up And Down Arrow Buttons: If the UP-ARROW or DOWN-ARROW button is held down for more than approximately one half of a second, the button press will repeat at a slow rate of approximately 10 times a second. Once slow button repeat has begun, fast button repeat can be initiated by using a second finger to press down the second arrow button. During fast button repeat, the button press will repeat at a rate of approximately 100 times a second. Switching back and forth between repeat speeds can be accomplished by pressing and releasing the second arrow button while keeping the first arrow button held down.

3.1.2.3 Status LEDs

ML/MIN	When lit, the digital display shows flow rate in mL/min.
PUMP RUN	Lights to indicate that the pump is running.
FAULT	Lights when a fault occurs and stops the pump.

3.1.2.4 Power-up Configuration

Pressure Compensation: On power-up, press the PRIME button on the front panel while pressing the Power On switch on the rear of the pump. The pump will display a number from 0 to 50 (10ml head). This represents the running pressure of the pump from 0 psi to 5000 psi. Each digit represents 100 psi. To change the pressure compensation number use the up arrow and down arrow buttons. When you have selected the correct pressure compensation press the RUN button to return to normal operation of the pump.

Non-volatile Memory Reset: If the pump is operating erratically, there is the possibility that the memory has been corrupted. To reset the memory and restore the pump to its default parameters, press and hold the UP-ARROW button when the power is switched on. Release the button when the display reads "rES". The parameters stored in non-volatile memory, i.e., the flowrate, the pressure compensation, the voltage/frequency select, the lower pressure limit, and the upper pressure limit will be set to the factory default values. The head type setting is the only parameter not changed by the non-volatile memory reset function. If the firmware is upgraded to a newer version, a non-volatile memory reset will automatically occur the first time the power is switched on.

3.1.3.5 Power-Up Tests

Display Software Version Mode: The software version can be displayed during power-up by pressing and holding the RUN/STOP and the UP-ARROW buttons when the power is switched on. Release the buttons when the display reads "UEr". The decimal point number displayed on the display is the software version. To exit this mode, press the RUN/STOP button.

Align Refill Switch Mode: The signal that initiates the refill phase can be displayed during power-up by pressing and holding the PRIME and the UP-ARROW buttons when the power is switched on. Release the buttons when the display displays "rFL". When the slotted disk allows the light beam to pass from the emitter to the detector on the slotted optical switch a pulse will be generated which signals the beginning of refill. When this pulse occurs the three horizontal segments displayed at the top of the display will turn off and the three horizontal segments at the bottom of the display will turn on. To exit this mode, press the RUN/STOP button.

Serial Port Loopback Test Mode: If an external device will not communicate to the pump via the serial port, the serial port loopback test can be used to verify that the serial port is functioning properly. During power-up press and hold the UP-ARROW and the DOWN-ARROW buttons when the power is switched on and then release the buttons. The display must display "C00" for the first half of the test to pass. Plug in the serial port loop back plug (A modular plug with pins 2 & 5 jumpered together and pins 3 & 4 jumpered together.). The display must read "C11" for the second half of the test to pass. To exit this mode, press the RUN/STOP button.

3.2 Rear Panel, Remote Inputs, and Output

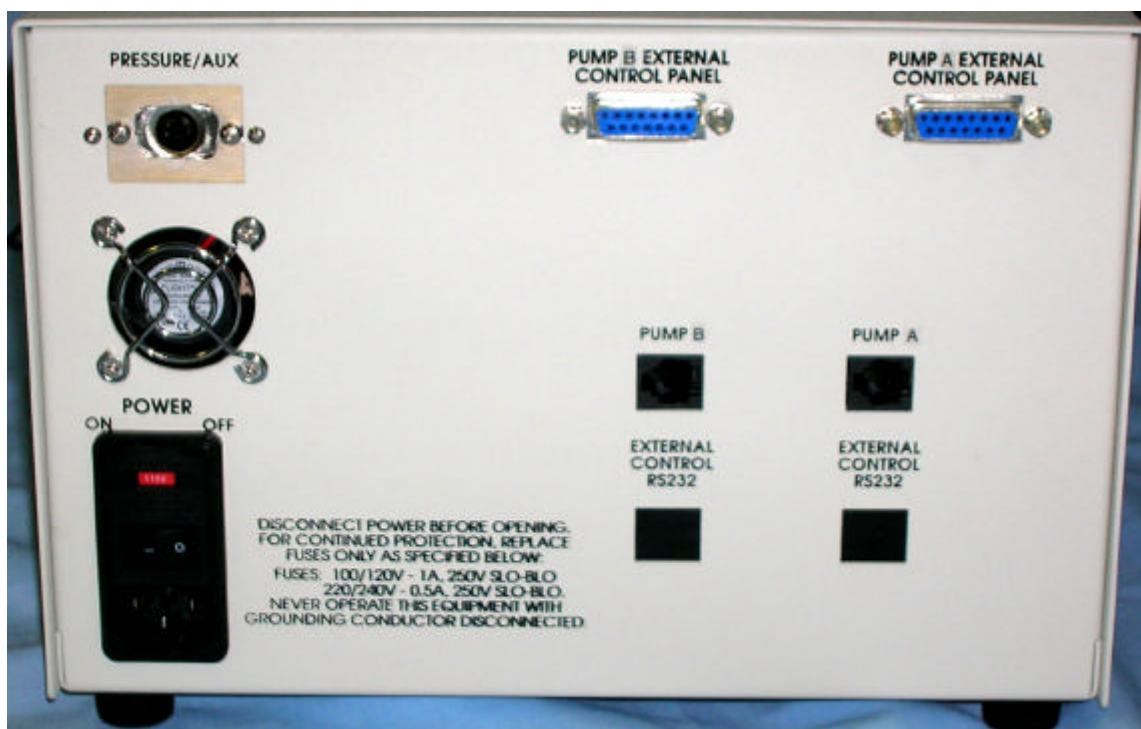


Figure 3-2. DPM-200 Pump Rear Panel

RS-232C modular jacks are provided on the back panel. The Star-Chrom HPLC Management software is used to control the pump operation via these connections.

Attach the diagnostic hand held controller to the *EXTERNAL CONTROL PANEL CONNECTORS*

3.2.1 Hardware Implementation

The EXTERNAL CONTROL RS232 serial communications port (RJ11) is configured for 9600 baud, 8 data bits, 1 stop bit, and no parity. The connector is a standard RJ-11 modular telephone type jack. The pin-out is:

<u>Pin</u>	<u>Function</u>
1, 6	Ground
2	DSR (Handshaking input to pump)
3	RXD (Serial data input to pump)
4	TXD (Serial data output from pump)
5	DTR (Handshaking output from pump)

Special wiring considerations: Use the following chart for interfacing the DPM-200 pump's serial communications port to either a 25-pin or a 9-pin COM port on an IBM-PC type computer.

<u>Pump (RJ11)</u>	<u>Signal</u>	<u>IBM (DB25)^a</u>	<u>IBM (DB9)^b</u>
1, 6	Ground	7	5
2	DSR	20	4
3	RXD	2	3
4	TXD	3	2
5	DTR	6	6

^a Jumper pins 4, 5, and 8 on DB25.
^b Jumper pins 1, 7, and 8 on DB9.

3.2.2 Hand-Shaking

The DPM-200 pump uses hardware handshaking. The pump monitors the DSR input and disables the DTR output when the pump is in refill. In addition, the pump will not transmit if the DSR input becomes active.

3.2.3 Command Interpreter

The DPM-200 pump's high-level command interpreter receives and responds to command packets. The pump will not send a message except when prompted, and it will send a response to every valid command as described below. The response to an invalid command is "Er".

Each command is characterized by a unique two-letter command code, and only one command can be issued per line. Case is not important; that is, the command codes "PR" "Pr" "pR" and "pr" are all equivalent. Response strings sent by the pump are terminated by the "/" character. The command packets are as follows:

Command	Response	Comments
RU	OK/	Sets the pump to the RUN state.
ST	OK/	Sets the pump to the STOP state.
FLxxx	OK/	Sets the flowrate to x.xx or xx.x mL/min where the range is fixed for the pump head size, i.e., for 0.01 to 9.99 mL/min xxx = 001 to 999
FOxxxx	OK/	Sets the flowrate to xx.xx or xxx.x mL/min where the range is fixed for the pump head size, i.e., for 0.01 to 10.00 mL/min xxxx = 0001 to 1000
PR	OK,x/ (x, xx, xxx, or xxxx)	Reads the pump's current pressure, where: x, xx, xxx, or xxxx = Current pressure in PSI
CC	OK,x,y.yy/ (x, xx, xxx, or xxxx) (y.yy, yy.yy, or yy.y)	Reads the pump's current pressure and flowrate, where: x, xx, xxx, or xxxx = Current pressure in PSI y.yy, yy.yy, or yy.y = Flowrate in mL/min The format is y.yy and yy.yy for a standard pump head or yy.y for a macro pump head.
CS	OK,x.xx,y,z,PSI,w,v,u/ (x.xx, xx.xx, or xx.x) (y, yy, yyy, or yyyy) (z, zz, zzz, or zzzz)	Reads the current pump setup, where: x.xx, xx.xx, or xx.x = Flowrate in mL/min y, yy, yyy, or yyyy = Upper pressure limit z, zz, zzz, or zzzz = Lower pressure limit PSI = Units (PSI, ATM, MPA, BAR, or KGC) w = Pump head size (0 = standard, 1 = macro) v = Run status (0 = stopped, 1 = running) u = Pressure Board present = 0; otherwise 1
ID	OK,vx.xx SR3O firmware/	Identifies the pump type and EPROM revision x.xx
SF	OK/	Puts the pump in fault mode. Turns on the FAULT LED and stops the pump immediately.
RF	OK,x,y,z/	Reads the fault status, where: x = Motor stall fault (0 = no, 1 = yes) y = Upper pressure limit fault (0 = no, 1 = yes) z = Lower pressure limit fault (0 = no, 1 = yes)
KD	OK/	Disables the keypad. (Default status at power-up is enabled.)
KE	OK/	Enables the keypad.
PCxx	OK/	Sets the pressure compensation value, where xx = the operating pressure (in PSI divided by 100), i.e., for 0 PSI xx = 00, for 5000 PSI xx = 50.
RC	OK,x/ (x or xx)	Reads the pressure compensation value in hundreds of PSI, i.e., for 0 PSI x = 0, for 5000 PSI xx = 50.
HTx	OK/	Sets the pump head type, where: x = 1 for a stainless steel 10 mL/min pump head x = 2 for a plastic 10 mL/min pump head (n/a) The pump is stopped; and, the pressure compensation and pressure limits are initialized, when the head type is changed.

RH	OK,x/	Reads the pump head type, where: x = 1 for a stainless steel 10 mL/min pump head x = 2 for a plastic 10 mL/min pump head (n/a)
PI	OK,a.aa,b,c,d,e,f,g,h,i,j,k,l, m,n,o,p,q/ (a.aa, aa.aa, or aa.a) (c or cc)	Reads the current pump setup, where: a.aa, aa.aa, or aa.a = Flowrate in mL/min b = Run status (0 = stopped, 1 = running) c or cc = Pressure compensation d = Pump head type (see RH command) e = Pressure Board present = 0; otherwise 1 f = External control mode (0 = frequency, 1 = voltage) g = 1 if pump started and frequency controlled, else 0 h = 1 if pump started and voltage controlled, else 0 i = Upper pressure limit fault (0 = no, 1 = yes) j = Lower pressure limit fault (0 = no, 1 = yes) k = Priming (0 = no, 1 = yes) l = Keypad lockout (0 = no, 1 = yes) m = PUMP-RUN input (0 = inactive, 1 = active) n = PUMP-STOP input (0 = inactive, 1 = active) o = ENABLE IN input(0 = inactive, 1 =active) p = Always 0 q = Motor stall fault (0 = no, 1 = yes)
RE	OK/	Resets the pump configuration to its default power-up state.
#	(no response)	Clears all characters from the command buffer.

If the pump's response is "Er/", send a "#" to clear any characters which may be remaining in the command buffer. The pump will automatically clear all characters in the command buffer after one second elapses from the time at which the last character of an incomplete command was sent.

4. THEORY OF OPERATION

4.1 Mechanical Operation

4.1.1 Liquid System Flow Path

The flow path of the DPM-200 pump starts at the inlet of the pump head, passes through the pump head into the prime/purge valve through the pulse damper, then finally through the bulkhead and out the front panel of the pump module.

4.1.2 Pump Cycle

The pump cycle consists of two phases, the pumping phase and the refill phase.

During the pumping phase, the pump piston moves at a constant linear speed, driven by a specially shaped cam which is in turn driven by the motor using a toothed-belt drive. This results in a constant, stable flow from the pump at high pressure.

At the end of the pumping phase, the pump enters the refill phase. The cam is shaped so that the piston quickly retracts, refilling the pump head with solvent. The piston then moves forward again as the pumping phase begins. Since the output flow completely stops during refill, a pulse damper is necessary to provide some of the lost flow (see 4.1.3 below). In addition, the motor speed is adjusted by the microprocessor to facilitate an efficient refill phase.

The combination of increased motor speed and the rapid refill design of the cam generates refill times of less than 12.5% of the pump cycle (the refill time at 1 mL/min is less than 5% of the pump cycle).

4.1.3 Pulse Damping

The diaphragm-type pulse damper consists of a compressible fluid (isopropanol) held in an isolated cavity by an inert but flexible diaphragm. During the pumping phase of the pump cycle, the fluid pressure of the mobile phase displaces the diaphragm, compressing the fluid in the cavity and storing energy. During the pump refill phase the pressure on the diaphragm is reduced and the compressed fluid expands, releasing the energy it has stored. This helps to stabilize flow rate and pressure. The amount of mobile phase in contact with the pulse damper is small, only 1.2 mL at 6,000 psi, and the geometry used insures that the flow path is completely swept, so solvent "memory effects" are virtually eliminated.

4.2 Electronic Control

4.2.1 Microprocessor Control

The pump is controlled by hybrid microprocessor circuitry which (1) provides control signals to the motor power board, (2) interfaces with the keyboard/display, (3) receives signals from

the pressure transducer and refill flag, and (4) provides external input/output and remote control interfacing. Firmware programming is stored in an EPROM.

The motor power board contains programmed logic components which (1) provide suitable motor micro-stepping modes, (2) allow appropriate motor power adjustment, (3) maximize motor power output, (4) reduce motor resonance effects, and (5) customize motor stepping uniformity. MOSFET power transistors efficiently control the motor power provided by a 36 Vdc linear power supply. This board also provides the 12 Vdc (linear power supply) and the 5 Vdc (switching power supply) used by the pump circuits.

A specially shaped cam provides refill in a fraction of the full cam revolution. The remaining revolution of the cam provides a linear piston displacement for constant flow of the mobile phase. In addition to the rapid refill characteristics of the cam, the onset of refill is detected by an infrared optical sensor. The microprocessor changes the refill speed of the motor to an optimum for the set flow rate. As a result, at 1mL/min flow the refill rate is more than five times faster than if the motor operated at constant speed. The optimum refill minimizes the resulting pulsation while avoiding cavitation effects in the solvent entering the pump head.

The flow rate of any high pressure pump can vary depending on the operating pressure and the compressibility of the fluid being pumped. The pump is calibrated at 2,000 psi using a 80:20 mixture of water and isopropanol.

The pulse damper of the DPM-200 pump has a built-in pressure transducer which senses fluid pressure. The output is sent to the microprocessor circuit, which provides the information presented on the digital display. This pressure information is compared with the user-set upper and lower pressure limits to control pump shut-off if the limits are exceeded.

4.2.2 DC Power Supply

Power for the pump is provided by an isolation transformer, which has taps to accommodate voltages of 110/120 or 220/240 volts AC. Selection is accomplished by changing the transformer jumpers. A different transformer is used for 100 volts AC. The transformer input is provided with two fuses for line current. A fused linear rectifier circuit provides 36 Vdc to drive the stepping motor. A linear 12 Vdc supply and a switching 5 Vdc supply are also provided to power control and display circuits.

4.2.3 Remote Interfacing

An RS-232C modular jack is provided on the back panel. See Section 3.2 for information on pump operation via this connection.

4.2.4 Motor Stall Detector

The motor can stall and create a loud buzzing sound if the flow path connected to the pump's outlet becomes plugged, if the pressure exceeds the maximum pressure rating of the pump, or

if the mechanism jams. In the event a motor stall occurs, the electrical current being supplied to the motor is turned off and the fault light is turned on.

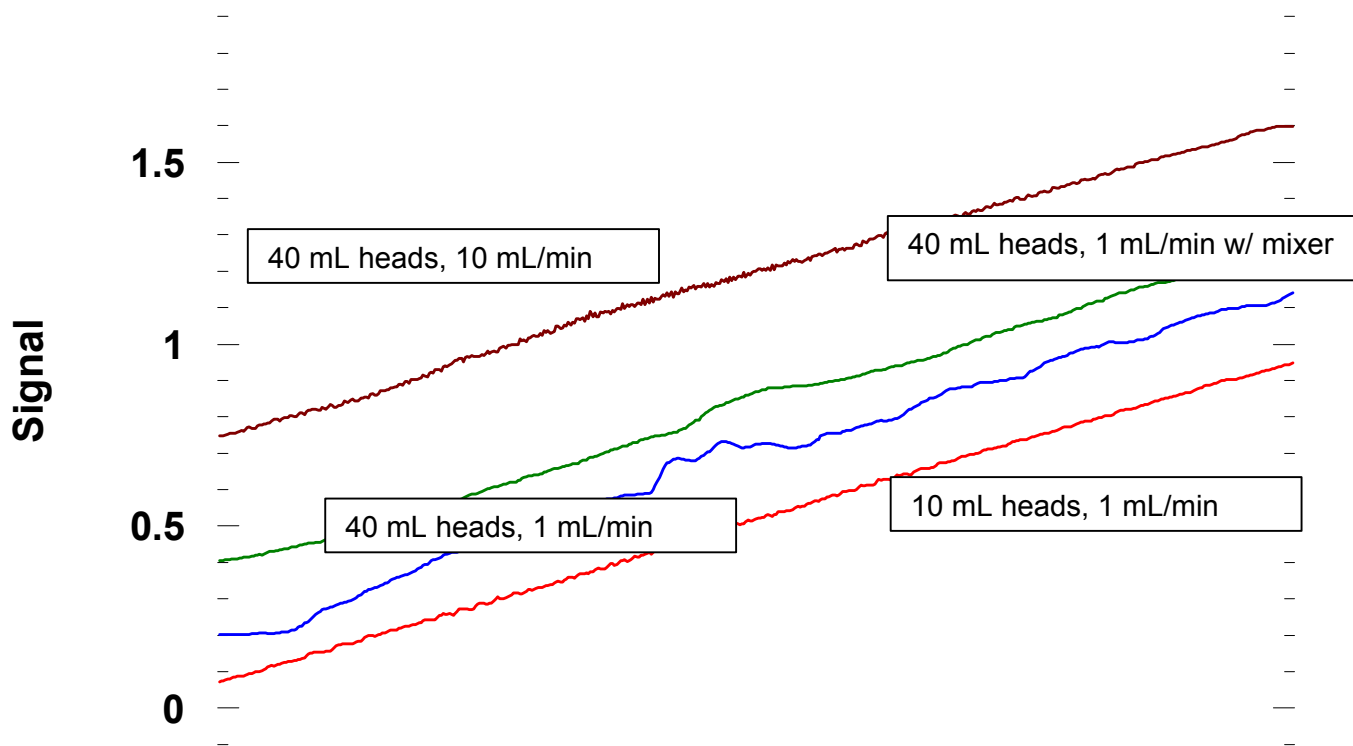
The Motor Stall Detector is enabled or disabled during power-up by pressing and holding the RUN/STOP and the PRIME buttons when the power is switched on. Release the buttons when the display displays "SFE". To enable the Motor Stall Detector press the UP-ARROW button and the display will display "On". To disable the Motor Stall Detector press the DOWN-ARROW button and the display will display "OFF". To exit this mode and store the current setting in non-volatile memory, press the RUN/STOP button.

The Motor Stall Detector uses a timer to determine if the cam shaft has stopped turning or if the refill switch is defective. The timer begins timing after the pump accelerates or decelerates to its set-point flow rate. If the Motor Stall Detector has been enabled, and the cam shaft stops turning or the refill switch stops operating, the fault will be detected between the time it takes to complete 1 to 2 pump cycles. A pump cycle is defined as the time it takes for the cam shaft to complete one complete revolution. One revolution of the cam shaft produces a delivery phase and a refill phase. Each specific flow rate has a corresponding cycle time. For a pump with an analytical (standard) 10 mL/min pump head, the cycle time is approximately: 30 seconds at 0.1 mL/min, 3 seconds at 1.00 mL/min, and 0.3 seconds at 10.00 mL/min.

The fault is canceled by using one of the following methods: (1) by pressing the RUN/STOP button on the front panel, (2) by sending a stop command "ST" via the serial communications port on the back panel, or (3) by connecting the PUMP-STOP input to COM on the back panel, or removing the connection between the PUMP-RUN input and COM if the PUMP-STOP input is permanently jumpered to COM on the back panel. Note: the PUMP-RUN, PUMP-STOP, and COM are an option and do not exist on the standard pump.

4.3 Gradient Operations

An optional gradient mixer may be ordered. It has approximately 2 mL of volume. It may be necessary to use the mixer at reduced flowrate to provide additional mixing to the three way prime purge valve on the front panel. See the chart below for actual traces of the gradient formation at different flowrates and pump heads



COMPARISON OF GRADIENT FORMATION WITH
DIFFERENT PUMP HEADS AND FLOW RATES

5. MAINTENANCE

Cleaning and minor repairs of the DPM-200 pump can be performed as outlined below.

Note: Lower than normal pressure, pressure variations, or leaks in the pumping system can all indicate possible problems with the piston seal, piston, or check valves. Piston seal replacement could be necessary after 1,000 hours of running time. See Section 5.2.3.

5.1 Filter Replacement

5.1.1 Inlet Filters

Inlet filters should be checked periodically to ensure that they are clean and not restricting flow. A restriction could cause cavitation and flow loss in the pump. Two problems that can plug an inlet filter are microbial growth and impure solvents. To prevent microbial growth, use at least 10-20% organic solvent in the mobile phase or add a growth-inhibiting compound. If you pump 100% water or an aqueous solution without any inhibitors, microbes will grow in the inlet filter over time, even if you make fresh solution every day. Always use well filtered, HPLC grade solvents for your mobile phase.

5.2 Changing Pump Heads

5.2.1 Removing the Pump Head

As a guide to pump head assembly, the standard pump heads are shown in Figures 5-1 through 5-4. All of the DPM-200 pump heads have a similar arrangement.

1. Turn OFF the power to the DPM-200 pump.
2. Remove the inlet line and filter from the mobile phase reservoir. Be careful not to damage the inlet filter or crimp the Teflon™ tubing.
3. Remove the inlet line from the inlet check valve.
4. Remove the outlet line from the outlet check valve.
5. Remove inlet and outlet self-flush check valves, or just the lines.
6. Momentarily turn ON the DPM-200 pump and quickly turn OFF the power upon hearing the refill stroke. This reduces the extension of the piston and decreases the possibility of piston breakage.
7. Unplug the power cord.
8. Carefully remove the two knurled nuts at the front of the pump heads.

CAUTION: Be careful not to break the piston when removing the pump head. Twisting the pump head can cause the piston to break.

- Carefully separate the pump head from the pump. Move the pump head straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the seal and seal backup washer from the piston if they did not stay in the pump head.
- Carefully separate the flush housing from the pump. Move the flush housing straight out from the pump and remove it from the piston. Be careful not to break or damage the piston. Also remove the self-flush seal from the piston if it did not stay in the flush housing.

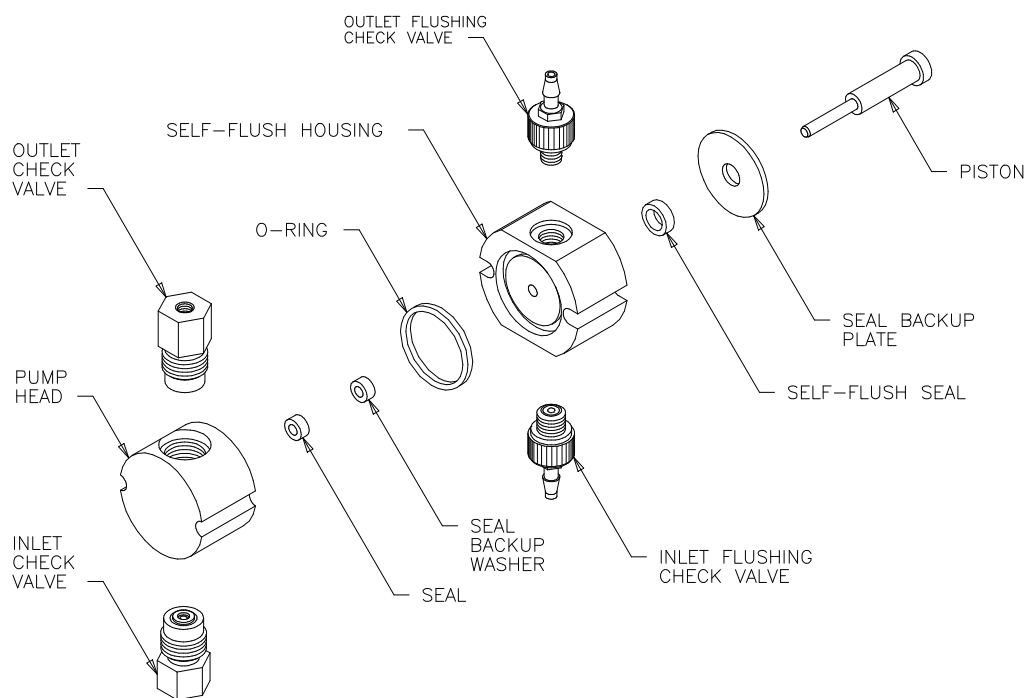


Figure 5-1. Stainless Steel Self-Flushing Pump Head Assembly

5.2.2 Cleaning the Pump Head Assembly

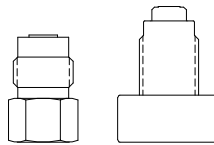
Note: If you choose to remove the piston seal or self-flush seals, you should have a new set on hand to install after cleaning. It is not recommended that you reinstall used piston or self-flush seals since they are likely to be scratched and damaged during removal and would not provide a reliable seal if reused. If you decide to remove the seals, use only the flanged end of the plastic seal removal tool supplied with the seal replacement kit and avoid scratching the sealing surface in the pump head. See Section 5.2.3 for seal replacement instructions.

- Inspect the piston seal cavity in the pump head. Remove any foreign material using a cotton swab, or equivalent, and avoid scratching the sealing surfaces. Repeat for the self-flush housing. Be sure no fibers from the cleaning swab remain in the components.

2. The pump head, check valves, and self-flush housing may be further cleaned using a laboratory grade detergent solution in an ultrasonic bath for at least 30 minutes, followed by rinsing for at least 10 minutes in distilled water. Be sure that all particles loosened by the above procedures have been removed from the components before re-assembly.

CAUTION: When cleaning check valves, be sure that the ball is not against the seat in the ultrasonic bath. This may destroy the precision matched sealing surface and the valve will not check.

WARNING: If removing the check valves, keep them in the orientation shown below the entire time they are not installed in the pump head. The assemblies may fall apart, parts may be lost, and they may not operate properly when re-assembled.



3. If the check valves have been removed, tighten the check valves on stainless steel pumps to 75 inch-pounds or enough to seal at maximum pressure.

Note: The inlet check valve has a larger opening (1/4"-28, flat-bottom seat) for the 1/8" inlet tubing; the outlet check valve has a smaller opening (#10-32, cone seat) for the 1/16" outlet tubing. The inlet check valve must be connected at the larger opening in the pump head. See Figure 5-5.

If the piston and flushing seals have been removed, insert new seals as described in Section 5.2.3, then continue with Section 5.2.5 to replace the pump head.

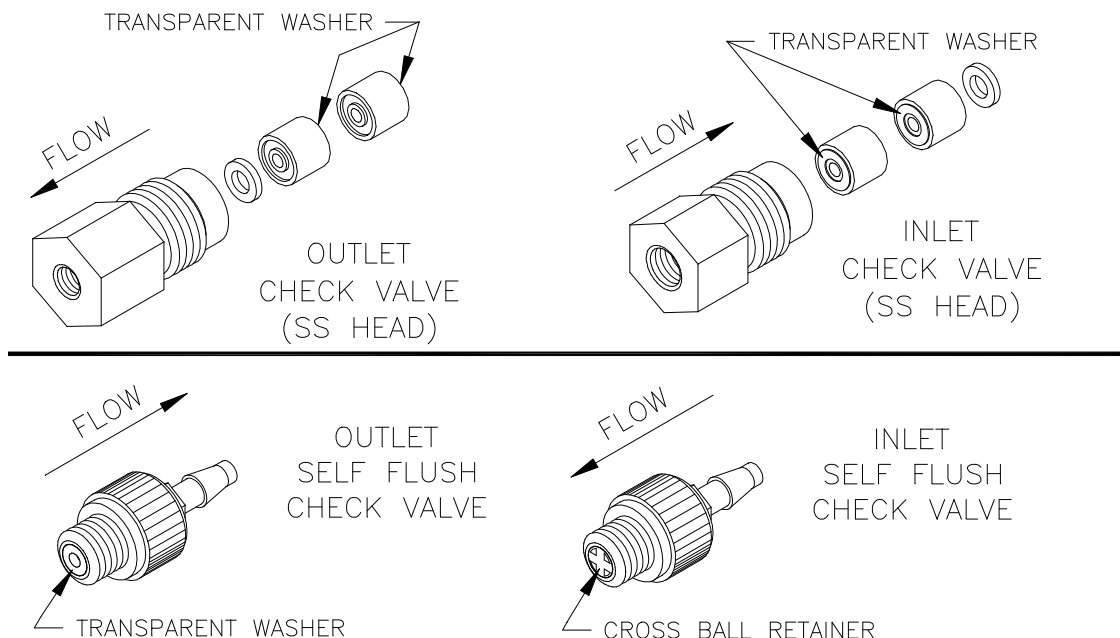


Figure 5-2. Check Valves

5.2.3 Replacing Piston Seals

Lower than normal pressure, pressure variations, and leaks in the pumping system can all indicate possible problems with the piston seal. Depending on the fluid or mobile phase used, piston seal replacement is often necessary after 1000 hours of running time.

Each replacement seal kit contains one seal, one backup washer, one self-flush seal, one non-flush guide bushing, two seal insertion/removal tools, and a pad to clean the piston when changing the seal.

5.2.3.1 Removing the Seals

1. Remove the pump head as described in Section 5.2.1.
2. Insert the flanged end of the seal insertion/removal tool into the seal cavity on the pump head. Tilt it slightly so that flange is under the seal and pull out the seal.

CAUTION: Using any other “tool” will scratch the finish.

3. Repeat the procedure for the low pressure seal in the flush housing.
4. Inspect, and if necessary, clean the pump head as described in Section 5.2.2.

5.2.3.2 Cleaning the Piston

1. Once the pump head and self-flush housing are removed, gently remove the seal back-up plate by using either a toothpick or small screwdriver in the slot on top of the pump housing.
2. Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up.
3. Use the scouring pad included in the seal replacement kit to clean the piston. Gently squeeze the piston within a folded section of the pad and rub the pad along the length of the piston. Rotate the piston frequently to assure the entire surface is scrubbed. Do not exert pressure perpendicular to the length of the piston, as this may cause the piston to break. After scouring, use a lint-free cloth, dampened with alcohol, to wipe the piston clean.
4. Grasp the metal base of the piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot.

5.2.3.3 Replacing the Seals

1. Place a high pressure replacement seal on the rod-shaped end of the seal insertion/removal tool so that the spring is visible when the seal is fully seated on the tool. Insert the tool into the pump head so that the open side of the seal enters first, facing the high pressure cavity of the pump head. Be careful to line up the seal with the cavity while inserting. Then withdraw the tool, leaving the seal in the pump head. When you look into the pump head cavity, only the polymer portion of the seal should be visible.
2. Place a self-flush replacement seal on the seal insertion/removal tool so that the spring in the seal is visible when the seal is on the tool. As in the previous step, insert the tool and seal into the seal cavity on the flushing housing, taking care to line up the seal with the cavity, and then withdraw the tool. When the seal is fully inserted only the polymer part of the seal will be visible in the seal cavity.
3. Place seal back-up washer over the high pressure seal. Place seal back-up plate back into pump housing if it was removed. Orientation is not important in these cases.
4. Attach the pump head as described in Section 5.2.5.
5. Condition the new seal as described in Section 5.3.

5.2.4 Changing the Piston

1. Remove the pump head as described in Section 5.2.3.
2. Grasp the metal base of the piston assembly so that you avoid exerting any side load on the sapphire rod, and remove the piston from the slot in the carrier by sliding it up.

3. Grasp the metal base of the replacement piston assembly, and insert it into the slot in the piston carrier until it bottoms in the slot.
4. Attach the pump head as described in Section 5.2.5.

5.2.5 Replacing the Pump Head

1. Make sure that the inlet valve is on the bottom and the outlet valve is on the top. Carefully align the self-flush housing and gently slide it into place on the pump. If misalignment with the piston occurs, gently push up on the piston holder.
2. Line up the pump head and carefully slide it into place. Be sure that the inlet valve is on the bottom and the outlet valve is on the top. Do not force the pump head into place.
3. Finger tighten both knurled nuts into place. To tighten firmly, alternately turn nuts 1/4 turn while gently wiggling the pump head to center it.
4. Re-attach the inlet and outlet lines. Reconnect the self-flush lines and fittings to the self-flush check valves. Change the flushing solution.

5.3 Conditioning New Seals

Note: Use only organic solvents to break-in new seals. Buffer solutions and salt solutions should never be used to break-in new seals.

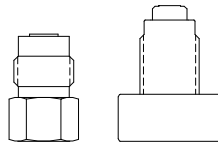
Using a restrictor coil or a suitable column, run the pump with a 50:50 solution of isopropanol (or methanol) and water for 30 minutes at the back pressure and flow rate listed under PHASE 1 below and according to the pump head type. Then run the pump for 15 minutes at a back pressure and flow rate listed under PHASE 2 below.

Pump Head Type	PHASE 1		PHASE 2	
	Pressure	Flow Rate	Pressure	Flow Rate
10 mL SS	2000 psi	<3 mL/min.	3000-4000 psi	3-4 mL/min.

5.4 Check Valve Cleaning and Replacement

Many check valve problems are the result of small particles interfering with the operation of the check valve. As a result, most problems can be solved by pumping a strong solution of liquid, laboratory grade detergent through the check valves at a rate of 1 mL/min for one hour. After washing with detergent, pump distilled water through the pump for fifteen minutes. Always direct the output directly to a waste beaker during cleaning. If this does not work, the check valve should be replaced.

WARNING: When removing the check valves, keep them in the orientation shown below the entire time they are not installed in the pump head. The assemblies may fall apart, parts may be lost, and they may not operate properly when re-assembled.



5.5 Pulse Damper Replacement

5.5.1 Removing the Pulse Damper

WARNING: There are potentially lethal voltages inside the DPM case. Disconnect the line cord before removing the cover. Never bypass the power grounds.

1. Make certain that the system has been depressurized. Unplug the power cord and remove the cover.
2. Disconnect the tubing from the pulse damper.
3. Disconnect the transducer from the circuit board.
4. Remove the four screws that secure the pulse damper from the underside of the chassis.
5. Remove the pulse damper.

5.5.2 Pulse Damper Refurbishing

Refurbishing the pulse damper is a time-consuming procedure. You may want to return the pulse damper to have it rebuilt. Do not attempt to refill or refurbish the pulse damper until you have a refurbishing kit. Instructions are furnished with the kit.

5.5.3 Pulse Damper Installation

1. Position the pulse damper, aligning it with the four mounting holes in the bottom of the cabinet. The pressure transducer should be pointed toward the rear of the cabinet.
2. From the underside of the pump cabinet, tighten the four screws to hold the pulse damper in place.
3. Connect the tubing from the pump head to the port at the rear of the pulse damper (i.e., toward the rear of the cabinet). Connect the line from the prime/purge valve to the other port, toward the front panel.
4. Connect the transducer's wire harness connector to power connectors and to the external pressure connector on the rear panel.
5. Replace the cover on the pump.

5.6 Cleaning the Pump

1. Disconnect the column inlet tube from the column.
2. Direct the column inlet tube (the tube from the injector outlet) to a waste beaker.
3. Set the flow rate to maximum.
4. Turn the injector to the INJECT position.
5. Pump 100% isopropanol through the pump and injector for 3 minutes.
6. Pump 100% filtered, distilled water through the pump and injector for 3 minutes.

WARNING: Use standard laboratory procedures and extreme care when handling strong acids and bases.

7. Pump a 20% nitric acid/water solution through the pump and injector for 3 minutes.
8. Flush the pump and injector with 100% filtered, distilled water for at least 3 minutes.
9. Pump 100% isopropanol through the pump and injector for 3 minutes.

The pump is now prepared for any mobile phase or short- or long-term shutdown.

5.7 Lubrication

The DPM-200 pumps have modest lubrication requirements. The bearings in the pump housing and piston carrier are permanently lubricated and require no maintenance. A small dab of a light grease such as Lubriplate 630-AA on the cam is the only recommended lubrication. Be sure not to get lubricant on the body of the piston carrier, as this can retard its movement and interfere with proper pumping.

Note: Keeping the interior of the pump free of dirt and dust will extend the pump's useful life.

5.8 Fuse Replacement

Three fuses protect the DPM-200 pumps. Two of the fuses are located in the power entry module (Corcom) at the rear of the cabinet and are in series with the AC input line. The other fuse is located on the printed circuit board and is in series with the 36 Vdc supply.

Troubleshooting the fuses is straightforward. If the power cord is plugged in and the ON/OFF power entry switch is ON and the fan does not run, check the two fuses in the power entry module. To gain access to these fuses, gently pry off the cover plate with a small flat-bladed screwdriver. Replace with fuses of the correct rating: 1 A slow-blo for 115 Vac pumps, or 1/2 A slow-blo for 230 Vac pumps.

If the above appears to function normally but the pump motor does not run, check the fuse located on the motor power circuit board. Replace it with a 5 A fast-blo fuse

5.9 Battery Replacement (*High Pressure Only*)

The battery provides power for the memory that holds the current pump configuration. If the pump is set at a flowrate other than 1.00 or 10.0 and the power is turned off, when the power is turned back on the flowrate should appear as it was set. If this flowrate does not appear the battery will need replaced.

1. Unplug the unit.
2. Remove the cover.
3. Turn the unit so that the control panel is to the right. The battery can be seen in the lower right corner of the circuit board. The battery is circular and has a positive pole mark (+) on the top. Gently pull it from its socket.
4. With the positive mark (+) up, gently slide the new battery into the battery socket. Be sure the battery is all the way into place. It must contact the base of the battery socket.
5. Replace the cover to the unit.
6. Plug the unit back in.

6. TROUBLESHOOTING

Quick Guide to Problem Solving

You Notice	This May Mean	Possible Cause	You Should
<ol style="list-style-type: none"> 1. Uneven pressure trace. 2. Pressure drops. 3. Pump shuts OFF. 4. No flow out the outlet check valve. 	<ol style="list-style-type: none"> 1. Bubble in check valve. 2. Leaks in system. 3. Dirty check valve. 4. Bad check valve. 	<ol style="list-style-type: none"> 1. Solvent not properly degassed. 2. Fittings are not tight. 3. Mobile phase not properly filtered. 4. Particles from worn piston seal caught in check valve. 5. Plugged inlet filter. 	<ol style="list-style-type: none"> 1. Check to be certain that mobile phase is properly degassed. 2. Check connections for leaks by tightening fittings. 3. Prime the system directly from the outlet check valve. 4. Clean or replace the check valves. See Section 5.4. 5. Clean or replace inlet filter. See Section 5.1.1.
<ol style="list-style-type: none"> 1. Uneven pressure trace. 2. Pressure drops. 3. Fluid between the pump head and the chassis. 	<ol style="list-style-type: none"> 1. Leaks in system. 2. The piston seal(s) are worn. 	<ol style="list-style-type: none"> 1. Fittings not tight. 2. Long usage time since last seal change. 3. Salt deposits on seal (especially if buffered aqueous mobile phases are used without the self-flush head.) 	<ol style="list-style-type: none"> 1. Check all connections for leaks. 2. Replace piston seal. See Sections 5.2 and 5.3. 3. Check the piston for salt deposits. Clean as necessary. See Section 5.2.4.
Pump makes a loud clanging or slapping noise (intermittent contact with cam).	Piston carrier is catching in piston guide.	<ol style="list-style-type: none"> 1. Cap nut screws on the pump head are loose. 2. Seal(s) are worn. 3. Piston guide is worn 4. Salt build-up on piston carrier from use of buffers. 5. Excess lubricant on piston carrier. 	<ol style="list-style-type: none"> 1. Check cap nut screws on pump head. Tighten if necessary. 2. Replace seals. 3. Replace piston guide and seals. See Sections 5.2 and 5.3. 4. Consider changing to a self-flushing pump head if using buffers. 5. Clean excess lubricant and dirt off piston carrier. See Section 5.7.
Blue dye in mobile phase.	Pulse damper diaphragm has burst.	Sudden pressure drop when purging system.	Replace pulse damper. See Section 5.5.
Pump runs for 50 pump strokes, then shuts down.	Lower pressure limit is activating.	<ol style="list-style-type: none"> 1. Mobile phase is not properly filtered. 2. Particles from worn seal trapped in the system (e.g., tubing, filters, injection valve, column inlet). 	<ol style="list-style-type: none"> 1. Check to be certain the low pressure limit is set to 0 psi. 2. Only increase the low pressure limit after the pump attains operating pressure. 3. Contact service technician.
<ol style="list-style-type: none"> 1. Pump shuts down after run is called even with no column connected. 2. Pump runs to maximum pressure and shuts down. 	Clog in fluid system.		<ol style="list-style-type: none"> 1. Remove and clean both the inlet and bulkhead filters. See Section 5.2. 2. If the problem persists, remove tubing from system one piece at a time until you find the clogged piece. Most clogs occur outside the pump itself.
No power when pump turned ON. Fan does not run.	Blown fuses in the power entry module.	<ol style="list-style-type: none"> 1. Power surge. 2. Internal short. 	<ol style="list-style-type: none"> 1. Replace only with the appropriate fuses (1A for 100-120 Vac or 1/2A for 220-240 Vac). 2. Contact service technician if problem persists.
Front panel appears OK but pump motor does not run.	Blown fuse on the motor power circuit board.	<ol style="list-style-type: none"> 1. Power surge. 2. Internal short. 	<ol style="list-style-type: none"> 1. Replace only with the appropriate fuse . 2. Contact service technician if problem persists.

Fittings or components leak.	You cannot force the parts with interference to seal by brute force tightening.	<ol style="list-style-type: none"> 1. Film of fluid between surfaces. 2. Salt crystals between surfaces. 3. Scratches in mating surfaces. 	<ol style="list-style-type: none"> 1. Clean and dry mating surfaces. 2. If scratched, replace defective part. 3. Replace scratched part.
Self-flush heads leak flush solution.	Flush area not sealed.	<ol style="list-style-type: none"> 1. Large (Size 016) O-ring is flattened and no longer seals. 2. Head not sufficiently tightened. 3. Scratches in mating surfaces. 4. Leaky self-flush seal. 	<ol style="list-style-type: none"> 1. Replace O-ring. 2. Tighten head. 3. Replace leaky parts.

7. ACCESSORIES, REPLACEMENT AND SPARE PARTS

	PART NO.	NOTES
PUMP		
Check Valve Capsules, SS, Replacement 2pc/pkg	025-0007	RSP
Check Valve Capsules, PEEK, Replacement 2pc/pkg	025-0007	RSP
Piston assembly, 10 mL, Series I	025-0010	
Piston seal kit, 10 mL, Series I	025-0006	
Solvent Inlet filter, 20 micron	025-0067	
Replacement filter elements for 025-0067 10pc/pkg	025-0029	RSP
TUBING AND OTHER FITTINGS		
Tubing, SS, 1/16" OD, 0.010" ID 5ft	025-0173	
Tubing, SS, 1/16" OD, 0.030" ID 5ft	025-0172	
Tubing, PEEK, 1/16" OD, 0.010" ID 5ft	025-0061	
Tubing, PEEK, 1/16" OD, 0.030" ID 5ft	025-0049	
Tubing, TFE Teflon, 1/8" OD, .085" ID (Inlet Filter) 5ft	025-0053	
Nut, 10-32, 1/16" SS (for outlet/PPV tubing) 10pc/pkg	025-0012	RSP
Ferrule, 1/16" SS (for outlet/PPV tubing) 10pc/pkg	025-0013	RSP
Nut, 10-32, 1/16" PEEK (for outlet tubing/bulkhead/column/Flowcell) 10pc/pkg	025-0012	RSP
Ferrule, double-end, 1/16" PEEK (for outlet tubing/bulkhead/column/Flowcell) 10pc/pkg	025-0013	RSP
Plug, column #10-32, PEEK 10pc/pkg	025-0062	
Plug, column 1/4"-28, PEEK 10pc/pkg	025-0063	
Nut, flangeless, 1/8" ID, PEEK (Inlet Filter) 10pc/pkg	025-0068	
Ferrule, Flangeless, Tefzel, 1/8" ID (Inlet Filter) 10pc/pkg	025-0052	RSP
PULSE DAMPER REPAIR KIT	025-0009	
PRIME/PURGE VALVE SEAL KIT	Contact Dealer	
INJECTION VALVE (Outlet Bulkhead)		
Ferrule, C1 ,Injection Valve (Outlet Bulkhead), SS 10pc/pkg	025-0041	
Nut, 1/16" C1, Injection Valve (Outlet Bulkhead), SS 10pc/pkg	025-0043	
Ferrule, C1 ,Injection Valve, PEEK 10pc/pkg	025-0040	
Nut, 1/16" C1, Injection Valve, PEEK 10pc/pkg	025-0042	
Sample loop, 20 ul, Replacement, C1, Injection Valve, SS _i	025-0164	
Sample loop, 20 ul, Replacement, C1, Injection Valve, PEEK _i	025-0064	
Rotor, Replacement, C1, Injection Valve, SS	025-0103-1	
Rotor, Replacement, C1, Injection Valve, PEEK	025-0103	
Stator, Replacement, C1, Injection Valve, SS or PEEK	Contact Dealer	
Socket Wrench Fittings Tool, C1 Injection Valve	025-0044	
i Other size loops available	Contact Dealer	
FUSES:		
Fuse, Pump, 5 x 20 mm, FAST, 250V, 5A 20pc/pkg	025-0055	RSP
Fuse, Corcom 115V, 5 x 20 mm, FAST, 250V, 1.0A 20pc/pkg	025-0056	RSP
Fuse, Corcom 230V, 5 x 20 mm, FAST, 250V, 0.5A 20pc/pkg	025-0057	RSP
POWER CORD, N. AMERICA, NEMA 5-15/IEC320	025-0091	

POWER CORD, International, CEE 7STD/IEC 320

025-0108

CABLES

Cable Adapter RJ-11 to DB25

PART NO.

025-0058

NOTES

Cable Adapter RJ-11 to DB9

025-0059

Modular RS-232 Cable

025-0060

PC BOARDS

Contact Dealer Tech

**REPLACEMENT ACCESSORY KIT (CABLE, TOOLS, CLIPS
FUSES)**

025-0122

OPERATOR'S MANUAL (Paper Copy)

050-0020

LEGEND: RSP = Recommended spare part

Tech = Maintenance Technician Recommended

APPENDIX A

A.1 Rear Panel Serial Communications Port

An RS-232C modular jack is provided on the back panel. A computer, with appropriate software, can be used as a remote controlling device for pump operation via this connection.

A.1.1 Hardware Implementation

The REMOTE INPUT serial communications port is configured for 9600 baud, 8 data bits, 1 stop bit, and no parity. The connector is a standard RJ-11 modular telephone type jack. When looking at the connector on the rear panel of the pump, pin 1 is at the top and pin 6 is at the bottom. The pin-out is:

<u>Pin</u>	<u>Function</u>
1, 6	Ground
2	DSR (Handshaking input to pump)
3	RXD (Serial data input to pump)
4	TXD (Serial data output from pump)
5	DTR (Handshaking output from pump)

Special wiring considerations: Use the following chart for interfacing the pump's serial communications port to either a 25-pin or a 9-pin COM port on an IBM-PC type computer.

<u>Pump (RJ11)</u>	<u>Signal</u>	<u>IBM (DB25)^a</u>	<u>IBM (DB9)^b</u>
1, 6	Ground	7	5
2	DSR	20	4
3	RXD	2	3
4	TXD	3	2
5	DTR	6	6

^a Jumper pins 4, 5, and 8 on DB25.
^b Jumper pins 1, 7, and 8 on DB9.

Part Description	Part Number
Modular Cable	consult Factory
Adapter RJ-11 to DB9	consult Factory
Adapter RJ-11 to DB-25	consult Factory

A.1.2 Hand-Shaking

The pump uses hardware handshaking. The pump will not transmit on the TXD output if the DSR input is at a low logic level. And, the pump will not receive on the RXD input when the DTR output is at a low logic level. A low logic level is -3.0 to -15 volts and a high logic level is 3.0 to 15 volts.

A.1.3 Command Interpreter

The pump's high-level command interpreter receives and responds to command packets. The pump will not send a message except when prompted, and it will send a response to every valid command as described below. The response to an invalid command is "Er/".

Each command is characterized by a unique two-letter command code, and only one command can be issued per line. Case is not important; that is, the command codes "PR" "Pr" "pR" and "pr" are all equivalent. Response strings sent by the pump are terminated by the "/" character.

If the pump's response is "Er/", send a "#" to clear any characters which may be remaining in the command buffer. The pump will automatically clear all characters in the command buffer after one second elapses from the time at which the last character of an incomplete command was sent.

The command packets are as follows:

Command	Response	Comments
RU	OK/	Sets the pump to the RUN state.
ST	OK/	Sets the pump to the STOP state.
FLxxx	OK/	Sets the flowrate to x.xx or xx.x mL/min where the range is fixed for the pump head size, i.e., for 0.01 to 9.99 mL/min xxx = 001 to 999
PR	OK,x/ (x, xx, or xxx)	Reads the pump's current pressure, where: x, xx, or xxx = Current pressure in PSI
CC	OK,x,yyy.y/ (x, xx, or xxx) (y.y, yy.y, or yyy.y)	Reads the pump's current pressure and flowrate, where: x, xx, or xxx = Current pressure in PSI y.y, yy.y, or yyy.y = Flowrate in mL/min
CS	OK,xxx.x,y,z,PSI,w,v,u/ (x.x, xx.x, or xxx.x) (y, yy, or yyy) (z, zz, or zzz)	Reads the current pump setup, where: x.x, xx.x, or xxx.x = Flowrate in mL/min y, yy, or yyy = Upper pressure limit z, zz, or zzz = Lower pressure limit PSI = Units (PSI, ATM, MPA, BAR, or KGC) w = Pump head size (0 = standard, 1 = macro) v = Run status (0 = stopped, 1 = running) u = Pressure Board present = 0; otherwise 1
ID	OK,vx.xx SR3P firmware/	Identifies the pump type and EPROM revision x.xx
UPxxxx	OK/	Sets the upper pressure limit in PSI. The maximum value is 500; the minimum value is the lower limit plus 10. The value must be expressed as four digits, i.e., for 400 PSI xxxx = 0400.
LPxxxx	OK/	Sets the lower pressure limit in PSI. The maximum value for xxxx is the current upper pressure limit setting minus 10; the minimum value is 0. The value must be expressed as four digits, i.e., for 50 PSI xxxx = 0050.

SF	OK/	Puts the pump in fault mode. Turns on the FAULT LED and stops the pump immediately.
RF	OK,x,y,z/	Reads the fault status, where: x = Motor stall fault (0 = no, 1 = yes) y = Upper pressure limit fault (0 = no, 1 = yes) z = Lower pressure limit fault (0 = no, 1 = yes)
KD	OK/	Disables the keypad. (Default status at power-up is enabled.)
KE	OK/	Enables the keypad.
PCxx	OK/	Sets the pressure compensation value, where xx = the operating pressure (in PSI divided by 100), i.e., for 0 PSI xx = 00, for 0500 PSI xx = 05.
RC	OK,x/	Reads the pressure compensation value in hundreds of PSI, i.e., for 0 PSI x = 0, for 0500 PSI x = 5.
PI	OK,a.aa,b,c,d,e,f,g,h,i,j,k,l, m,n,o,p,q/ (a.a, aa.a, or aaa.a) (c or cc)	Reads the current pump setup, where: a.a, aa.a, or aaa.a = Flowrate in mL/min b = Run status (0 = stopped, 1 = running) c or cc = Pressure compensation d = Pump head type (see RH command) e = Pressure Board present = 0; otherwise 1 f = External control mode (0 = frequency, 1 = voltage) g = 1 if pump started and frequency controlled, else 0 h = 1 if pump started and voltage controlled, else 0 i = Upper pressure limit fault (0 = no, 1 = yes) j = Lower pressure limit fault (0 = no, 1 = yes) k = Priming (0 = no, 1 = yes) l = Keypad lockout (0 = no, 1 = yes) m = PUMP-RUN input (0 = inactive, 1 = active) n = PUMP-STOP input (0 = inactive, 1 = active) o = ENABLE IN input (0 = inactive, 1 = active) p = Always 0 q = Motor stall fault (0 = no, 1 = yes)
RE	OK/	Resets the pump configuration to its default power-up state.
#	(no response)	Clears all characters from the command buffer.

Operating Instructions
FRONT LOADING THROUGH-THE-HANDLE
DIV-1 INJECTION VALVE
Part Number DS 025-0101 PEEK / 025-0101-1 SS
(DLC HPLC Systems)

Contents

<u>Section</u>	<u>Contents</u>
1	Introduction
2	Specifications
3	Unpacking and Orientation
4	Installation
5	Operation - Sample Loading
6	Disassembly and Reassembly
7	Replacement and Spare Parts
8	Warranty

Installation, Use, and Maintenance

Section 1.0 Introduction

The Model DIV-1 valve is a 6-port front loading, through-the-handle loop injector. Direct syringe loading permits "partial fill injection" where the volume injected into the loop is determined by the syringe, or "full loop injection" where the volume injected is determined by the volume of the sample loop. The design of the injector port prevents contact (scratching) between the syringe needle and the rotor and stator faces (sealing surfaces). The DIV-1 valve has a position sensor built in to the body to provide event mark capability (contact closure signal when "injected").

Section 2.0 Specifications

Type:	Analytical
Port Diameter:	0.016" (0.40mm)
Wetted Materials:	PEEK Version: Rotor - Valcon E; Stator - PAEK; Fittings - PEEK SS Version: Rotor - Valcon H; Stator - N60 SS; Fittings - SS
Standard Pressure:	5000 psi (liquid)
Standard Temperature:	50°C
External Loop:	20µl
Ports:	6
Actuation:	Manual
Fittings:	1/16", 10-32
Valve Lifetime:	Typically exceeds 50,000 cycles

3.0 Unpacking and Orientation

3.1 Unpacking - Packing List [If your valve was factory installed, proceed to Section 3.2.]

025-0101	DIV-1 Injection Valve with end cap, 20µl loop and fittings, fittings 1/16", 10-32 Nut and Ferrules x 4, mounting hardware (2 socket head cap screws (SHCS) and 2 split lockwashers)
250-0065	Hole Plug, 3/8", Nylon, Black
310-0013	Tubing, Waste, TFE, 24" (Port 6)

540-0003 5/64" Hex Wrench (for C2 handle setscrew)

Remove the red end cap and use it to cover the valve being replaced.

3.2 Orientation

The DIV-1 Valve is made up of three primary parts (Figure 1):

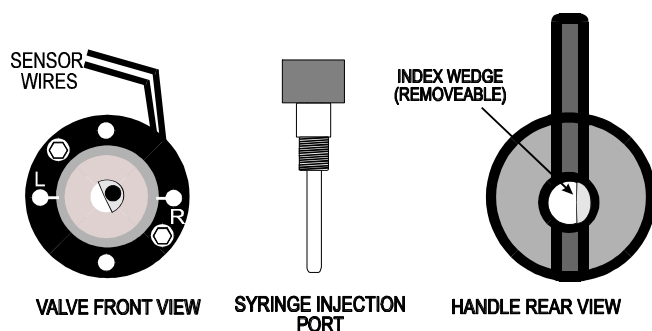


Figure 1

The Valve Body
The Valve Handle; and
The Syringe Injection Port.

The flow diagram is shown at Figure 2. The sample is introduced through the syringe injection port denoted by the letter P on the flow diagram.

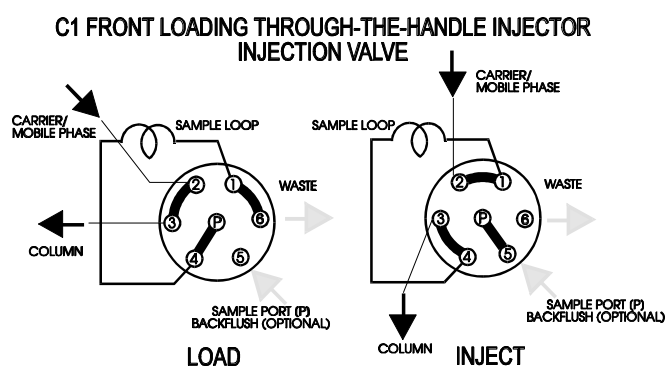


Figure 2

4.0 Installation

[If your valve was factory installed, proceed to Section 4.3.]

4.1 Removing Old C2 Injection Valve

4.1.1 Standard C2 Valve

- 4.1.1.1 Remove the fittings from all the tubing and set them aside.
- 4.1.1.2 Use a 5/64" hex wrench to remove the handle from the C2 valve rotor. Use a 9/64" hex wrench (D2 Lamp Strap) to unscrew the 8-32 socket head cap screws (SHCS) on the front panel.
- 4.1.1.3 Remove the valve and cover the fitting end of the valve with the red plastic cap from the new valve. Reattach the 8-32 screw and the handle. Store the valve and fittings in a clean area.
- 4.1.1.4 Remove the SAMPLE INJECTION PORT by unscrewing the plastic nut on the front panel. Store the assembly with the old valve for future use.

4.1.2 C2 Valve with Position Sensor

- 4.1.2.1 Remove the fittings from all the tubing and set them aside.
- 4.1.2.2 Disconnect the connector for the position sensor wires (black and white) from the event mark header located on the rear of the front panel display PCB (**for DLC-10 see special instructions attached**).
- 4.1.2.3 Use a 5/64" hex wrench to remove the handle from the C2 valve rotor. Use a 9/64" hex wrench (D2 Lamp Strap) to unscrew the black 8-32 socket head cap screws (SHCS) from the position sensor on the front panel. Keep the black SHCS and all lockwashers with the C2 position sensor and valve.
- 4.1.2.4 Remove the valve and cover the fitting end of the valve with the red plastic cap from the new valve. Reattach the 8-32 screws, lockwashers, position sensor, and the handle to the valve. Store the valve and fittings in a clean area.
- 4.1.2.5 Remove the SAMPLE INJECTION PORT by unscrewing the plastic nut on the front panel. Store the assembly with the old valve for future use.

4.2 Mounting the DIV-1 Valve to the Panel

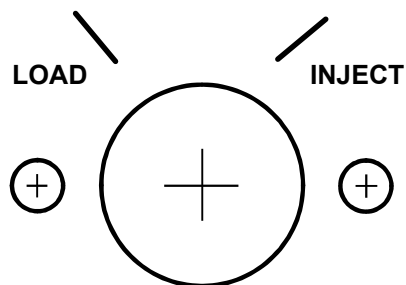


Figure 3
 DLC Mounting Holes

Note the mounting holes in **Figure 3**.

4.2.1 Unscrew the knurled Syringe Injection Port and remove the knob by pulling it outward, away from the valve body.

4.2.2 Put the two screws with lockwashers through the small holes in the panel and screw them into the valve to secure it to the panel. Ensure the valve is orientated as shown in Figure 1.

4.2.3 Push the knob back onto the valve and screw the Syringe Injection Port part way into the knob. Insert a syringe into the injection port and continue screwing until the syringe feels snug. Do not overtighten.

4.2.4 Insert the 3/8" black nylon hole plug into the SAMPLE INJECTION PORT.

4.2.5 Connect the position sensor wire 2 pin connector to the Signal Processor (SP) PCB event mark header J8. The orientation of the connector does not matter. Request special instructions for units that do not have the SP header.

4.3 Connecting Fittings

WARNING: DO NOT USE Rheodyne model fittings. The difference in pilot depth yields unswept volume.

4.3.1 Connect the sample loop to the ports 1 and 4. The pulse damper is connected at port 2 and the column at port 3 as shown in Figure 4.

4.3.2 In order for the syringe port to be backflushed with mobile phase, a piece of PTFE tubing ("Flush") should be connected from port 5. Place it in a small bottle of mobile phase. If the port will vent to atmosphere, ensure that the Teflon flush tube is clear and not pinched.

4.3.3 Connect a piece of Teflon tubing ("Waste") to port 6 to carry excess sample to a containment vessel appropriate to the sample.

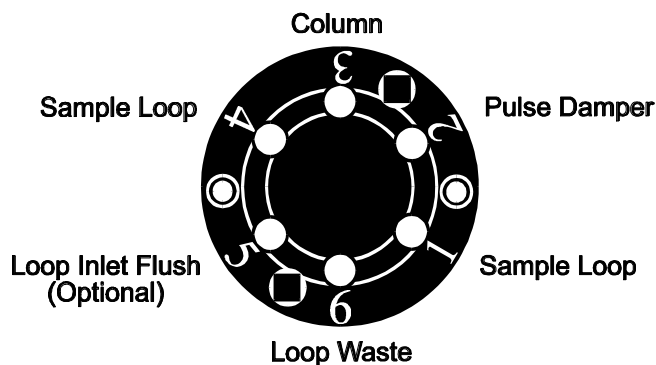


Figure 4
 Injection Valve Rear View

5.0 Operation - Sample Loading



Figure 5

The loop on a Model **DIV-1** front-loading valve is loaded by using a syringe with a 2" or longer flat end #22 gauge needle (0.028" O.D.). The loop may be either partially filled with a sample volume as low as 0.1 μ l, or completely filled, up to the capacity of the loop. When the handle is turned all the way counterclockwise, the valve is in the **LOAD** position. Turning the handle clockwise moves it to the **INJECT** position. (Figure 5.)

5.1 Partial-Fill Method (and Flushing)

On the first sample insertion, be certain that an extra 0.1 μ l is loaded into the loop to allow for the small amount of sample which will remain between the needle and the stator. Before a different sample is loaded, this remaining sample must be cleared out by following a simple procedure:

5.1.1 With the valve still in the **INJECT** position, draw at least 1 μ l of solution back into the syringe. (Use air if no flush solvents are connected to port 5.)

5.1.2 Withdraw the syringe from the valve and turn the valve handle back to the **LOAD** position, ready for the next injection. This effectively clears out the remaining sample and flushes the space between the needle and the stator with mobile phase. Since there is no opportunity for contamination, there is no need to flush the needle port before proceeding with the next sample injection. If no flush solvent is provided, a minute amount of sample may remain on the fill port walls but will not usually be a problem. An alternate method of effectively cleaning the remaining sample is to use a syringe to flush out the syringe port with either mobile phase or a solvent while the valve is in the **INJECT** position. A few microliters is often sufficient.

5.2 Partial-Filling with the "Bubble Method"

It has been demonstrated that laminar flow mixing of the sample and mobile phase in the loop can be controlled by the introduction of an air bubble between the sample and the mobile phase. The reliability of this technique arises from the excellent seal formed around the needle during loading. To use the "bubble method" when the same sample will be re-injected:

5.2.1 Physically remove the syringe from the syringe port while the valve is in the **INJECT** position to allow the port to be exposed to the atmosphere.

5.2.2 Turn the handle to the **LOAD** position and then reinsert the syringe into the syringe port. This will cause a 1 μ l bubble to develop.

5.2.3 To use the "bubble method" with a new sample: Flush the syringe port first, and then follow Steps 1 and 2 as above.

5.3 Complete Loop Filling

On the first insertion, use a syringe that exceeds the volume of the loop so fluid will overflow into the waste tube (2-3 loop volumes) in port 6. Removing the syringe prior to loading the loop will form an air bubble, insuring that there can be no mixing of the sample with the mobile phase. If this is done, laminar flow distortion is minimized and only a slight excess of sample will be required to overflow the loop. Before a different sample is loaded, this remaining sample may be cleared out by following a simple procedure:

5.3.1 Place a syringe filled with the new sample material into the syringe port while the valve is still in the INJECT position.

5.3.2 Purge the old sample out of the port hole by inserting $>.1 \mu\text{l}$ of new sample. The plug of air resulting from the simple insertion of the needle into the valve port will force any old sample out of the port; however; a residue may adhere to the walls.

5.3.3 Move the handle to the LOAD position and inject the exact amount of sample. (An air bubble will not be present using this method unless the syringe is removed after clearing the old sample from the port and then replaced after the valve is turned to its LOAD position.)

6.0 Maintenance

Cleaning a valve can often be accomplished by flushing all the lines with appropriate solvents. Do not disassemble the valve unless system malfunction is definitely isolated to the valve.

6.1 Disassembly (Refer to Figure 6)

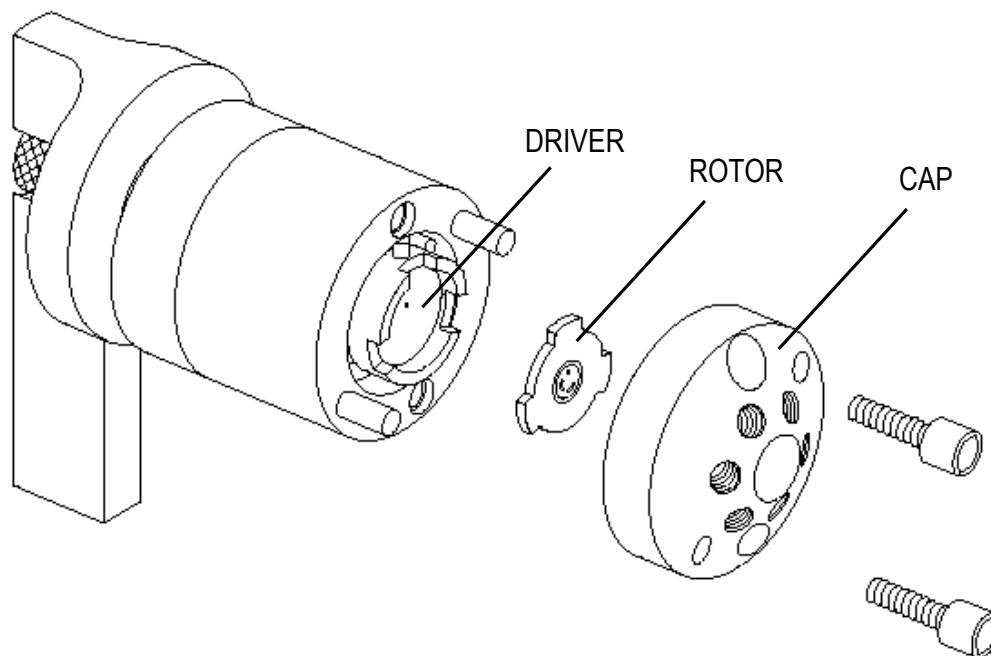


Figure 6 DIV-1 Valve Exploded View

- 6.1.1 Use a 9/64" hex driver to remove the socket head screws that secure the cap on the valve.
- 6.1.2 To ensure that the sealing surface of the cap is not damaged, rest it on its outer face. Or, if the tubing is still connected, leave it suspended by the tubing.
- 6.1.3 With your fingers or a small tool, gently pry the rotor away from the driver.
- 6.1.4 Examine the rotor sealing surface for scratches. If scratches are visible to the naked eye, the rotor must be replaced. If no scratches are visible, clean all the parts thoroughly with an appropriate solvent, taking care that no surfaces get scratched. (The most common problem in HPLC is the formation of buffer crystals, which are usually water-soluble.) It is not necessary to dry the rotor.

6.2 Reassembly

- 6.2.1 Replace the rotor in the driver, making sure that the rotor-sealing surface with its engraved flow passages is facing out. The pattern is asymmetrical to prevent improper placement.
- 6.2.2 Replace the cap. Insert the two socket head screws and tighten them gently until both are snug. Do not overtighten them – the screws simply hold the assembly together and do not affect the sealing force, which is automatically set as the screws close the cap against the valve body.
- 6.2.3 Test the valve by pressurizing the system. If it doesn't hold pressure, the valve should be returned to your dealer for repair.

7.0 Replacement and Spare Parts

<u>PART</u>	<u>PART NO.</u>
Replacement Rotor, PEEK	025-0103
Replacement Rotor, SS	025-0103-1
Replacement Stator, PEEK or SS	Contact Dealer
Sample Injection Loop 20 µl, PEEK	025-0064
Sample Injection Loop 20 µl, SS	025-0164

8.0 Warranty

- No other warranty exists, expressed or implied, except as shown here and in D-Star Instruments' Conditions of Sale.
- Fitness to a particular application must be determined by the user.
- Parts & labor are warranted for one (1) year, provided the unit is not damaged by improper use, abuse or by chemical spill.
- Request a Return Authorization (RA) before returning any parts.
- Return defective unit to Dealer/Representative for repair. Clean/sterilize all components prior to shipment. No parts will be accepted which present a health or safety hazard to service personnel. Repaired unit will be returned via parcel service or mail.

These instructions were adapted from VICI Technical Note 802, Rev 05/28/96, ©Valco Instruments Co., Inc. 1992, 1994, (1996). Used with Permission J.G. 10/15/97.

MODEL DVW-10 VARIABLE WAVELENGTH UV-VIS DETECTOR

OPERATOR'S MANUAL



TABLE OF CONTENTS

	page
PART A: GENERAL	
1.0 Introduction.....	1
1.1 General	1
1.2 Layout	1
1.3 Graphics Conventions	1
1.4 DVW-10 Features.....	1
1.5 Common Applications.....	2
1.6 Warranty.....	2
1.7 DVW-10 Detector Specifications	3
2.0 System Orientation	4
2.1 Front Panel.....	4
2.2 Rear Panel	5
2.3 Side Panel.....	6
3.0 Safety	7
4.0 Installation	8
4.1 Packing List.....	8
4.2 Installation	8
4.2.1 General	8
4.2.2 Flowcell	9
4.2.3 Power Cord and Recorder/Integrator Cable	9
4.2.4 Problems.....	9
PART B: DETECTOR OPERATION	
5.0 Description of the DVW-10 Detector.....	10
5.1 Introduction.....	10
5.2 Operating Principles	10
5.3 Optical System	10
5.4 Operating Wavelengths.....	11
5.5 Flow-Through Fluid Cells (Flowcells).....	11
5.6 Limits to Performance.....	11
6.0 Installation and Operation	13
6.1 Orientation of the Detector	13
6.2 Flowcell and Tubing Connections	14
6.3 AC Power	14
6.4 Controls.....	14
6.5 Indicators.....	16
6.5.1 Range Selection Status.....	16
6.5.2 Absorbance Display.....	16
6.5.3 Wavelength Display.....	16
6.6 Input/Output Connectors	17
6.7 Recorder/Integrator Cable	17
6.8 Power-on Warm-up	17

6.9	Wavelength	18
6.9.1	Wavelength Lamp	18
6.9.2	Wavelength Selection.....	18
6.9.3	Wavelength Scrolling.....	18
6.10	Performing Chromatography Runs	19
6.10.1	Test Runs.....	19
6.10.2	Record Runs	19
6.11	Shutting the Unit Down	19
7.0	Remote Control (Accessory Cable).....	20
7.1	External Control.....	20
7.2	Remote Operation of the DVW-10.....	20
7.2.1	Lamp On / Lamp Off Control	20
7.2.2	Photocell Energy - Sample & Reference	20
7.2.3	Wavelength UP/Wavelength DOWN	20
7.2.4	Offset	21
7.2.5	Event Mark	21
7.2.6	Autozero.....	21
7.3	Remote Control Technical Details	21
7.3.1	Recorder/Integrator and Remote Connectors	21
7.3.2	Alternate uses of Pins/Wires	22
7.3.3	Technical Discussion.....	22
7.4	Circuit Protection	22
8.0	Safety and Maintenance	23
8.1	Safety	23
8.1.1	General	23
8.1.2	Electrical Shock Hazard	23
8.1.3	UV Radiation	24
8.2	Maintenance.....	25
8.2.1	Electrical.....	25
8.2.2	Chemical Cleanup	25
8.2.3	UV Radiation	26
8.2.4	Flowcells	26
8.2.4.1	Installing/Removing a Flowcell.....	26
8.2.4.2	Cleaning Flowcells.....	27
8.2.5	Replacing/Changing the D2 Lamp (195-360 nm).....	27
8.2.6	Replacing/Changing the W Lamp (360-800 nm).....	28
8.2.7	Calibration	29
8.2.8	Replaceable Parts	30
8.2.9	Tubing Preparation.....	30
8.2.9.1	Cutting and Finishing	30
8.2.9.2	Cleaning	30
8.2.10	Fitting Assembly	30

PART C: MISCELLANEOUS

9.0 Power Management.....	32
9.1 General	32
9.1.1 AC Power	32
9.1.2 DC Power Supply	32
9.2 Power Entry Module	32
9.2.1 Description	32
9.2.2 Changing Voltages (Conversion).....	33
9.3 Fuse Replacement	33
10.0 Preparation for Storage or Shipping.....	34
10.1 Isopropanol Flush	34
10.2 Storage.....	34
10.3 Shipping	34
11.0 Troubleshooting Guide.....	35
12.0 Accessories, Replacement and Spare Parts	36
13.0 Trademarks	37
APPENDIX A - Flowcell Assemblies	
A-1 Analytical: 7mm, 10µl, PEEK	A - 1
A-2	A - ___
A-3	A - ___
APPENDIX B - Instrument Standardization Log.....	B - 1
APPENDIX C - Certificates of Conformity.....	C - 1

PART A: GENERAL

Section 1.0 Introduction

1.1 General This manual contains information for the installation, operation, and minor maintenance of the DVW-10 Variable Wavelength UV-VIS Detector.

1.2 Layout This manual is divided as follows:

Part A - Sections 1.0 through 4.0 contain instrument information and safety warnings.

Part B - Sections 5.0 through 8.0 contain information on operation of the variable wavelength detector.

Part C - Sections 9.0 through 13.0 contain information on power management, troubleshooting, shipping and storage, and accessories, spare and replacement parts.

1.3 Graphics Conventions The following symbols and graphics are used in this manual to alert the operator to essential information.



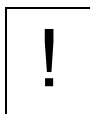
Electrical Shock Hazard - Information is necessary to prevent operator injury due to electrical shock.



Caution - Information is necessary to prevent operator injury or equipment damage due to mechanical or chemical condition(s).

Shadow Box

or



NOTE - Information concerning operator required action or for optimizing instrument performance.

1.4 DVW-10 Features

The DVW-10 system is the first in a new generation of variable wavelength detectors. Some of the key features of the detector are:

- Complete UV-Vis operation from 195 nm to 800 nm.
- Double beam optics.
- All wettable components constructed of PEEK™, Teflon™, Tefzel™, and quartz for total compatibility with biochemical systems.
- Flowcells available for analytical, industrial prep work, and biocompatible systems.
- Eight absorbance ranges via scrolling up and down controls.
- Sensitivity levels available for over 95% of all applications.
- Digital read-out of absorbance and wavelength.

- Analog outputs for absorbance and wavelength available at rear of instrument.
- Absorbance and wavelength remote control available via rear connector contact closures.
- Scrolling selection of wavelength.
- Voltage selection - simple, external user access.

1.5 Common Applications

The DVW-10 Series was designed to offer absorbance detection of high performance liquid chromatography (HPLC) separations at the best price/performance ratio. The unit can be used for the most complex analyses, as well as for routine assays. The detector provides the chromatographer with automation capability during separation runs not previously possible on similarly priced detectors. Integrated with current sophisticated personal computer (PC) data acquisition systems such as *Star-Chrom™*, the DVW-10 can be controlled by the PC to respond to pre-set methods that have been stored by the operator. The DVW-10 also allows simple manual operation with a standard stripchart recorder or integrator. Lamps and flowcells are easy to change, making the detector a versatile workhorse for analytical, semi-prep or preparative work.

D-Star is committed to offering a broad range of components to the chromatography market. Therefore, it will be continuously adding to its product line not only detectors but a complete assortment of flowcells and accessories for many applications.

Contact your Dealer/Representative for additional product offerings.

1.6 Warranty

No other warranty exists, expressed or implied, except as shown here and in D-Star Instruments' Conditions of Sale.

Fitness to a particular application must be determined by the user.

Parts & labor are warranted for one (1) year, provided the unit is not damaged by improper use, abuse or by chemical spill.

Request a Return Authorization (RA) before returning any parts.

Return defective unit to Dealer/Representative for repair. Clean/sterilize all components prior to shipment. No parts will be accepted which present a health or safety hazard to service personnel. Repaired unit will be returned via parcel service or mail.

The flowcell and all tubing supplied with the instrument are warranted at the time of installation only.

Lamps are warranted on a pro-rated basis only. The deuterium (D2) lamp is warranted for six (6) months from the sale by D-Star, or for 1000 hours, whichever occurs first. If the lamp fails prior to 500 hours, and the lamp is returned to D-Star within the warranted six months, it will be replaced at no charge. The lamp will be replaced within the time frame on a pro-rated basis after the first 500 hours on the lamp, at a cost consistent with the hours on the lamp divided by the lamp warranty of 1000 hours times the lamp replacement cost.

1.7 DVW-10 Detector Specifications

Wavelengths:	195 to 800 nm
Lamps:	Standard - Deuterium (D2) (195-360 nm) Accessory - Tungsten (W) (360-800 nm)
Flowcells	
Analytical:	7mm pathlength, 10 μ l volume, 200 psi; wetted materials: PEEK, quartz or sapphire, Tefzel, Teflon; 1/16" O.D. tubing.
Preparative:	2.0 mm pathlength, 4 μ l volume, 100 psi; wetted materials: PEEK, quartz or sapphire, Tefzel, Teflon, 1/8" O.D. tubing.
Linearity:	Better than 2%
Wavelength Accuracy:	2 nm
Wavelength Reproducibility:	1 nm
Spectral Resolution:	5 nm
Stability (Drift):	Less than 2.5×10^{-4} AU/Hr (at 254 nm and constant temperature)
Noise:	Less than $\pm 2.5 \times 10^{-5}$ AU (5×10^{-5} AU peak to peak) at 254nm and constant temperature
Recorder Output (A):	10mV
Integrator Output:	1.0 V/AU to 2V (Equivalent to 2.0 AU)
Display:	
Absorbance:	4.5 digits to 1.999 AU
Wavelength:	3 digits for operating range 195-800 nm
Absorbance Ranges:	0.005, 0.01, 0.02, 0.05, 0.10, 0.20, 0.50, 1.0 AU
Front Panel Controls:	Wavelength (λ) selection, Range selection, Autozero, Event Mark
Rear Panel Controls:	Subminiature D-type, 15 position female connector for detector recorder/ integrator output, and remote control (Wavelength [λ] Up/Down, Event Mark, Autozero, Lamp On/Off, Sample/Reference Energy)
Power:	110/115, 220/230V factory preset
Dimensions:	7.1" H x 8.6" W x 16.0" L
Shipping Weight:	23 lbs

Tubing used in the biocompatible systems (standard analytical):

Column to Detector Flowcell Inlet: 1/16" OD, 0.015 ID PEEK
Detector Flowcell Outlet to Waste: 1/16" OD, 0.015 ID PEEK

Section 2.0 SYSTEM ORIENTATION

2.1 Front Panel

Manual controls for the DVW-10 Detector are located on the front of the unit (Figure 1). Each function is described in the Detector Operation Section. Additional types of controls are described in the Remote paragraphs of the same section.

The "1.000" Range LED and Absorbance/Wavelength Display should be lighted when the power is turned on.

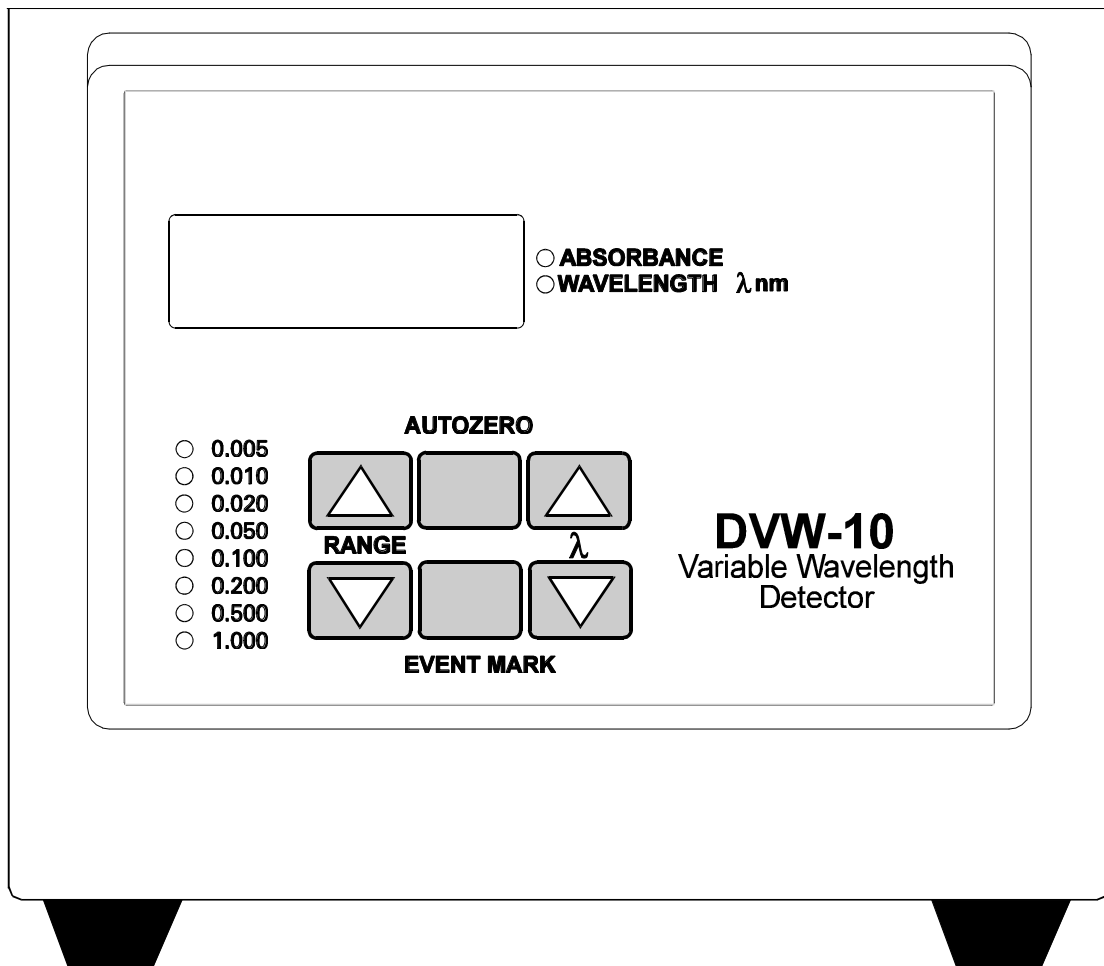


Figure 1 Front Panel

2.2 Rear Panel

The rear panel or chassis rear is shown at Figure 2.

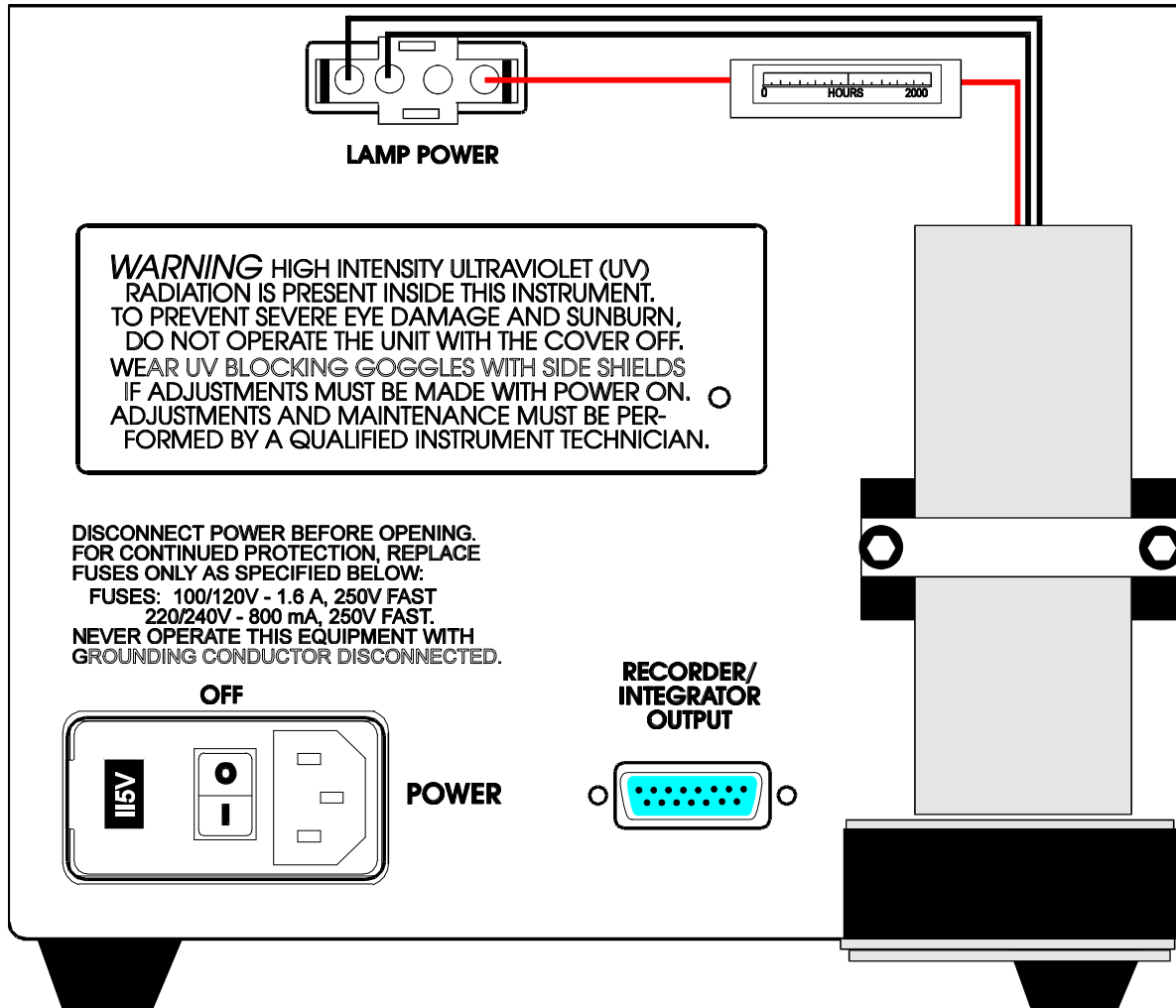


Figure 2 Rear Panel

The ON (I) and OFF (O) power switch is located on the Corcom™ power module. The Corcom may be programmed for 110/115 or 220/240 volt operation. No other power changes are required in the system. See the Fuse Changing/Replacement section in Part C for additional details.

The data output and remote control port (Recorder/Integrator Output) is located on the rear panel.

The DVW-10 lamps are mounted on the rear for easy access.



Notice the UV warning labels on the chassis and on the D2 lamp. Do not remove the flowcell without turning the power off or attempt to operate the unit with the cover off. UV energy will cause severe eye damage and sunburn.

See the Safety section for additional discussion.

2.3 Side Panel

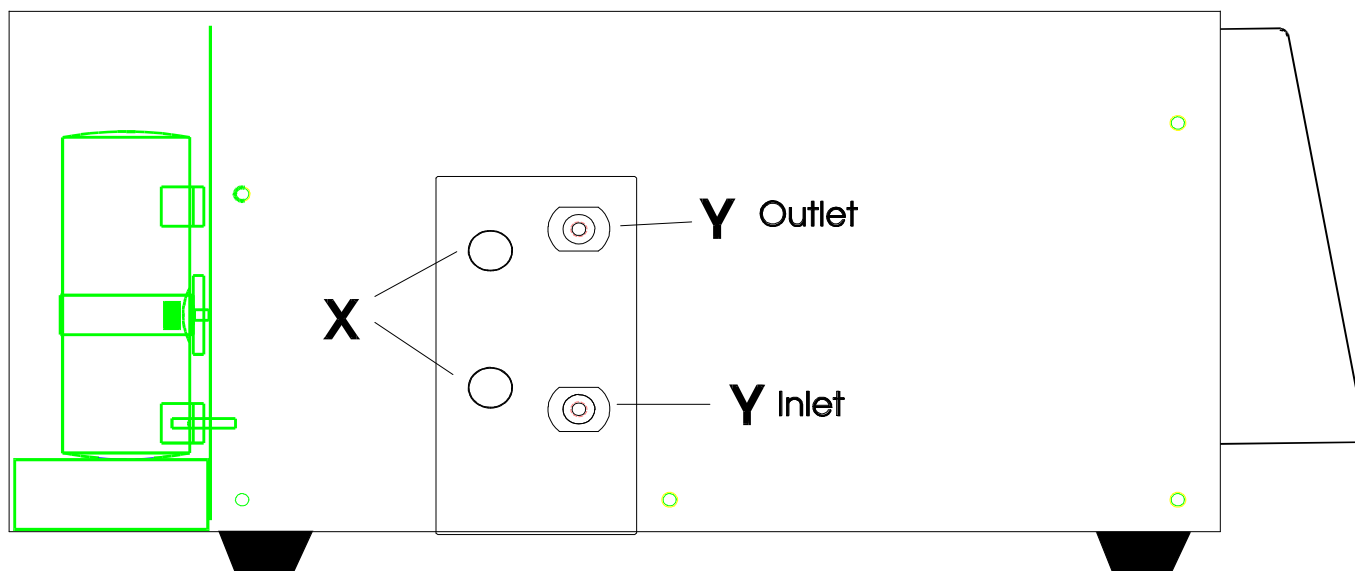


Figure 3 Side Panel

Figure 3 shows the side panel. An analytical flowcell is shown in its mounted position with the cover on. Thumbscrews (X) hold the cell assembly on the monochromator. The bulkhead fittings (Y) enable plumbing the unit from the column and to waste or other system components. The lamp and fan assembly are shown for reference. The lamp is located behind and under the cover to preclude possible side/top damage, as well as to prevent stray ultraviolet energy from causing operator injury.



Do not operate the unit with the cover/cabinet lid removed.

CAUTION: The lamp may be HOT. Do not remove the lamp while it is turned ON. There is danger of electrical shock and exposure to ultraviolet (UV) energy which causes severe damage to eyes and skin.

Section 3.0 Safety

The DVW-10 has been designed with user and application safety in mind. The following are the detector safety features and their purposes:

Grounding plug on AC power cord - must be plugged into a grounded wall outlet.
Factory supplied power transformer - do not substitute.
Enclosure/Cabinet/Cover - for electrical grounding and shielding, protection from liquid spray, UV energy blockage, and to minimize stray light.

WARNINGS:



DANGER!! Lethal voltages are present on the D2 Power Supply & Signal Processor Boards. With power OFF, the D2 Board can still be Dangerous. Wait until all RED LEDs go out. **SEE SECTION 8 OF THIS MANUAL**

Do Not Try To Defeat Any of the DVW-10's Safety Features

Do not attempt any electrical repairs without unplugging the power cord.
Unplug the power cord before opening the cabinet lid.
Do not remove or defeat the grounding pin on the power plug.
Do not attempt to defeat power grounds.
Do not defeat any of the system's other grounding schemes.
Contact an electrician if you are not sure of wall outlet voltage or grounding.
Do not operate the unit with the cabinet lid off or unscrewed.

The cover is attached to the chassis by a ground wire!

Ensure liquid fittings and connections are tight to avoid spraying solvents.

Do not operate the detector with liquid without eye protection.

Tubing may weaken with time, pressure and solvents. Inspect periodically.
The liquid system may be under injury-causing high pressure.
Exercise safe practices when using solvents, especially under pressure.

Do not look at the UV energy source. Damage to the eyes will occur. Use UV blocking goggles to prevent injury.

Exposure to UV energy will also cause *sunburn* to eyes and skin.

Section 4.0 INSTALLATION

4.1 PACKING LIST (Standard for DVW-10)

DVW-10 Variable Wavelength Detector with Deuterium lamp, 110/115V [DS014-0014], or 220/240V [DS014-0014-1]
Analytical Flowcell Assembly [DS010-0067] Standard
Accessory Kit Package - DVW-10 [DS010-0081] includes:
Recorder/Integrator Cable [DS 010-0024] ...1 ea
Hex Key Wrenches:
 for Flowcell Sockethead Cap Screws - 3/32" [DS 540-0004] ...1 ea
 for Lamp Sockethead Cap Screws - 9/64" [DS 540-0011] ...1 ea
Fuses:
 Corcom (115V): 5 x 20 mm, FAST, 250V, 1.6A [DS430-0009]...2 ea
 Corcom (230V): 5 x 20 mm, FAST, 250V, 800mA [DS430-0014]...2 ea
 Signal Processor Board: 5 x 20 mm, FAST, 250V, 200mA [DS430-0012]...2 ea
 D2 Power Supply Board: 5 x 20 mm, FAST, 250V, 500mA [DS430-0004]...1 ea
External Bulkhead Fittings, 1/16" O.D. Tubing (Male 10-32 Nut with Ferrule) [DS250-0043/250-0044]...2 sets
US Power Cord (115V) [DS610-0004] **or** Int'l Power Cord (230V) [DS610-0005]...1 ea
Operating Manual (this document) [DS 050-0004]...1 ea

NOTES:



1. Part numbers shown above are for shipped kits. Parts numbers shown elsewhere in the manual are for accessory/replacement/spare parts ordering.
2. Items indicated in the manual with the word "optional" or "accessory" are not supplied unless specifically ordered (additional cost).

4.2 INSTALLATION

4.2.1 General

1. After opening the shipping container, inspect the DVW-10 for any damage. Record damage to the shipping container and the detector in the event a claim needs to be filed.

CAUTION: Do not pick up the DVW-10 by the plastic front control panel enclosure (with overlay). Hold on to the base of the chassis.

2. Check the packing list (4.1 above) to ensure all items were shipped.
3. Place the unit in a well-ventilated area to avoid heat build-up.
4. Ensure that the factory set voltage setting on the Corcom is correct for your power supply. If it is different, consult the fuse changing section of this manual or contact your Dealer/Representative.
5. Ensure that the cover/cabinet lid is positioned correctly and the cover screws are tightened.
6. Check that the lamp plug and the lamp screws are tightly connected.

4.2.2 Flowcell

Two nylon flowcell plug screws are installed on the analytical flowcell prior to shipping to prevent flowcell contamination and to protect the bulkhead fittings from damage. Remove the plug screws and save them for future shipping or storage. The bulkhead fittings are located in the Accessory Kit Package. Instructions for preparing your flowcell for operation are located as Appendices at the rear of this Manual.

Write the name of each flowcell on the table of contents to provide easy reference to its location.

4.2.3 Power Cord and Recorder/Integrator Cable.

Attach the Power Cord to the Corcom.

Attach the data cable to the output plug. Screw in, but DO NOT OVERTIGHTEN, the screws on the external cable shell to the hex nut head on the output plug.

Use the wire hook-up instructions located on the end of the cable for the appropriate data device.

4.2.4 Problems.

If problems are encountered, consult the Troubleshooting Guide in Part C.

PART B: DETECTOR OPERATION

Section 5.0 Description of the DVW-10 Detector

5.1 Introduction

This instrument is a variable wavelength spectrophotometer that uses a flow-through cell to measure solute concentration in liquid streams. A special signal processing system converts the signal from a photodetector to a voltage directly proportional to concentration (absorbance). An autozero system provides push-button nulling of the output voltage level. There are output connections for a computing integrator of 1 volt per absorbance unit to 2 volts (equivalent to 2.0 AU). In addition, there is an output with eight levels of sensitivity provided for a 10 millivolt strip chart recorder. Front Panel LED characters display the same absorbance measurement. An Event Mark system triggers a voltage spike output to the data system.

5.2 Operating Principles

Photon energy supplied by an energy source lamp (UV or Visible) is passed through a flowcell monochromator to a photodetector. Electronic circuitry comprised of a differential logarithmic amplifier and signal conditioning system processes the photometric signal to provide an output voltage proportional to absorbance of the solution in the flowcell. Output sensitivity is a function of flowcell pathlength, concentration, and molecular extinction coefficient of the substance in solution (Beer's law). For measurements at specific wavelengths, a high efficiency plane ruled grating is rotated by a stepper motor to break out the energy to the desired wavelength in nanometers (nm).

5.3 Optical System

The monochromator, a unique optical bench design, is the heart of the DVW-10 detector. The optical system is shown below.

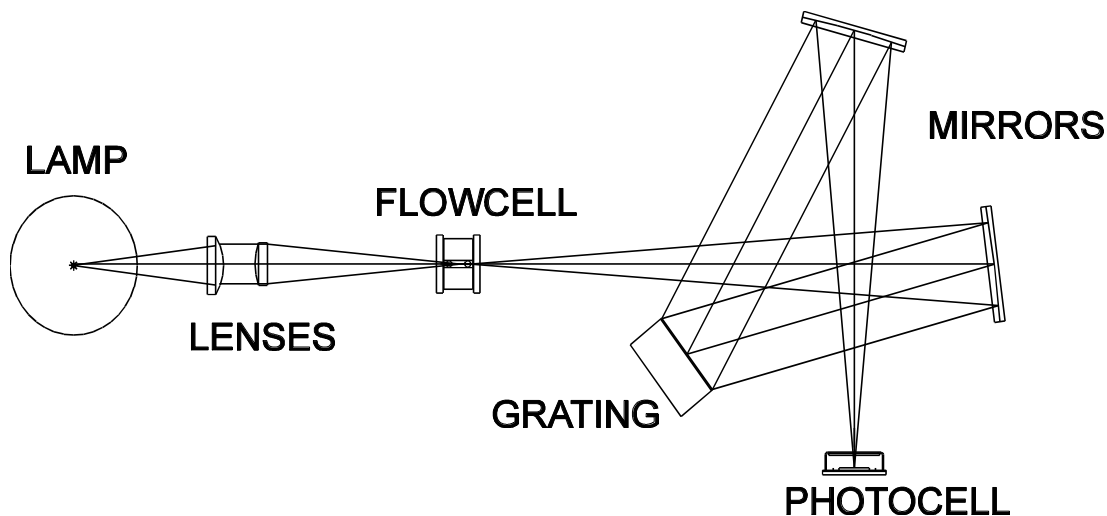


Figure 4

The double beam system with its sample and reference beams traveling the same paths to a matched dual element photodiode provides the most accurate (optimal) means for absorbance detection. The monochromator is compact and easily accessible to qualified service technicians.

5.4 Operating Wavelengths

A pre-aligned deuterium (D2) lamp is used to obtain maximum UV energy from 195 nm to 360 nm. A Tungsten (W) lamp assembly (accessory) provides visible light from 360 nm to 800 nm. Lamps may be easily changed by the operator. Only one lamp may be installed at one time. Using a lamp outside of the optimal ranges shown above will result in substandard performance.

The lamps are powered by a custom designed power supply.

5.5 Flow-Through Fluid Cells (Flowcells)

Various models are available. The standard cell for Analytical HPLC has a pathlength of 7.0 mm and an illuminated volume of 10 μ L. The analytical flowcell assembly is standard on the DVW-10.

A Preparative (Prep) cell is available as an accessory with a pathlength of 2.0 mm (4 μ L volume).

Wetted materials are chosen for chemical resistance as shown below:

Analytical cell: PEEK, quartz or sapphire, Teflon, and Tefzel.

Prep cell: PEEK, quartz or sapphire, Teflon, and Tefzel.

5.6 Limits to Performance

Solvents and/or mobile phases must be selected that will be compatible with the materials of construction (see list of materials above) for safety, proper operation and to prevent damage to the components of the flowcell assembly.

Backpressure on the flowcell may be necessary to prevent bubbles from forming which can cause poor baselines and noise.

See the Flowcell Appendix for backpressure advice.

Linearity depends upon the correspondence between the absorbance maximum of the sample substance and the measurement wavelength. For optimum performance these should be matched as closely as possible. Also, at absorbances above 0.5 AU, linearity can be expected to be less than at lesser absorbances.

Noise & drift are affected adversely by many factors including:

- Non-degassed mobile phases.
- Entrapped gas bubbles in the flowcell.
- Certain gradient combinations.
- Large or rapid temperature changes in the environment or fluids.
- Accumulation of residues in the flowcell.
- Mobile phases which absorb energy at the measurement wavelength.
- Faulty check valves on a pump.
- Chemical vapors in the atmosphere.

- Contaminated (dirty) windows and/or optics.
- Insufficient time allowed for warm-up of lamp and electronics.
- Source lamp aging or damage.
- Improper grounding (including ground loops).

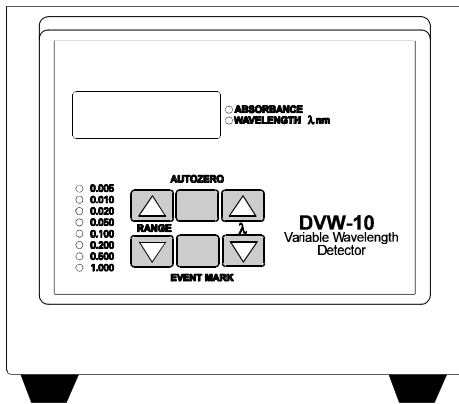
Consult the Troubleshooting Guide in Section C for problem identification and possible solutions.

Section 6.0 - INSTALLATION AND OPERATION

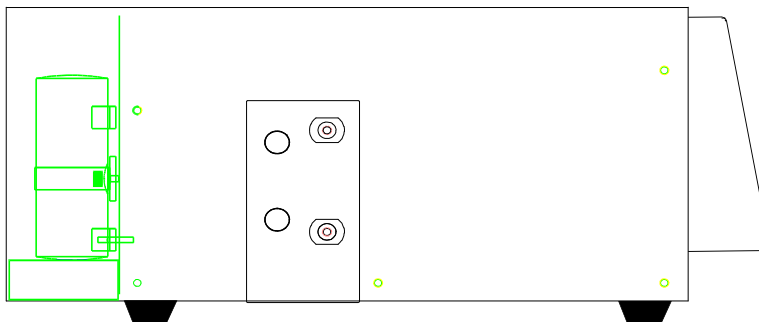
6.1 Orientation of the Detector

To simplify description of the detector component locations, the following terminology is used:

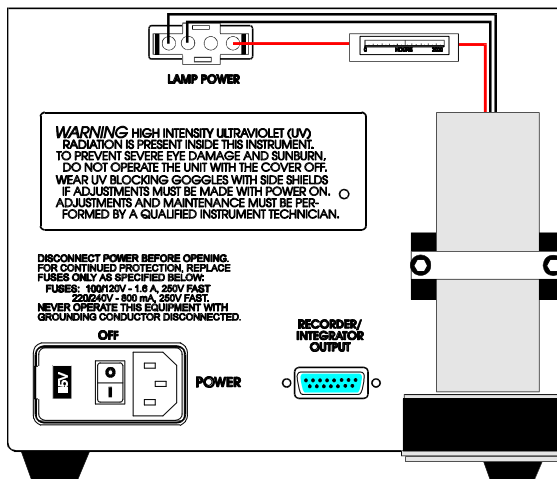
- Front Panel: The surface where the controls, switches and displays are located.
- Side Panel: The surface where the flowcell assembly is mounted.
- Rear Panel: The surface where the lamp, and electrical and input/output connectors are located.



Front panel



Side Panel



Rear Panel

Figure 5

6.2 Flowcell and Tubing Connections

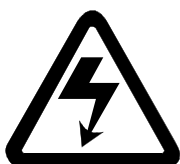
Consult the Appendix for each flowcell for installation instructions.

It is a good idea to check the tightness of the bulkhead fittings occasionally to prevent leaks. Do not overtighten.



NOTE: Metallic tubing and fittings may damage biocompatible flowcell parts and may require special strain relief considerations. Contact your Dealer/ Representative for details.

6.3 AC Power



CAUTION: Do not remove or defeat the grounding pin on the power plug. Contact an electrician if you are not sure whether the wall outlet is properly grounded.

AC power is required for operation of this instrument. The unit is internally wired to the transformer and is grounded to operate at 115 VAC. No 230 VAC adjustment is required except at the Corcom.

6.4 Controls

Figure 6 highlights the controls of the DVW-10 Variable Wavelength Detector. Individual graphics are shown with the specific descriptions.

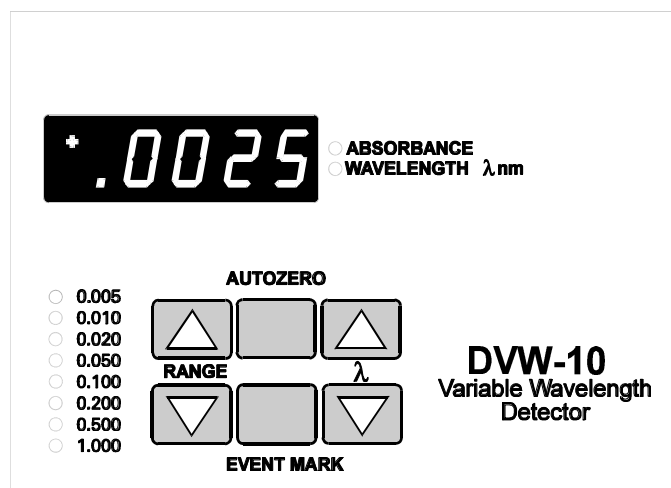
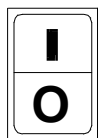


Figure 6 Detector Control Panel

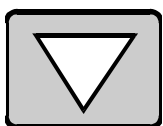


Power ON/OFF (Rear Panel) - Switch to the ON position [I] to operate the detector.



Range UP (Control Panel) - Press anytime to change recorder range. The detector "wakes up" in the 1.000 AU position. Actuation does not affect the integrator output level.

RANGE



Range DOWN (Control Panel) - Press anytime to change recorder range. Actuation does not affect the integrator output level.

NOTE: A sudden shift in the recorder position is normal when changing the range.

AUTOZERO



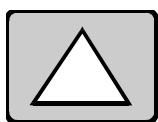
Autozero (A/Z) (Control Panel) - Press anytime to return output signal level to the baseline position. The autozero affects both the recorder and integrator outputs.



EVENT MARK

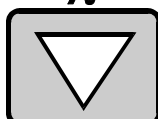
Event Mark (Control Panel) - Press to trigger an "event mark" on the recorder. The Event Mark should deflect the recorder approximately 1 to 3% full scale, depending on recorder pen speed.

In order to obtain an event mark signal for an integrator a remote cable is required. (See Section 7 and your Integrator manual for "Start Integration Input")



Wavelength UP (Control Panel) - Press to increase the wavelength display setting step by step. Holding down the button enables rapid stepping.

λ



Wavelength DOWN (Control Panel) - Press to decrease the wavelength display setting.



Internal limit switches prevent the monochromator grating from rotating too far and binding. **Please note that it is advisable NOT TO OPERATE BELOW 185 NM OR ABOVE 800 NM TO FURTHER SAFEGUARD THE WAVELENGTH LIMIT CONTROL FROM BEING DAMAGED AND FAILING TO MEET SPECIFICATION.**

6.5 Indicators

6.5.1 Range Selection Status

- 0.005
- 0.010
- 0.020
- 0.050
- 0.100
- 0.200
- 0.500
- 1.000

Absorbance ranges (8 red/orange LEDs).

The LEDs display the full scale output range (AUFs) to a recorder attached to the Recorder/Integrator Cable on the rear panel. The range selected does not affect the 1V/AU output to an integrator.

Power On is indicated by one of the red/orange Range LEDs being lighted.

6.5.2 Absorbance Display



● **ABSORBANCE**
○ **WAVELENGTH** λ nm

When powered ON, the Absorbance Display will be lighted as will the red/orange "ABSORBANCE" status LED. The display will output both positive and negative voltages, with a range of .0000 to 1.999 absorbance units (AU).

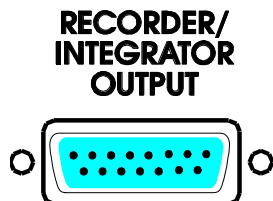
6.5.3 Wavelength Display



○ **ABSORBANCE**
● **WAVELENGTH** λ nm

The Wavelength Character Display and the "WAVELENGTH NM" status LED will be lighted when either the Wavelength (λ) UP or DOWN buttons is pressed. The display will revert (or time-out) to the ABSORBANCE display ten (10) to twenty (20) seconds after the last λ button push.

6.6 Input/output Connectors



15-position female subminiature D-type on rear panel for:
Recorder - 10 mV full scale range
Integrator - 1.0 V equivalent to 1.0 Absorbance
Remote Control - Contact closure inputs (see Section 7 of this manual for details).

6.7 Recorder/Integrator Cable

Attach the Recorder/Integrator cable to the Recorder/Integrator Output port on the rear of the unit. Do not over-tighten the cable hood screws onto the output hex nuts.

The cable supplied has a 15-position male subminiature D-type connector on one end and stripped and tinned leads on the other. The colors of the wires are associated with their purpose:

Black	- Integrator [-]
Red	- Integrator [+]
Green	- Recorder [-]
White	- Recorder [+]
Green/Yellow	- Shield

The recorder cable is also marked with the wire connections.



IMPORTANT FOR BEST OPERATION:

Connect the Green/Yellow wire of the recorder/integrator cable to the recorder or integrator's guard terminal, if available. Otherwise, connect the Green/Yellow wire to the recorder or integrator's ground terminal. Make sure that the recorder or integrator power cord is plugged into the same outlet as the DVW-10. Isolate (cover) the tinned leads of unused wires so that they do not create ground loops and introduce noise into the system.

6.8 Power-on and Warm-up

Switching the unit ON at the CORCOM turns on power for both the electronics and the lamp. The deuterium (D2) lamp will cycle through a 10 second filament warm up before it ignites. The tungsten (W) lamp will ignite almost immediately.

NOTE: Allow the measurement electronics and the source lamp a minimum of 60 minutes to warm up prior to initiating your analytical run.

The absorbance display will show a large reading, either + or - before, during, and after the lamp is turned on. Once the lamp has been on for approximately 30 seconds, push the autozero (AZ) button. The absorbance LED characters should display from +.0000 to +.0030. See the Troubleshooting Guide in Section C if the display is outside of the above range.

If the unit still shows instability please contact your dealer. The warm-up requirement should be reduced with the continued use of the instrument.

6.9 Wavelength

6.9.1 Wavelength Lamps

Wavelength is dependent on the energy source. The optimum wavelength ranges for DVW-10 lamps are:

195-360 nm for the Deuterium (D2) Lamp.

360-800 nm for the accessory Tungsten (W) Lamp.

Select the lamp appropriate for the wavelength range desired before beginning operations. See Section 8 for lamp changing instructions.



Attempting to use a lamp outside its optimum wavelength range will result in substandard performance.

6.9.2 Wavelength Selection

Wavelength selection is achieved by moving the system optics with a high ratio geared stepper motor. Each push of the wavelength button advances the grating position approximately 0.45 nm. As in all geared mechanisms, there is a certain amount of slack or backlash between the gears when the motor changes direction. The DVW-10's backlash has been minimized to enable precision wavelength location. Due to the backlash, there is a slight hesitation when changing directions, as the gears reverse and the slack is taken out of the system. The direction change takes up to four (4) pushes of the wavelength button.

To assure wavelength reproducibility, it is recommended that the operator always select the final wavelength setting from the same direction: Downward

Example: Use the DOWN direction as standard. Determine the desired wavelength (λ). If the desired setting is at a **lower** wavelength than the current wavelength, push the DOWN button until the desired λ is reached.

However, if the desired wavelength is **above** the current setting, push the UP button until the desired λ is passed by at least five (5) nanometers. Then push the DOWN button several times (do not hold it down) until the wavelength display reaches the target λ .

To display the current wavelength, always press the UP button to change the display from absorbance to wavelength. The backlash will prevent the current wavelength from being disturbed for about two or three presses of the button. After that, each pressing of the button may cause the wavelength to move upward. If this happens, move the wavelength upward by another five nanometers and then downward to the desired wavelength. Again, downward should be the last button pressed when setting the wavelength.

Each time the instrument power is turned on, wavelength may start to change. Each power up sequence is equivalent to one UP button press. Always check to see that the wavelength displayed is the wavelength you want for the next chromatographic run.

6.9.3 Wavelength Scrolling

The wavelength selection may be sped up or scrolled by holding down one of the buttons. After about two seconds, the scrolling will begin on the display.



Internal limit switches prevent the monochromator grating from rotating too far and binding. **Please note that it is advisable NOT TO EXCEED 185 NM OR 810 NM TO FURTHER SAFEGUARD THE WAVELENGTH LIMIT CONTROL FROM BEING DAMAGED AND FAILING TO MEET SPECIFICATION.**

6.10 Performing Chromatography Runs

6.10.1 Test Runs

After setting the wavelength and range (for recorders) it is recommended that a test run be performed.

- Autozero the detector.
- Inject the sample.
- (For Recorders) Push the Event Mark button and note the mark that it makes.
- Observe the peaks on the data collection system and determine which settings or parts of the system need to be adjusted to provide the best chromatography.
- Make the adjustments and retest.

6.10.2 Record Runs

Ensure that the detector has had at least one hour to warm up, and that the baseline noise is stable.

Autozero the unit before each sample run or injection.

6.11 Shutting the Unit Down

It is recommended that the detector be shut down during long periods of inactivity. This will extend lamp life.

After use, it is also recommended that the flowcell be flushed with a fresh, non-ionic solution. Flushing the cell will help remove any retained sample or other contaminants. Flushing is also essential if ionic buffers have been used (to remove buffer salts). Flush the flowcell with several volumes (flowcell and connector tubing volumes) of solution. Typical non-ionic solutions include:

- Distilled water
- Isopropyl alcohol
- Methyl alcohol



DO NOT PUSH OR INJECT THE FLUSH SOLUTION. Always draw fluid into the flowcell to avoid over-pressuring the cell or spraying eluant and causing injury or spillage.

SECTION 7.0 - REMOTE CONTROL (ACCESSORY CABLE)

7.1 External Control

One of the unit's key attributes is that it enables the operator to remotely control the detector with a personal computer (PC). This section outlines the remote control features and the 15-pin D Subminiature connections.

In order to avoid damaging the unit and voiding the warranty due to improper wiring, it is strongly recommended that a factory supplied Remote Control Cable (PN: 025-0079) be obtained from your Dealer/Sales Representative.

In order to ensure that the remote control works properly with your PC (and to avoid damaging your PC) it is recommended that a technician qualified to install interactive instrument control devices be contacted to assist in selection of the correct interface module for your computer system.

D-Star's *Star-Chrom*™ HPLC Management System will control the detector, acquire data, and provide analysis capability. Contact companies such as Keithley Metrabyte, National Instruments, or your data acquisition system supplier and provide them with the technical information in the table below.

7.2 Remote Operation of the DVW-10

The following detector functions may be controlled through the remote cable:

7.2.1 Lamp On / Lamp Off Control (Digital Input)

The Lamp On/Off function enables the operator to turn the lamp OFF during times when measurements are not being made but the detector remains ON. This function helps to conserve lamp life. Once the lamp is turned back on, sufficient time must be allowed for the lamp to warm up and to stabilize.

7.2.2 Photocell Energy - Sample & Reference (Analog Output)

Sample Energy and Reference Energy from the photocell may be monitored with these outputs. This facilitates external diagnosing of suspected flowcell or photocell problems if changes from factory specifications/measurements are observed.

7.2.3 Wavelength UP/Wavelength DOWN

This function is the same as pushing the Front panel buttons to select a wavelength. When these functions are selected they will cause the front panel LED display to show the wavelength changing. Each instrument has a constant wavelength setting of 1.0 mV/nm.

To initialize the computer wavelength:

1. Determine the Wavelength Voltage (in mV) from the wavelength output (see chart below).
2. Subtract the Front panel wavelength display from the Wavelength Voltage. This is the Wavelength Voltage Offset and is constant for each individual instrument and is always positive.
3. Configure the computer's software to subtract the Wavelength Offset (voltage) from the Wavelength Voltage to provide the PC Wavelength measurement.

Activating the Up or Down function will cause the motor drive to move the grating to a new position. The new wavelength setting is calculated by subtracting the Wavelength Offset from the Wavelength Voltage.

7.2.4 Offset (Analog Output)

The Offset function measures the amount of coarse autozero required to compensate for flowcell imbalance. Possible uses include the detection of protein or other deposit buildups on the flowcell windows.

7.2.5 Event Mark

This function enables the operator to trigger a voltage spike to the recorder. It does not affect an integrator. It may also be used to detect that the internal event mark function has been triggered by the front panel pushbutton or another device on this line. For the DVW-10 that would be from pushing the Front panel Event Mark Button.

7.2.6 Autozero

The Autozero function is the same as the Front panel AZ button. It returns the output signal level to the baseline position for both recorders and integrators.

7.3 Remote Control Technical Details

7.3.1 Recorder/Integrator and Remote Connections

Signal Name	DB-15M Pin No.	Wire No./Color	Logic or Analog	Description
Recorder (+)	1	1 / White, Rec/Int Cable	Analog output	Output to 10 mV strip chart recorder, positive terminal
Digital Ground	2	3 / Black, Remote Cable	Digital	Signal return wire, logic low
Wavelength	3	5 / Gray, Remote Cable	Analog output	Voltage proportional to wavelength setting, approximately 1.0 mV/nm, requires zero offset
Integrator (-)	4	7 / Black, Rec/Int Cable	Analog output	Output to integrator or data system negative terminal, at analog ground potential
Offset	5	9 / Red, Remote Cable	Analog output	Voltage proportional to last autozero balance operation
Wavelength Up	6	11 / Green, Remote Cable	Digital input	Steps wavelength upscale, approximately 0.6 nm/step
Sample Energy Out	7	13 / White, Remote Cable	Analog output	Remote sample energy monitoring (photocell)
Autozero	8	15 / Orange, Remote Cable	Digital input	Starts autozero sequence to balance (null) integrator and recorder outputs
Reference Energy Out	15	14 / Violet, Remote Cable	Analog output	Remote reference energy monitoring (photocell)
Wavelength Down	14	12 / Yellow, Remote Cable	Digital input	Steps wavelength downscale, approximately 0.6 nm/step
Lamp On/Off Control	13	10 / Brown, Remote Cable	Digital input	Remote lamp shut down
Shield	None	8 / Green-Yel. Rec/Int Cable	Chassis Ground	Cable shield, ground potential, tie to recorder or integrator guard or to chassis ground
Recorder (-)	11	6 / Green, Rec/Int Cable	Analog output	Output to strip chart recorder negative terminal, at analog ground potential
Event Mark	10	4 / Blue, Remote Cable	Digital input or output	Generates a marking pulse on the recorder output; also used to start data collection when used with Star-Chrom
Integrator (+)	9	2 / Red, Rec/Int Cable	Analog	Output to integrator or data system positive terminal, 1 volt per absorbance

7.3.2 Alternate uses of Pins/Wires

Certain of the Remote Cable 15-pin DB Connector (DB-15M) pins and wires may be reprogrammed

(see below). The different functions include Range Up, Range Down, and Range Reset (to least sensitive recorder range); however you may lose Energy readings and/or Lamp On/Off functions.

Additionally, a customized cable to enable a 200 mV per absorbance output to an integrator or data system positive terminal may be ordered. Consult Factory for options.

7.3.3 Technical Discussion

The output impedance of the integrator (+) terminal is less than 100 ohms, the recorder output (+) terminal is 5.0K ohms or less depending upon the range selected. All digital inputs are to be driven with a momentary open collector or contact closure to digital ground; active LOW; open circuit voltage is approximately +5 V. Contact closure time should be between 10 and 200 milliseconds to assure proper actuation of the remote function.

Optical isolation is recommended to prevent ground loop noise being induced into the measurement system.

The Autozero selection may be actuated by a momentary external contact closure. Using opto-isolators or isolated relay contacts are the preferred methods of providing external control since no ground loops are created (a potential source of noise) and release time of the contact is predictably fast.

Open collector logic drivers may be used. However, since the common of the logic circuit must be tied to power ground of the detector, a ground current between the controlling device and the detector may be induced into the measurement circuit. Please be aware that a ground loop may occur that increases measurement noise.

Lamp Off requires that the circuit be pulled low to ground (green/yellow) and held for the period that *Lamp Off* is required.

Event mark signal is both a logic input and an output (open collector OR or "party line"). Data systems may use this signal output to begin data collection when a front panel EVENT pushbutton is pressed (selected models) or an external signal is received (consult Factory).

Caution: Disconnect the power cord from AC power (mains) when it is necessary to open the instrument cover to make any internal changes or to perform maintenance.



Alternate uses for remote control connections require program jumper changes from factory default positions. Consult the Factory for further information.

7.4 Circuit Protection

Although the detector's circuitry has static protection, it is best to avoid static discharge to any line. Each controlled circuit has a **4.7K** pull-up resistor to an internal **+5 V**. A pull-down to logic low discharges a 0.1 uF capacitor through a 100 ohm resistor on the signal processor PC board of the detector.

Section 8.0 - SAFETY AND MAINTENANCE

8.1 Safety

8.1.1 General

The DVW-10 has been designed with the user's safety in mind. The following are the safety features of the detector:

- Enclosure - for UV blockage, electrical grounding and shielding, and to minimize stray light;
- Cover is attached to the chassis by a ground wire;
- Power Entry Module (CORCOM) - for power OFF only fuse changing, as well as RFI filter, and IEC connector.



NOTE: Do Not Try To Defeat Any Safety Feature

8.1.2 Electrical Shock Hazard

In addition to normal line voltage hazards at the Corcom, and the transformers, there is line voltage on both the Signal Processor circuit board and the D2 Power Supply board. Location of the boards is shown in figure 7 below.

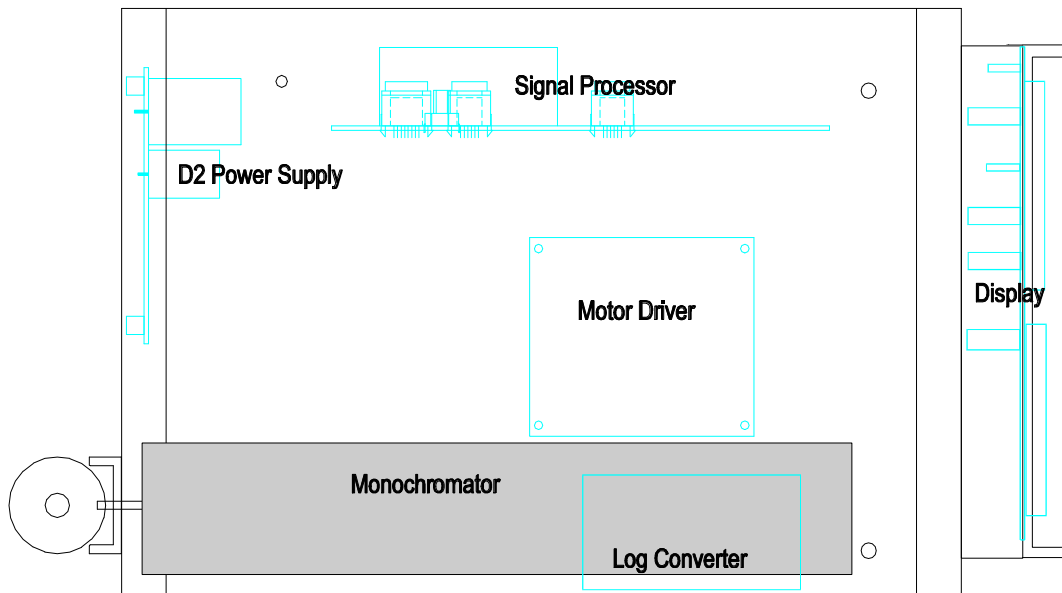
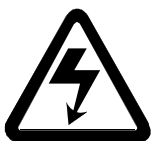


Figure 7



DANGER!!! WITH POWER OFF, THE D2 BOARD CAN STILL PRESENT A SHOCK HAZARD. Wait until all three of the red LEDs go out. Illuminated, they indicate that the capacitors are still charged. Location of the LEDs is shown in figure 8 below

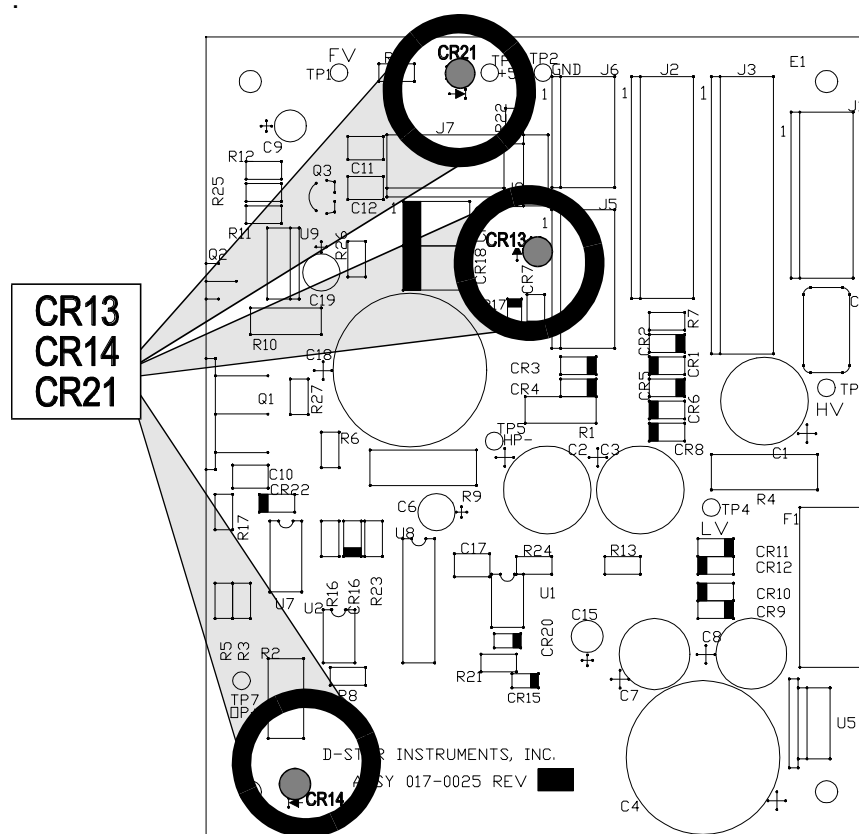


Figure 8

8.1.3 UV Radiation

When in use the Deuterium (D2) lamp (and to a lesser extent, the Tungsten lamp) emits ultraviolet radiation.

The lamps were placed at the rear of the case to allow easy changing by the operator and to reduce heat in the case. The lamps have been encased and the energy path into the instrument enclosure has been shielded. There is, however, danger of UV exposure if you look directly at the D2 lamp or operate the lamp outside of its housing.

UV is also present in the flowcell housing (direct or reflected) when the flowcell has been removed with power ON. Turn power OFF before removing the flowcell.



CAUTION: Only a qualified Technician should open the Monochromator. Damage to the optical system may occur.

8.2 MAINTENANCE

User/Operator Maintenance includes:

- Cleaning the Flowcell cavity
- Cleaning leaks on the floor of the chassis
- Changing or Cleaning the Flowcell
- Changing or Replacing the Lamp (D2, W)
- Changing or Replacing Corcom Fuses (see Section 9).

All other maintenance and service should be performed by a qualified instrument technician.

8.2.1 Electrical

Disconnect the instrument from the power outlet before attempting any maintenance **(including flowcell removal)**.

Avoid chemical or mechanical damage to cables and the enclosure.

Do not substitute components such as power transformer or internal electrical components.

There are no user serviceable parts inside the enclosure. Refer servicing of the internal components of the detector to a qualified instrument technician.

Power supply is user set at the Corcom on the Rear Panel.

Internal power supply jumpers - factory setting only.

8.2.2 Chemical Cleanup

Clean up any chemical or solvent spills immediately.

Do not allow liquids to spill into the detector enclosure.

In the unlikely event of a leak, the DVW-10 flowcell enclosure has been sealed to prevent liquid flow into the rest of the monochromator. It also has a drain hole to channel liquid to the underside of the chassis, away from the optics, electronics, and electrical components. Clean up the housing with a non-ionic solution to eliminate vapors and contaminants. Rinse off the flowcell bulkhead assembly with the same solution.



CAUTION. Exercise safe practices when using solvents. Liquids are under pressure and may cause injury. Wear eye protection, even when performing routine maintenance.

8.2.3 UV Radiation

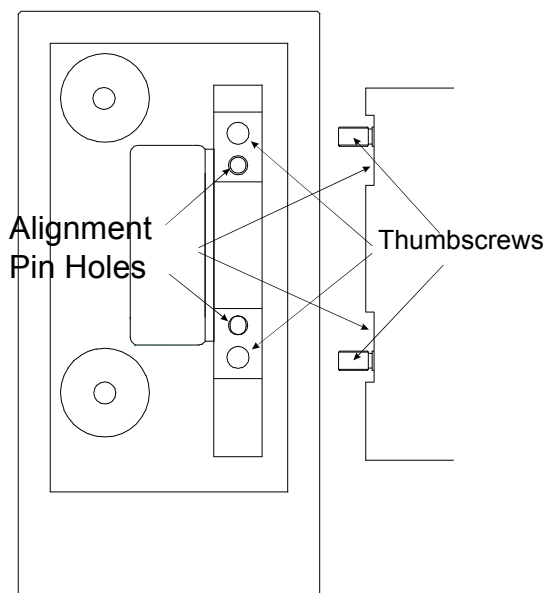
Disconnect power cord from the Corcom and the power outlet before removing the flowcell or performing any maintenance inside the DVW-10 Detector.



Wear UV protective goggles or safety glasses with side shields if the lamp must be operated during maintenance of the monochromator or while the flowcell is removed.

8.2.4 Flowcells

NOTE: The design of the DVW-10 DOES NOT require that you take off the cover/cabinet lid. You DO NOT have to take out the flowcell if you must remove the cover/cabinet lid.



Flowcell Bulkhead Assembly

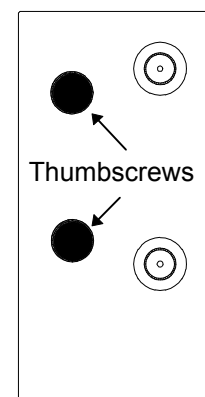
8.2.4.1 Installing/Removing a Flowcell

The flowcell bulkhead assembly is factory pre-aligned and is designed to enable precise orientation of the flowcell sample and reference paths with the optical path. Alignment of the flowcell on the bulkhead is a factory operation.

Two pins in the monochromator guide the bulkhead into place. Two thumbscrews secure the bulkhead to the internal wall of the optical bench. The thumbscrew is held on to the bulkhead by an "E" ring.

8.2.4.1.1 Installation

- Ensure flowcell windows are free of fingerprints, lint, and contaminants. Clean with pure Isopropyl Alcohol.
- Attach external tubing to the bulkhead fittings provided with the flowcell in use. Consult the Appendix for your flowcell. Always attach tubing and secure fittings to components before introducing liquid into the flowpath. Dry fittings assure the best seals.
- Ensure fittings are tight. Do not attempt to loosen and tighten fittings while there is liquid in the flow path. THIS CAN CAUSE LEAKS.
- Insert the flowcell into the flowcell housing.
- Push the bulkhead on to the alignment pins.



Dressplate

- Slowly turn the thumbscrews clockwise. Alternate between the upper and lower screws after several turns. If both screws are turned equally, you will feel no resistance.



Caution! The bulkhead does not require any force to be installed or removed. If you feel any resistance there is something binding. Reverse procedures and start over.

- Finger tighten the screws. The flowcell should be rigid.

8.2.4.1.2 Removing a Flowcell

If the flowcell is to be changed or stored, clear liquid from the flowpath and disconnect external tubing. Clean the cell as provided below.

- DO NOT REMOVE THE COVER/CABINET LID.
- Turn the thumbscrews counter clockwise, alternating between each screw after several turns. IF THE UNIT BINDS, SLIGHTLY REVERSE THE LAST SCREW TURNED.
- Once the screws turn freely, pull the bulkhead assembly straight out to avoid damaging the alignment pins.

NOTE: If the bulkhead assembly is to be removed for an extended period of time, cover the flowcell housing hole to keep dust and particles from contaminating the optical system.

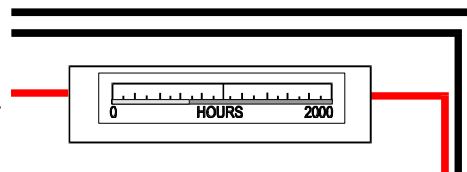
8.2.4.2 Cleaning Flowcells

Use a syringe to clean the flowcell with solvents or other chemical solutions. DRAW THE LIQUID into the cell. DO NOT FORCE the cleaning solution into the cell. A luer adapter attached to the flowcell tubing facilitates connecting the syringe, thereby reducing the likelihood of leaks.

If the flowcell must be disassembled for cleaning or to replace items, see the Appendix for the particular flowcell.

8.2.5 Replacing/Changing the Standard DVW-10 Deuterium (D2) Lamp (195-360 nm)

1. The D2 lamp is equipped with a lamp power chronometer (see figure 9 below). The meter indicates elapsed time that the lamp has been on. The expected useful life of the lamp is 1000 hours (or 1/2 of the original energy). To ensure good chromatography, the lamp should be changed before the 1000 hour mark. Lamps which fail under 500 hours will be replaced. Lamps failing with over 500 hours will be replaced at a prorated cost.



2. Removing the D2 Lamp Assembly
Figure 9

- Turn the power OFF and disconnect the power cord at the Corcom (rear panel).
- Disconnect the lamp plug on the rear panel.



CAUTION: Allow the lamp to cool off before handling to avoid burns!

- Using the 9/64" hex key wrench, remove the socket head screws holding the lamp and the "U" clamp (attached by ground wire to the chassis).



CAUTION: The lamp assembly and housing are attached to the monochromator. DO NOT TURN OR TWIST THE LAMP ASSEMBLY!

- Gently pull the lamp straight out from the rear panel. The lamp is held to the monochromator by a pin on the lower lamp housing clamp and by a lamp transition tube at the center lamp housing clamp.
- Do not touch the center hole of the lamp. Fingerprints on the lamp will absorb energy and degrade performance. Clean the lamp with Isopropyl Alcohol to remove any lint.
- Do not remove lamp end caps, wires or connectors.
- Store the lamp in a cushioned container if it is to be used again.
- Consult local ordinances for instructions on disposing of the lamp.

3. Installing the D2 Lamp Assembly

NOTE: ALWAYS KEEP LAMP TIGHT IN THE LAMP HOUSING.

Moving the lamp from its prealigned position will degrade detector performance.

- Ensure that the exposed lamp surface is free of finger prints and lint.
- Orient the lamp with the wires on top of the lamp.
- Slide the lamp tube onto the center lamp transition tube and the lower clamp pin. DO NOT FORCE THE LAMP. The lamp should slide easily onto the tube and pin.
- Secure the lamp with the "U" clamp and the hex screws. The lamp should be snug. The U clamp may be distorted if there is any twisting movement. Push in slightly at the center of the U until the lamp is snug against the housing to ground the lamp.
- Connect the lamp plug to the receptacle on the rear panel. It will only fit onto the receptacle in one direction. Ensure that the wing clips snap into place to avoid a lamp power disconnect.

8.2.6 Changing/Replacing the Tungsten (W) Lamp (360-800 nm) [Accessory]

1. The pre-aligned Tungsten lamp is an accessory item and may be ordered from your Dealer/Sales Representative.
2. The average life of the Tungsten Lamp is about 1000 hours.

The W lamp should be used at wavelengths above 360 nm. The D2 lamp may appear to have energy at wavelengths above 360 nm; however, this energy is usually second order spectrum of D2 and cannot be relied upon to be true energy at the displayed wavelength.

For visible operations (360 to 800 nm), remove the D2 lamp assembly per instructions above and replace it with the pre-aligned W lamp assembly.

3. Installing the W Lamp Assembly

NOTE: ALWAYS KEEP LAMP TIGHT IN THE LAMP HOUSING.

Moving the lamp from its prealigned position will degrade detector performance.

- Ensure that the exposed lamp surface is free of finger prints and lint.
 - Orient the lamp with the wires on top of the lamp.
 - Slide the lamp tube onto the center lamp transition tube and the lower clamp pin. **DO NOT FORCE THE LAMP.** The lamp should slide easily onto the tube and pin.
 - Secure the lamp with the "U" clamp and the hex screws. The lamp should be snug. The U clamp may be distorted if there is any twisting movement. Push in slightly at the center of the U until the lamp is snug against the housing to ground the lamp.
 - Connect the lamp plug to the receptacle on the rear panel. It will only fit onto the receptacle in one direction. Ensure that the wing clips snap into place to avoid a lamp power disconnect.
4. Removing the W Lamp Assembly
- Turn the power OFF and disconnect the power cord at the Corcom (rear panel).
 - Disconnect the lamp plug on the rear panel.



CAUTION: Allow the lamp to cool off before handling to avoid burns!

- Using the 9/64" hex key wrench, remove the socket head screws holding the lamp retaining clamp.



CAUTION: The lamp assembly and housing are attached to the monochromator. DO NOT TURN OR TWIST THE LAMP ASSEMBLY!

- Gently pull the lamp straight out from the rear panel. The lamp is held to the monochromator by a pin on the lower lamp housing clamp and by a lamp transition tube at the center lamp housing clamp.
- Do not touch the center hole of the lamp. Fingerprints and lint on the lamp will absorb energy and degrade performance. Clean the lamp with Isopropyl alcohol to remove any lint.
- Do not remove lamp end caps, wires or connectors.
- Store the lamp in a cushioned container if it is to be used again.

8.2.7 Calibration

The DVW-10 is pre-calibrated at the factory and is expected to remain in calibration under normal (controlled) operating conditions.

Due to the dynamic nature of HPLC, all detectors produce slight variation in their output in response to chromatographic peaks. These differences are a function of numerous chromatographic variables which include: time, temperature, tubing volume, fittings, eluent concentration, pump, column, and other system components, and the integrator/data acquisition response time.

To assure that your detector provides accurate and consistent performance, it is recommended that you conduct standardization runs for your applications or methods.

Test the unit with substances of known composition and concentration under static and repeatable conditions. Record and save the data so that instrument performance can be compared over time. An Instrument Standardization Log format is provided at Appendix B.

This methodology is also helpful in eliminating the detector as a source of problems. (See also Troubleshooting Guide.)

If the instrument exhibits major change from its historical standardization, contact your Dealer/Sales Representative.

8.2.8 Replaceable Parts (see also Section 12, Part C)

Flowcell Assembly - Prep or Analytical

Source Lamp - D2 or W

Cables, Tubing, Fittings

PC boards (Maintenance technician)

8.2.9 Tubing Preparation

8.2.9.1 Cutting and Finishing (Roughening) PEEK Tubing

Make certain that all tubing ends are cut square with the tube axis, and that both the ID and the OD are thoroughly deburred.

Inspect the end of the tubing where the ferrule will seat for visible scratches which are not acceptable. Scratches behind the front edge of the ferrule will not interfere with the integrity of the fitting. Minor scratches can often be eliminated by folding a small piece of fine emery cloth or wet-or-dry sandpaper (200 to 400 grit) around the end of the tubing and rolling the tubing between two fingers. This leaves concentric axial lines in the area where the ferrule seats, which, while not ideal, are less likely to cause a leak than longitudinal scratches.

8.2.9.2 Cleaning

After it has been finished, the tubing should be cleaned to remove residual shavings and grit caused by the sandpaper. This is best accomplished by using a syringe or pipette to flush a solvent such as methyl or isopropyl alcohol or acetone through the tubing and then drying it with clean, dry compressed air or carrier gas.



CAUTION: Exercise good laboratory safety practices when using solvents, particularly when subjecting them to pressure.

8.2.10 Fitting Assembly

1. Slide the nut and ferrule onto the tubing.
2. Insert this assembly into the fitting detail, screwing the nut in two or three turns by hand.
3. Push the tubing all the way forward into the detail so that it seats firmly. This is essential for a proper zero dead volume connection.
4. Manually turn the nut into the detail until it is finger tight.
5. Turn the nut 1/4 turn (90°) past the point where the ferrule first starts to grab the tubing. Fittings larger than 1/8" may require more than 1/4 turn (as much as 120°). The amount of force required can vary considerably due to the friction between the nut and the

threads and the composition and wall thickness of the tubing used. Because of these variables a torque specification is unreliable.

6. Remove the fitting and inspect it. When made up properly, the ferrule may be free to spin axially on the tubing, but should have no lateral movement along the tubing. If the ferrule moves laterally, reinstall the fitting into the detail and tighten it another 1/8 turn past finger tight.
7. Remove, reinspect, and repeat, if necessary.

PART C: MISCELLANEOUS

Section 9.0 Power Management

9.1 General



CAUTION: Do not remove or defeat the grounding pin on the power plug. Contact an electrician if you are not sure whether the wall outlet is properly grounded.

9.1.1 AC Power

The DVW-10's AC power configuration is shown at the rear panel on the Corcom above the power cord inlet hole. Plug the AC power plug into a **grounded**, operating 115 VAC outlet (or 230 VAC if so required). Other voltages such as 100V (for parts of Japan) require a power transformer change.

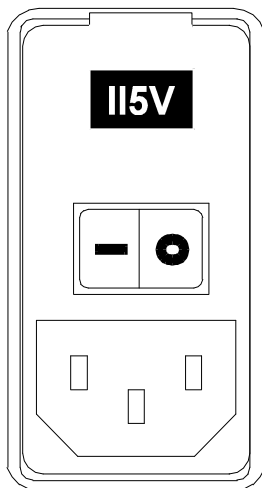
**A different power system must be supplied for use at 100 volts AC.
Contact your Dealer/ Sales Representative.**

9.1.2 DC Power Supply

DC power for the detector is provided by the DVW-10's transformers.

9.2 Power Entry Module

9.2.1 Description



The DVW-10 Corcom power entry module (referred to as "the Corcom"), provides dual voltage selection integrated into the fuse holder. It has a SPST switch for the dual primary transformer.

The fuse holder can hold two 5 x 20 mm (supplied) or two 1/4" x 1-1/4" fuses. Use 250V rated fuses.

The Corcom provides general filtering for both line-to-ground and line-to-line noise and will generally allow compliance with FCC and EC limits for line power supplies.

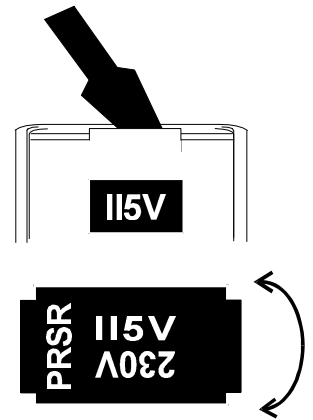
The Corcom also provides an RF shield of the filter components. The shield covers the filter portion of the module and increases performance of the filter by protecting the components from radiated noise. The shield improves RF ground connection to the case.

9.2.2 Changing Voltages (Conversion)



WARNING: Unplug the power cord from the Corcom before attempting to check the fuses.

Gently pry off the Corcom panel cover plate with a small flat-bladed screwdriver to access to the voltage fuses. Remove the fuses and set them aside. Check the replacement fuses to ensure that they are for the correct voltage conversion (see below). Rotate the fuse holder 180° to make the voltage conversion. Seat the correct fuses into the clips on the holder. Smaller 5 x 20 mm fuses must be located towards the rear of the fuse holder. Reinsert the power entry fuse holder with the correct voltage showing through the small window.



- Fuse, Corcom 115V, 5 x 20 mm, FAST, 250V, 1.6 A
- Fuse, Corcom 230V, 5 x 20 mm, FAST, 250V, 800 mA
- Fuse, Signal Processor Board, 5 x 20 mm, FAST, 250V, 200 mA
- Fuse, D2 Power Supply, 5 x 20 mm, FAST, 250V, 500 mA

9.3 Fuse Replacement

A total of five fuses protect the DVW-10 system. Two of the fuses are located in the power entry module (Corcom) at the rear of the cabinet and are in series with the AC input line. One fuse is located on the D2 Power Supply. The other two are located on the Signal Processor circuit board [under plastic fuse block covers] and are in series with an on-board transformer.

Troubleshooting the fuses is straightforward. The following table will assist in identifying fuse problem areas.



DANGER. For fuses other than the Corcom, only a qualified technician should work with the electrical system inside the unit.

				<u>SYMPTOM</u>	
Lamp	Fan*	Display	AZ	Check Fuses @	
OFF	OFF	OFF	OFF	Corcom	
OFF	OFF-12V	ON	OFF	D2 Power Supply	
ON	OFF-24V	OFF	OFF	Signal Processor	

*Models have both 12V and 24V fans. See 12V marking on the underside of fan bracket.



DANGER!!! WITH POWER OFF, THE D2 BOARD CAN STILL PRESENT A SHOCK HAZARD. Wait until all three of the red LEDs go out. Illuminated, they indicate that the capacitors are still charged.

If fuses have been changed by a competent technician and service manual troubleshooting cannot correct the electrical problem, a board replacement is probably required. Contact your Dealer/Sales Representative for assistance.



WARNING: Unplug power cord from the Corcom before checking fuses.

To gain access to the Corcom fuses, gently pry off the cover plate with a small flat-bladed screwdriver. Replace with fuses of the correct rating shown below. Smaller 5 x 20 mm fuses must be seated in the rear of the fuse holder. Insert the power entry fuse holder with the correct voltage showing through the small window.



WARNING: Unplug power cord from the Corcom before removing cabinet lid.

10.0 Preparation for Storage or Shipping

10.1 Isopropyl Alcohol Flush

Disconnect the tubing from the column. Insert inlet tubing into Isopropyl Alcohol. Use the syringe to draw a minimum of 50 mL through the flowcell.

10.2 Storage

Clean the detector with an Isopropyl Alcohol solution. Then drain and dry it and its tubing.

Seal the solvent inlet port and outlet ports on the flowcell bulkhead assembly with the nylon flowcell plug shipping screws.

10.3 Shipping

Flush, drain, and dry the detector as above and seal it with the shipping plug screws.

CAUTION: Reship in the original carton, if possible. If the original carton is not available, wrap the system in several layers of bubble wrap and cushion the bottom, top, and all four sides with 2" of packaging foam. Although heavy, the DVW-10 is a delicate instrument and must be carefully packaged to withstand the shock and vibration of shipment.

Section 11.0 Troubleshooting Guide

There are numerous causes and solutions for problems with chromatograms. Only those caused by or affecting the detector are addressed here. Consult your Dealer/Sales Representative for additional troubleshooting references.

You Notice	This May Mean	Possible Cause	You Should
No power when detector turned ON.	Blown fuses in the power entry module.	1. Power surge, failed component. 2. Wall outlet dead.	1. Replace only with the appropriate fuses (See Section 9). 2. Check wall outlet and building circuit breakers.
Front panel appears OK but detector does not work.	Blown fuse on the circuit board.	1. Power surge. 2. Internal short.	1. Replace only with the appropriate fuse (See Section 9). 2. Check board (Maintenance Tech).
Fittings or components leak.	Interference with fittings seal	1. Film of fluid between surfaces. 2. Salt crystals between surfaces. 3. Scratches in mating surfaces.	1. Clean and dry mating surfaces. 2. Clean and dry mating surfaces. 3. If scratched, replace defective part.
Flowcell leaks	1. Cell gasket 2. Cracked windows 3. Leaky fittings 4. Blocked outlet line	1. Over pressure limit. 2a. Over pressure limit. 2b. Cell re-assembled incorrectly. 3. Interference with seals. 4. Lodged particles or tubing crimped.	1a. Reduce excess backpressure. 1b. Clean and reseal. 1c. Replace cell gasket. 2. Reduce excess backpressure and replace cell window. 3a. See above. 3b. Dry out cell reference path. 4a. Clear tubing (backflush). 4b. Replace tubing.
Baseline Drift/Noise	1. Column 2. Mobile Phase 3. Flowcell 4. Detector 5. Recorder input voltage	1a. Column temperature. 1b. Equilibrium. 2a. Not homogeneous. 2b. Mixing problem. 2c. Contaminated/Age. 2d. Recycled. 3a. Air bubbles. 3b. Contamination. 3c. Leaks. 3d. Loose assembly. 4a. Absorbance setting. 4b. External device. 4c. Insufficient warmup time. 5a. Wrong setting. 5b. 1 mV Recorder. 5c. 100 mV Recorder	1a. Regulate - Consult column guide. 1b. Flush - Consult column guide. 2a. Degas/sparge; replace. 2b. Correct flow. 2c. Replace. 2d. Reset detector baseline - AZ 3a. Apply backpressure (100 psi); flush with strong solvents; check for leaks in the system. 3b. Flush; Clean cell windows. 3c. See above. 3d. Tighten bulkhead assembly. 4a. Reset wavelength to maximum absorbance. 4b. Check electrical lines for interference; check for ground loops, especially recorder and data system hookup. 5a. Set recorder to normal input: 10 mV. 5b. Reduce sensitivity (range 3 steps down). 5c. Increase sensitivity (range 3 steps up).
Spurious noise in the detector readout	1. Bad lamp. 2. External influence	1a. Dirty lamp. 1b. Lamp at end of useful life or is damaged in transit. 2. Temperature fluctuation	1a. Clean lamp (see Lamp section). 1b. Change/replace lamp (see Lamp section). 2a. Attempt to stabilize ambient temperature. 2b. Avoid hot and cold drafts. Check for overhead air ventilators.
Peak size	1. Too small. 2. Too large.	1. Attenuation too high. 2. Attenuation too low.	1. Reduce attenuation (increase range sensitivity). 2. Increase attenuation (decrease range sensitivity).
Autozero Fails	Flowcell imbalance	1. Flowcell not inserted correctly 2. Flowcell dirty/contaminated. 3. Liquid in reference side.	1. Remove flowcell & reinstall per Appendix directions. 2. Flush; flowcell windows. 3. Flush (if non-volatiles in mobile phase); dry thoroughly.

Section 12.0 Accessories, Replacement and Spare Parts

	PART NO.	NOTES
ENERGY SOURCES		
Lamp Assembly, Deuterium (D2), Prealigned, Variable UV (195-360 nm)	025-0078	RSP
Lamp Assembly, Tungsten (W), Prealigned, Variable Vis (360-800 nm)	025-0076	RSP
FLOWCELL ASSEMBLIES		
Analytical: 7mm, 10 µL, 1/16" tubing	025-0077	RSP
Preparative: 2.0 mm, 4 µL, 1/8" or 1/16" tubing	025-0074	RSP
Analytical: 7mm, 10 µL, STAINLESS STEEL, 1/16" tubing	025-0123	
FLOWCELL FITTINGS & TUBING (see also Flowcell Appendix)		
Nut, Flangeless, Delrin, 1/16" ID (Analytical) 10pc/pkg	025-0047	RSP
Ferrule, Flangeless, Tefzel, 1/16" ID 10pc/pkg	025-0048	RSP
Nut, Flangeless, Delrin, 1/8" ID, X-Long (Prep) 10pc/pkg	025-0090	RSP
Ferrule, Flangeless, Tefzel, 1/8" ID (Prep) 10pc/pkg	025-0052	RSP
Nut, Flangeless, Delrin, 1/16" ID, X-Long (Prep) 10pc/pkg	025-0089	
Nut, Bulkhead, Analytical Flowcell Assy 2pc/pkg	Contact Dealer	
Male nut, 1/16" (for Analytical bulkhead fitting) 10pc/pkg	025-0012	RSP
Ferrule, double-ended, 1/16" (for Anal bulkhead fitting) 10pc/pkg	025-0013	RSP
Tubing, TFE Teflon, 1/16" OD, 0.010" ID 5ft	025-0066	RSP
Tubing, TFE Teflon, 1/16" OD, 0.030" ID 5ft	025-0050	
Tubing, TFE Teflon, 1/8" OD, .062" ID, Black (Prep) 5ft	025-0054	
Tubing, PEEK, 1/16" OD, 0.030" ID 5ft	025-0049	
Tubing, PEEK, 1/16" OD, 0.010" ID 5ft	025-0061	
FUSES:		
Fuse, Signal Process, 5 x 20 mm, FAST, 250V, 200 mA 20pc/pkg	025-0118	RSP
Fuse, D2 Power Supply, 5 x 20 mm, FAST, 250V, 500 mA 20pc/pkg	025-0084	RSP
Fuse, Corcom 115V, 5 x 20 mm, FAST, 250V, 1.6 A 20pc/pkg	025-0083	RSP
Fuse, Corcom 230V, 5 x 20 mm, FAST, 250V, 800 mA 20pc/pkg	025-0124	RSP
RECORDER/INTEGRATOR CABLE	025-0026	
REMOTE CONTROL CABLE-Variable (Includes Recorder/Integrator)	025-0079	
POWER CORD, No. AMERICA, NEMA 5-15P/IEC320	025-0091	
POWER CORD, INTERNATIONAL, CEE 7 STD/IEC 320	025-0108	
PC BOARDS	Contact Dealer	Tech
REPLACEMENT ACCESSORY KIT (CABLE, TOOLS & FUSES)	025-0075	
OPERATOR'S MANUAL (Paper Copy)	050-0004	
DATA ACQUISITION AND CONTROL		
Star-Chrom™ (S-C) HPLC Management System w/ Calibration	015-0013	
S-C Automated Remote Control Cable-Variable (Incl Rec/Integr)	025-0150	
LEGEND: RSP = Recommended spare part Tech = Maintenance Technician Recommended		

Section 13.0 Trademarks

Trademarks used in this manual are for identification purposes only and are the property of their respective trademark owners:

Delrin, Teflon and Tefzel - Du Pont Co.;
PEEK - VICTREX;
Cheminert, PAEK - VICI/Valco;
Omnifit - Omnifit Ltd.;
Corcom - Corcom, Inc.

Appendix B

INSTRUMENT STANDARDIZATION LOG

DATE: _____

OPERATOR: _____

SAMPLE/STANDARD:

CONDITIONS:

RESULTS:

=====

DATE: _____ OPERATOR: _____

RESULTS:

=====

DATE: _____ OPERATOR: _____

RESULTS:

=====

DATE: _____ OPERATOR: _____

RESULTS:

=====

DATE: _____ OPERATOR: _____

RESULTS:

MANUFACTURER'S DECLARATION OF CONFORMITY

D-STAR INSTRUMENTS, INC.
8424 QUARRY ROAD, #203
MANASSAS, VIRGINIA 20110-5326, USA

DECLARES UNDER SOLE RESPONSIBILITY THAT THE FOLLOWING PRODUCT(S):

- DFW-20/21 Fixed Wavelength Detector Series
- DVW-10 Variable Wavelength Detector Series
- DDW-10 Dual Wavelength Detector Series
- DLC-10/11 Integrated HPLC System Series
- DLC-20 Integrated HPLC System Series
- DGP-10 HPLC Gradient Pump Series

Serial Numbers: Valid with assigned D-Star serial numbers.

TO WHICH THIS DECLARATION RELATES ARE IN CONFORMITY WITH THE FOLLOWING STANDARD(S):

- EN55011: 1991 CLASS A, GROUP 1 and EN50082-1: 1997 to include: EN50140; ENV50204; IEC 1000-4-2; IEC 1000-4-4; ENV50142; EN61000-4-11 and EN61010-1

FOLLOWING THE PROVISIONS OF:

EC DIRECTIVE 73/23/EEC; EC DIRECTIVE 89/336/EEC; EC DIRECTIVE 93/68/EEC

An internal production control system to ensure compliance between the manufactured products and the technical documentation is in effect.

These products are marked CE in July 1998

ON BEHALF OF THE COMPANY


Dennis W. Jarzen
VP Product Development

MANASSAS, VIRGINIA USA
July 10, 1998

YEAR 2000 SOFTWARE COMPLIANCE CERTIFICATE (Y2K ✓)

D-STAR INSTRUMENTS, INC.
8424 QUARRY ROAD, #203
MANASSAS, VIRGINIA 20110-5326, USA

DECLARES THAT THE FOLLOWING PRODUCT:

Star-Chrom™ HPLC Management System

Serial Number: Valid with assigned D-Star serial number.

Equipment and software used for the purpose of collecting and processing data, and controlling certain analytical equipment used in high pressure liquid chromatography (HPLC)

TO WHICH THIS CERTIFICATE RELATES IS IN CONFORMITY WITH THE FOLLOWING:

BSI DISC PD2000-1 A Definition of Year 2000 Conformity Requirements

The date may be entered by the user at the start of a run or automatically entered into logs by the software. The year may be entered in two-digit or four-digit format. The performance of the software is not affected by the format. The Year 2000 is considered as a leap year.

This(ese) product(s) is(are) not impacted by the Year 2000, provided the user's Windows 95 Operating System is updated with the Microsoft Y2K modification. Caution may have to be exercised in the case where the dates are reprocessed by other software.


FURTHER, THE FOLLOWING PRODUCTS, USED FOR COLLECTING AND TRANSMITTING ANALOG DATA, ARE NOT DEPENDENT ON SOFTWARE/FIRMWARE AND ARE NOT IMPACTED BY THE YEAR 2000 PROBLEMS:

- DFW-20/21 Fixed Wavelength Detector Series
- DVW-10 Variable Wavelength Detector Series
- DDW-10 Dual Wavelength Detector Series
- DLC-10/11 and DLC-20 Integrated HPLC Systems Series
- DGP-10 HPLC Gradient Pump Series

FURTHER ALL PROGRAMS IN COMPANY BUSINESS SYSTEMS:

Are Windows operating system based and have been updated with Microsoft Y2K modifications.

ON BEHALF OF THE COMPANY


Dennis W. Jarzen
Director, Information Systems

MANASSAS, VIRGINIA USA
JUNE 11, 1998

Appendix A - 1

7mm ANALYTICAL HPLC FLOWCELL BULKHEAD ASSEMBLY

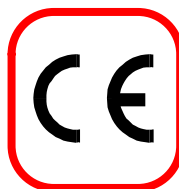
(Variable UV-Vis Detector)

OPERATOR'S MANUAL

Part Numbers

PEEK - DS 025-0077/025-0077-1

Stainless Steel - DS 025-0123/025-0123-1



Appendix A - 1

Operator's Manual
7mm ANALYTICAL HPLC FLOWCELL ASSEMBLY
Part Numbers DS 025-0077 & 025-0123
(Variable UV-Vis Detector)

Contents	Section	Contents
	1	Introduction
	2	Specifications
	3	Unpacking, Orientation, and Installation
	4	Maintenance I - Cleaning
	5	Maintenance II - Disassembly and Reassembly
	6	Troubleshooting
	7	Replacement and Spare Parts
	8	Warranty

Section 1.0 Introduction

This manual outlines the specifications, correct use, and maintenance procedures for the PEEK™ Analytical Flowcell PN 025-0077 (0077-1 Sapphire windows) and the Stainless Steel (SS) Flowcell PN 025-0123 (0123-1 Sapphire windows) used in the Variable Wavelength high performance liquid chromatography (HPLC) detectors. The PEEK flowcell is bio-compatible and is intended for use in HPLC applications where bio-inert or neutral materials are called for. The Stainless Steel flowcell is universal for all solvents except for applications requiring bio-compatibility. Sapphire windows should be used when quartz windows will be etched (pH above 8).

Write the name of each flowcell on the table of contents of your instrument manual to provide easy reference to its location. Then, place this manual in Appendix A.

Consult your instrument's Operator's Manual to determine if this flowcell assembly is appropriate for use in a particular detector.

2.0 Specifications - Analytical Flowcell Bulkhead Assembly

Sampling Technique:	Double Beam - Sample and Reference (see Section 3)
Pathlength:	7 mm
Cell Volume:	10 µl
Type:	HPLC
Max Operating Pressure:	200 psi
Wetted materials:	
Cell body:	PEEK™, Stainless Steel
Sealing Gaskets:	Teflon™
Windows:	Fused Silica (quartz); Sapphire (special "A1" cell)
Fittings:	1/16" ID, Tefzel™ Ferrules (PEEK)/ Tefzel™ & SS Rings (SS)
Tubing:	0.015" ID, 1/16" OD (PEEK)/ 0.010" SS Inlet & 0.020" SS Outlet
Bulkhead Union:	PEEK™/SS
Bulkhead Ferrules:	PEEK™/SS
Other Materials:	

Flowcell Housing	Anodized Aluminum
Bulkhead Housing	Anodized Aluminum
Fittings:	Flowcell: Nut, Flangeless 1/16" ID, Delrin™/PEEK™
	Bulkhead: Nut, 10-32, PEEK™/SS
Hardware:	Stainless Steel, Nylon

3.0 Unpacking, Orientation, and Installation

3.1 Packing List

DESCRIPTION	PART NO*	QTY
Analytical Flowcell Bulkhead Assembly, 7mm, 10 µL, 1/16" tubing, PEEK	010-0067 [or 010-0067-5 Sapphire windows]	1 ea or;
Analytical Flowcell Bulkhead Assembly, 7mm, 10 µL, 1/16" tubing, SS	010-0067-1 [or 010-0067-6 Sapphire windows]	1 ea
External Bulkhead Fittings:		
Male nut, 10-32, 1/16" ID, PEEK	250-0043	2 ea
Ferrule, double-ended, 1/16" ID, PEEK	250-0044	2 ea or;
Nut, hex head, 10-32 CPI Seat, 1/16" ID, SS	250-0033	2 ea
Ferrule, 10-32 CPI Seat, 1/16" ID, SS	250-0031	2 ea
Hex Key Wrench for Flowcell Sockethead		
Cap Screws - 3/32"	540-0004	1 ea
Operator's Manual (this document)	090-0013	1 ea

*See Section 7 for Spare Part Numbers

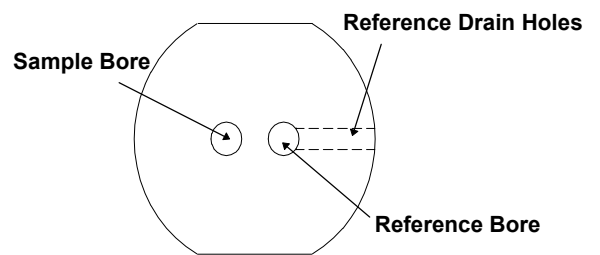
If the Analytical Flowcell was installed in your detector, the fittings and wrench will be in the Accessory Kit.

3.2 Orientation

3.2.1 The Flowcell

NOTE: Numbers in [] after parts refer to the numbers in the "Exploded View of Flowcell" diagram, in Section 5.2 (page A-5) below.

The unique design of the D-Star dual path Analytical Flowcell was developed to maximize HPLC performance while maintaining cost at a reasonable level. The Cell Body [1] is made of PEEK® polymer and is held in place in the Cell Holder [2] by two flat bottom flangeless fittings [6], pressure exerted by the Cell Clamp [3] and the Cell Mounting Plate [4]. Belleville Springs [13] apply balanced pressure on the windows [12]. TFE gaskets [5] seal the windows and body together. The flangeless ferrules [7] of the fittings (or flanged tubing, if so equipped) seal against flat surfaces machined into the cell body.



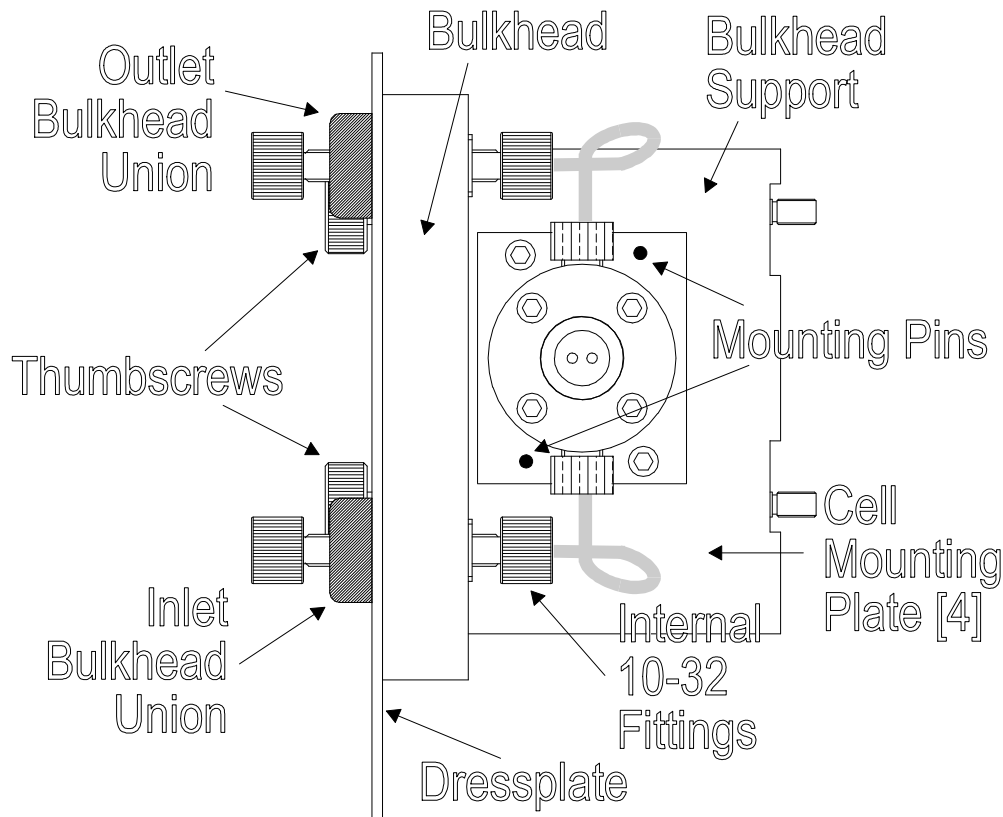
Cell Body [1]

The cell body [1] has two passages for absorbance measurement. One is used for the sample fluid (Sample Bore), the other for a reference (Reference Bore). The sample passage is connected to the fittings by small bore "vias" to allow fluid to flow from the inlet tubing to the

measurement passage and then on to the outlet tubing. An air reference passage has two vent holes (Reference Drain Holes) opening to the outside to equalize pressure with the environment and to prevent vapors that permeate through the gasket from the sample side from accumulating in the reference passage.

3.2.2 The Bulkhead Assembly

The entire Flowcell Bulkhead Assembly, with mounted Analytical flowcell, is shown below:

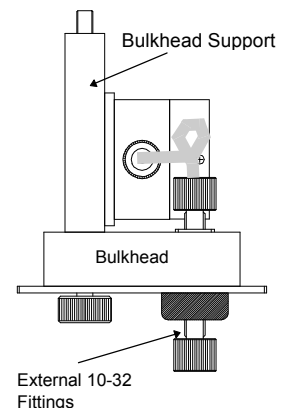


Analytical Flowcell Bulkhead Assembly

3.3 Installation

Two nylon flowcell plug shipping screws are installed on the analytical flowcell bulkhead union inlet and outlet ports prior to shipping to prevent flowcell contamination and to protect the bulkhead fittings from damage. Remove the plug shipping screws and save them for future shipping or storage. The bulkhead fittings are located in a marked bag. If the Analytical Flowcell was installed in your detector, the fittings will be in the Accessory Kit.

The flowcell is pre-aligned on the bulkhead assembly. Prior to mounting the assembly into the detector, ensure that the dry cell is clean and that the fittings are tight.



Analytical Flowcell Bulkhead Assembly Top View

Insert the flowcell assembly into the detector flowcell housing carefully. The flowcell is guided in place by two pins inside the housing. Once the pins are aligned in the holes, begin screwing in the Thumbscrews clockwise. Alternate each screw after a few turns to make sure the Support plate is advancing evenly. If one of the screws binds, back it off and retry using fewer turns. If both screws are snug and the Dressplate is flush against the cover/cabinet lid, the flowcell is mounted correctly.

If there is a gap between the Dressplate and the cover, or the cell is tilted, remove the assembly, check for obstructions, and then restart the mounting process.

It is recommended that a backpressure regulator be installed on the outlet fluid line of the detector to prevent air bubbles in the flowcell. Use a 40 -100 psi regulating device. See also Section 6.

3.4 Chemical Incompatibility

It is the user's responsibility to determine which solvents are compatible with the materials of construction listed in Section 2 above. Please note that the following advisories have been provided by suppliers of parts and materials:

PEEK - **Concentrated Nitric Acid and concentrated Sulfuric Acid** attack PEEK chemically and will cause severe effects. **NOT RECOMMENDED.**

Methylene Chloride (MeCl), DMSO, and THF cause PEEK to swell. **MeCl** has been observed to cause flaking. **NOT RECOMMENDED.**

FUSED SILICA (windows)- Avoid high pH solvents (8.0 - 8.5+) which will etch the window (synthetic quartz). High pH solutions may be used for short periods of time (one minute) to clean protein buildups.

SAPPHIRE (windows)- The alpha form monocrystal is non-porous and unaffected by any weathering, hydration, solvents, or mineral acids at room temperature. It is slowly etched by hydrofluoric or phosphoric acid and strong caustics at temperatures exceeding 600° C.

TEFZEL - Some **chlorinated chemicals** may cause swelling.

DELTRIN - Not suitable for use with **acids, bases, or oxidizing agents** [This is usually a non-wetted part in normal operation].

Contact your Dealer/Representative or D-Star Instruments for information on other flowcells which may be better suited for your application.

Section 4.0 Maintenance I - Cleaning the Flowcell

Use a syringe to clean the flowcell and fittings with solvents or other chemical solutions. DRAW THE LIQUID into the cell. DO NOT FORCE the cleaning solution into the cell. A luer adapter such as Upchurch P-658 or P-659 attached to the flowcell tubing facilitates connecting the syringe, thereby reducing the likelihood of leaks.

Use only cleaning solvents compatible with the materials of construction listed in Section 2 above.
Avoid the solvents mentioned in Section 3.4 above.

If the flowcell must be disassembled for cleaning or to replace items, see Section 5 below.

Section 5.0 Maintenance II - Disassembly and Reassembly

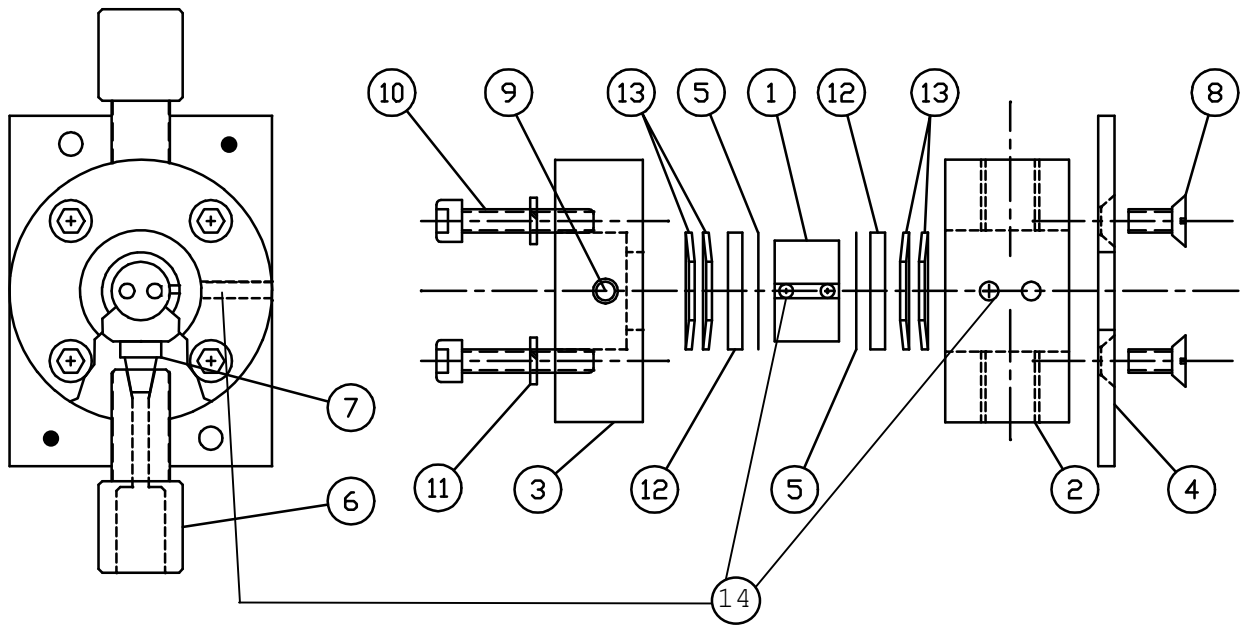
5.1 Tools Required

3/32" Hex Key Wrench - included with unit
 Lens Tissue
 Tweezers - to handle Body, Windows, Gaskets,
 Bellevilles

Cleaning Solvent
 Spray Dust Remover Can (Pressurized Air)

5.2 Disassembling the Flowcell

NOTE: Numbers in [] after parts refer to the numbers in the *Exploded View of Flowcell* diagram, below.



DS010-0003
Exploded View of Flowcell

ITEM*	QTY	DESCRIPTION
1	1	Flowcell Body, 7mm Path, 10 μ L Vol, PEEK®/SS
2	1	Cell Holder
3	1	Cell Clamp
4	1	Cell Mount
5**	2	Cell Gasket, 0.005" Thick, TFE®
6	2	Nut, Flangeless, 1/16" Tubing, Delrin® [Short]/Super Flangeless PEEK for SS
7	2	Ferrule, Flangeless, 1/16" Tubing, Tefzel® [PEEK]/Super-Flangeless Tefzel & SS Ring [SS]
8**	4	Screw, Machine, Flathead, Phillips, 4-40 X 1/4" Long, SS
9**	2	Setscrew, Socket Cup, 4-40 x 1/4" Long, SS
10**	4	Screw, Sockethead Cap Screw, 4-40 x 9/16" Long, SS
11**	4	Lockwasher, Split, #4, SS
12**	2	Window, Quartz (or Sapphire), 1/2" Diameter
13**	4	Spring, Belleville, SS
14		Reference Drain Holes

* See Section 7 for Spare Part Numbers ** Included in Flowcell Rebuild Kit, PN 025-0045

1. Identify each part as the flowcell is disassembled with the diagrams and the parts list to successfully rebuild the cell.
2. Disconnect the internal PEEK/SS 10-32 fittings and tubing from the bulkhead unions.
3. Loosen the two 3/8" sockethead cap screws on the mounting plate with the 3/32" wrench included with your unit. Remove the flowcell from the Bulkhead Support. Remove the flowcell fittings (and the PEEK/SS fittings and tubing) and set them aside.
4. Loosen and remove the four flowcell sockethead cap screws [10] with the 3/32" wrench. Remove the Cell Clamp [3] and set it aside. Turn the flowcell over and carefully dump the parts onto a soft surface. Separate the Cell Body [1] from the other parts. *Note the orientation of the parts, especially the belleville springs, for reassembly.*

5.3 Cleaning the Flowcell Parts

1. Clean the aluminum parts with 50/50 mixture of Isopropanol and water. Dry the parts with a lint-free towel.
2. Clean the Cell Body [1], the belleville springs [13], the windows [12], and the Teflon gaskets [5] in the IPA/water solution. Leave the gaskets in the solution until assembly. Dry all parts. Ensure that they are clean and lint- and particle-free.
3. Inspect the parts, particularly the windows. If any parts need to be replaced, order the Flowcell Rebuild Kit, PN 025-0045.

5.4 Reassembling the Flowcell

1. Insert two belleville springs [13] into the Cell Holder **with the spring curvature pointing up** (away from the Cell Mount [4]). Next, insert a clean window [12]. Avoid touching the faces of the window, and ensure that it is particle-free.
2. Place a gasket [5] on the window with the holes oriented as in the diagram. Place the PEEK Cell Body [1] over the gasket. Check the alignment of the gaskets with the sample and reference holes on the Cell Body, and the Reference drain holes [14] of the Cell Holder with those of the Cell Body [14]. If necessary, use a thin, dull wire inserted into the Reference Drain Holes to hold the Cell Body aligned in place.
3. Place the other gasket on the Cell Body and align the holes. Carefully place the second window over the gasket. Then place the remaining two belleville springs on the center of the window, **with the curvature down**, facing the Cell Body.

IMPORTANT: The **raised inner edge** of the two pairs of belleville springs [13] must face inward toward the windows. If installed raised portion out, the windows may break.

4. Insert the sockethead cap screws [10] and lockwashers [11] in the Cell Clamp [3].
5. Place the Cell Clamp on the bellevilles and finger-tighten the cap screws. Using the 3/32 hex wrench, tighten the cap screws, alternating corners (gradual crisscross fashion) to apply equal force. Check the belleville springs to ensure that they are centered. Tighten the cap screws until resistance is encountered. Overtightening will not make the cell seal any better. While this is not likely to break the windows, it could break off the screw or strip the cell holder threads. Dust the parts to remove any metal particles from the screws.
6. Replace the flangeless ferrules and nuts.

NOTE: For the SS flowcell, the Inlet tubing (lower) should be 0.010" ID (blue tag) and the Outlet tubing (upper) should be 0.020" ID, (yellow tag).

7. Pressure test the flowcell to 200 PSI if possible. If the cell leaks, disassemble it and clean and dry the components. Reassemble the parts and heat the cell at 200° F for eight hours [use a "cookie sheet" in a pre-heated oven]. Pressure test as above.
8. Reconnect the tubing and the fittings to the flowcell.
9. Push the Cell Mount on to the Bulkhead Support pins with the reference holes to the right. Press firmly and evenly to obtain a snug fit.
10. Secure the flowcell to the Support with the 3/8" sockethead cap screws and lockwashers.
11. Reattach the 10-32 PEEK or SS fittings and tubing to the bulkhead unions.

Section 6.0 Troubleshooting

6.1 Preventing Leaks

Although we do not generally expect problems which may be associated with flowcell leakage, we do offer the following information. For our discussion of potential leak problems please refer to the illustration "Exploded View of Flowcell" in Section 5 above. To understand the effects of leaks caused by loose fittings, exceeding pressure limits of the cell and gasket seal failure, it is important to review the nature of the design of the flowcell in Section 3.2 Orientation.

If liquid leaks from either of the fittings or from a poor gasket seal, liquid can accumulate between the cell body and the cell holder. If the liquid does not evaporate quickly, some of it can enter the Reference Drain Holes of the reference passage and eventually get into the reference bore side of the cell. The result is an unstable baseline (drift), often causing off scale readings. Buffers can cause additional problems because of evaporation. Residues in the reference side are not easily flushed out and may require disassembly to adequately clean the flowcell.

The most common causes of leaks can be prevented. **Do not remove or loosen the fittings while there is liquid in the tubing.** Sufficient liquid can get between cell components to cause it to get into the reference side. The best way prevent this is to **dry out the cell before removing the fittings** from the cell. A plastic disposable syringe (10 to 50 ml) may be used to draw liquid out of the sample side of the cell. A luer to 1/16" tubing adapter (Upchurch P658) is useful to make a leak-tight connection to the outlet tubing and allows the syringe to easily remove liquid from the cell. **Disconnect the inlet tubing from the outside of the bulkhead fitting before attempting to draw the liquid out.**

Over-pressure of the liquid in the cell is another cause of leaks. In this design, the windows rarely break from over-pressure. Usually a leak occurs that will heal upon release of the pressure. The healing time could, however, take a few days to achieve the rated sealing pressure. **Do not block the flow from the outlet tubing in an attempt to clear bubbles.** Excessive pressures are likely to result. **Use degassed solvents and a backpressure regulator** (75 psi, spare PN 025-0184) to prevent bubbles from interfering with operation of the cell.

Using the wrong size or type of fitting will probably result in a leak. The flowcell is designed to use 1/4-28 flangeless or super-flangeless fittings.

If liquid does get into the reference passage, ensure the fittings are tight. Then attempt to remove the liquid by using inert gas to flow liquid from around the cell parts and out of the reference vents and passage. Allow the cell to dry. If the liquid remains in the cell or residue is evident, the flowcell will need to be disassembled and dried out and /or cleaned.

The cell must be reassembled exactly as detailed in Section 5.4 above.

IMPORTANT: The **raised inner edge** of the two pairs of belleville springs (13) must face inward toward the windows. If installed raised portion out, the windows may break. Avoid getting finger marks, dust and lint on the cell body, windows or gaskets. Re-tighten the hex socket screws in a gradual crisscross fashion until snug. Overtightening will not seal the cell any better. While this is not likely to break the windows, it could break off the screw or strip the cell holder threads.

6.2 Troubleshooting Table

OBSERVATION	POSSIBLE PROBLEM	POSSIBLE CAUSE	POSSIBLE SOLUTION
Fittings or components leak.	Interference with fittings seal	1. Film of fluid between surfaces. 2. Salt crystals between surfaces. 3. Scratches in mating surfaces.	1. Clean and dry mating surfaces. 2. Clean and dry mating surfaces. 3. If scratched, replace defective part.
Flowcell leaks	1. Cell gasket 2. Cracked windows 3. Leaky fittings 4. Blocked outlet line	1. Over pressure limit. 2a. Over pressure limit. 2b. Cell re-assembled incorrectly. 3. Interference with seals. 4. Lodged particles or tubing crimped.	1a. Reduce excess backpressure. 1b. Clean and reseal. 1c. Replace cell gasket. 2. Reduce excess backpressure and replace cell window. 3a. See above. 3b. Dry out cell reference path. 4a. Clear tubing (backflush). 4b. Replace tubing.
Baseline Drift/Noise	Flowcell	a. Air bubbles. b. Contamination. c. Leaks. d. Loose assembly.	a. Apply backpressure (100 psi); flush with strong solvents; check for leaks in the system. b. Flush; Clean cell windows. c. See above. d. Tighten bulkhead assembly.
Spurious noise in the detector readout	1. Lamp. 2. External influence	1a. Dirty lamp. 1b. Lamp at end of useful life or is damaged in transit. 2. Temperature fluctuation	1a. Clean lamp (see Lamp section). 1b. Change/replace lamp (see Lamp section). 2a. Attempt to stabilize ambient temperature. 2b. Avoid hot and cold drafts. Check for overhead air ventilators.
Peak size	1. Too small. 2. Too large.	1. Attenuation too high. 2. Attenuation too low.	1. Reduce attenuation (increase range sensitivity). 2. Increase attenuation (decrease range sensitivity).
Display flashes "0000"	1. High Absorbance 2. Lamp 3. Flowcell	1a. Contaminated Mobile Phase. 1b. Concentration exceeds detector capacity. 2. Lamp failure. 3. Flowcell improperly seated.	1a. Replace with new mobile phase(s). 1b. Reduce sample size. 2. Check lamp circuitry. Replace if necessary. 3. Reinsert flowcell assembly, evenly tightening both thumbscrews.
Displays high negative absorbance value	1. Leak 2. Startup	1. Liquid in the reference bore. 2. Not AutoZero'd.	1. See above. 2. AutoZero the detector.

7.0 Replacement and Spare Parts

<u>PART</u>	<u>PART NO.</u>	<u>NOTES</u>
Replacement Assy, PEEK, Analytical: 7mm, 10 μ L, 1/16" tube	025-0077	RSP
Replacement Assy, SS, Analytical: 7mm, 10 μ L, 1/16" tube	025-0123	RSP
Flowcell Rebuild Kit, Analytical 7mm, 10 μ L [see below]	025-0045	RSP
Flowcell Assembly Fittings & Tubing		
Nut, Flangeless, Delrin, 1/16" ID (PEEK) 10pc/pkg	025-0047	RSP
Ferrule, Flangeless, Tefzel, 1/16" ID (PEEK) 10pc/pkg	025-0048	RSP
Nut, Super-Flangeless, PEEK, 1/16" ID (for SS) 10pc/pkg	025-0166	RSP
Ferrule, Super-Flangeless, Tefzel/SS , 1/16" ID (for SS) 10pc/pkg	025-0167	RSP
Nut, Bulkhead, Analytical Flowcell Assy 2pc/pkg	Contact Dealer	
Male nut, 1/16" (for PEEK bulkhead) 10pc/pkg	025-0012	RSP
Ferrule, double-ended, 1/16" (for PEEK bulkhead) 10pc/pkg	025-0013	RSP
Male nut, 1/16" (for SS bulkhead) 10pc/pkg	025-0043	RSP
Ferrule, 10-32 CPI, 1/16" (for SS bulkhead) 10pc/pkg	025-0041	RSP
Tubing, TFE Teflon, 1/16" OD, 0.030" ID 5ft	025-0050	
Tubing, PEEK, 1/16" OD, 0.030" ID 5ft	025-0049	
Tubing, PEEK, 1/16" OD, 0.010" ID 5ft	025-0061	
Tubing, SS, 1/16" OD, 0.010" ID, 10 cm [FC Inlet] 10pc/pkg	025-0174	
Tubing, SS, 1/16" OD, 0.020" ID, 10 cm [FC Outlet] 10pc/pkg	025-0175	
Tubing, SS, 1/16" OD, 0.030" ID 5ft	025-0172	
Tubing, SS, 1/16" OD, 0.010" ID 5ft	025-0173	

LEGEND: RSP = Recommended spare part

Flowcell Rebuild Kit (Analytical) Parts List [025-0045]

2 EA	Cell Gasket, 0.005" Thick, TFE®
5 EA	Screw, Machine, Flathead, Phillips, 4-40 X 1/4" Long, SS
3 EA	Setscrew, Socket Cup, 4-40 x 1/4" Long, SS
5 EA	Screw, Sockethead Cap Screw, 4-40 x 9/16" Long, SS
5 EA	Lockwasher, Split, #4, SS
2 EA	Window, Quartz, 1/2" Diameter
5 EA	Spring, Belleville, SS

NOTE: Consult factory for Sapphire window cell information [025-0077-1 PEEK & 025-0123-1 SS].

8.0 Warranty

The flowcell, and tubing supplied with the flowcell, are warranted at the time of installation only.

No other warranty exists, expressed or implied, except as shown here and in the D-Star Instruments' Conditions of Sale. D-Star disclaims any implied warranties of merchantability and fitness for a particular application or purpose.

Fitness to a particular application must be determined by the user.

Request a Return Authorization (RA) before returning any parts.

Return defective unit to your Dealer or Sales Representative for repair. Clean/sterilize all components prior to shipment. No parts will be accepted which present a health or safety concern such as radioactive or biohazard contamination. Repaired unit will be returned via parcel service or mail.

Guide to Using Star-Chrom HPLC Management Software

*Star-Chrom*TM HPLC Management System Software



D-Star Instruments
8424 Quarry Rd.
Manassas, VA 20110-5326
Phone: 703-335-0770
Fax: 703-335-9952
Toll-Free (USA): 800-dstar12
E-Mail: dstarinstr@aol.com
Web-site: <http://www.d-star.com/>

MAY 21, 2000

COMPUTER CONTROL OF THE STAR-CHROM HPLC MANAGEMENT SYSTEM.....	5
USER INTERFACE.....	5
MENU OPTIONS	5
<i>Method File</i>	<i>6</i>
New method.....	6
Open Method File.....	6
Save Method File.....	6
Save Method File As.....	6
Exit Program.....	6
<i>Ini File.....</i>	<i>7</i>
Open Ini File.....	7
Save Ini File.....	7
Save Ini file As.....	7
<i>Columns & Solvents.....</i>	<i>7</i>
New Col & Sol Params.....	8
Open Files.....	8
Edit Columns File.....	9
Edit Solvents File	9
<i>Peak Picking.....</i>	<i>10</i>
New Peak Picking Parameters	10
Open Peak Picking Parameters File.....	10
Save Peak Picking Parameters File.....	10
Save Peak Picking Parameters File As.....	10
Remove Vertical Lines from Plot	10
Remove All Events from Plot	10
Erase the Plot Coordinates Table.....	11
Perform Peak Picking	11
<i>Data File.....</i>	<i>11</i>
Save Data in Plot	11
Print Plot.....	11
Read Data File into Plot.....	11
Write Plot to Windows Metafile	12
Save Data File as ASCII Text	12
<i>Show</i>	<i>12</i>
Show Solvent Composition	12
Change Peak Slope and Threshold	13
<i>Resolution.....</i>	<i>13</i>
<i>Plates.....</i>	<i>14</i>
<i>Manual Areas</i>	<i>14</i>
<i>About</i>	<i>14</i>
DIRECT MODE BAR	15
<i>AutoZero.....</i>	<i>15</i>
<i>Control Buttons</i>	<i>15</i>
<i>Recycle.....</i>	<i>15</i>
<i>Solvent Saver.....</i>	<i>16</i>
<i>Injection.....</i>	<i>16</i>
<i>Pause</i>	<i>16</i>
<i>Start run.....</i>	<i>16</i>
<i>Stop run</i>	<i>17</i>
<i>Reset Alarms.....</i>	<i>17</i>
<i>Messages</i>	<i>17</i>
METHOD SETUP - GENERAL CONDITIONS	18
<i>Conditioning time</i>	<i>18</i>
<i>Separation time.....</i>	<i>18</i>
<i>Detector maximum.....</i>	<i>19</i>

<i>Operator</i>	19
<i>Method time & date</i>	19
<i>Frequency of data measurements</i>	19
<i>Detector Wavelength</i>	19
<i>Detector ID</i>	20
<i>Test mode</i>	20
<i>Check method</i>	20
<i>Zero Detector before Every Run</i>	21
<i>Use Commentary</i>	21
<i>Use two detectors</i>	21
SAMPLES	21
<i>Use</i>	21
<i>Sample ID</i>	22
<i>Sample Concentration</i>	22
<i>Sample dissolved in</i>	22
COMMENTS	23
<i>Method Comments</i>	23
<i>Comments for cycle of run</i>	23
DATA LOGGING.....	24
<i>Save data to disk after run</i>	24
<i>Print chromatogram after run</i>	25
<i>Print in Black/White</i>	25
<i>No data file as yet</i>	25
<i>Select data directory - current directory</i>	25
<i>Select data directory - disk to show directories of</i>	26
<i>Select data directory - files to show in files box</i>	26
<i>Select data directory - Add new directory</i>	26
<i>Select data directory - directories</i>	26
<i>Select data directory - files</i>	27
MTLS - COLS & SOLS	27
<i>Column 1</i>	27
<i>Solvent Bottle 1-2</i>	27
SOLVENT PROGRAM.....	28
<i>Initial Solvent %s</i>	28
<i>Solvent Program</i>	28
Time.....	29
%A, %B.....	29
<i>Current Solvent Table Values</i>	29
Solvent Table Time.....	29
Isocratic	30
%A, %B.....	30
<i>Solvent saver threshold</i>	30
<i>Detector to use</i>	30
<i>Flow Program</i>	31
<i>Initial Flow</i>	31
<i>Flow Program Table</i>	31
Time.....	31
Flow.....	31
PEAK PICKING	31
<i>Peak Picking Parameters</i>	32
Peak threshold	32
Minimum slope.....	32
Peak maximum	33
Range for Begin.....	33
Range for Valley.....	34
Range for End.....	34

# Points in Slopes	34
Min Area %	35
<i>Use peak picking</i>	35
<i>Use peak picking sounds</i>	35
<i>Detector to use</i>	35
<i>Pick Peaks in Current Chromatogram</i>	35
ALARMS	36
<i>Alarm limits</i>	36
Pump pressure - low	36
Pump pressure - high	36
<i>Alarms status</i>	36
Pump pressure low.....	37
Pump pressure high	37
<i>Allow check of alarms</i>	37
<i>Alarm reset</i>	37
<i>Audible alarms</i>	37
HISTORY LOG	38
PLOT (1) OF DETECTOR(S), EVENTS FIRED, PEAKS FOUND.....	38
PLOT (2) SOLVENT PROGRAM AND PRESSURE	39
SCALES & ZEROS	40
<i>Scales and zeros</i>	40
detector 1 scale - applied to readings	40
detector 1 zero - applied to readings.....	40
detector 2 scale - applied to readings	40
detector 2 zero - applied to readings.....	40
pressure scale - applied to readings	40
pressure zero - applied to readings	40
Dstar Detector Wavelength scale - applied to readings	40
Dstar Detector Wavelength zero - applied to readings	40
HARDWARE	41
Maximum flow of solvent pump in mLs/min.....	41
Maximum pressure reading for pressure transducer	41
Com port for hardware control	41
dead volume in mLs - mixer to injector.....	41
directory for sound files for notifying operator.....	41
Click here to read new parameters from the scales & zero screen and the hardware screen	42
PEAK IDS	42
Peak ID Parameters button	42
Reprocess Chromatogram in Plot	42
PEAK RESULTS	43
Peak Options button	43
Peak Areas	43
Plate Counts.....	43
Peak Resolutions	43
CALIBR	44
Calibration DB Files.....	44
Send Record to Peak IDs	44
REPORT	45
DESCRIPTION OF AUXILIARY FILES	45
Solvent File.....	45
Columns file	45
DStarLCMicro.ini.....	46
Peak-picking parameter file	47

COMPUTER CONTROL OF THE STAR-CHROM HPLC MANAGEMENT SYSTEM

User Interface

The user interface is used to input a set of parameters specifying how the apparatus is to be controlled during a separation. A set of control parameters which are acceptable to the program is called a method. Methods may be saved to and read from disk.

When the program first begins, it looks for a method called **startup.lcm** in the same directory where the program file resides. The file extension lcm denotes a method for liquid chromatography.

The program then looks in the same directory for a file called **DStarLCMicro.ini** from which it will read the parameters needed to connect the hardware interface to the computer. The file extension ini denotes a set of initialization hardware parameters.

Other needed files are :

PSLCRun.Nbr containing the latest in the series of unique run numbers used in creating the name for a data file.

To change a parameter in the method, position the cursor in the appropriate box, type the new information and either 1) press the Enter key, or 2) move the cursor to another box. Only then will the method be updated.

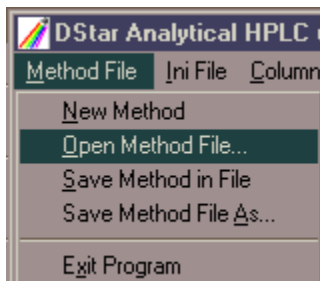
Menu Options



Many of these menu options are very similar because they provide a mechanism of reading and writing files which contain run parameters (methods), solvent details, column details, peak-picking parameters, and hardware parameters (Ini files). The remaining options include reading in previously collected data files, using the "About" box to see the program version and the free space on the hard disk(s). These options will now be explained.

Method File

This menu option allows you to read and save the parameters required to conduct a separation and, using the Exit option, exit the program. The method information consists of sample and sample solvent IDs, the times for equilibration, conditioning, and separation, the mode of sample injection, documentation of the columns and solvents used, solvent program to use as the run progresses, the peak-picking parameters and Solvent Saver parameter, and so on. This is the information found on the General Page, the contents of the Comments on Method box on the Comments Page, the Data Logging Page, the Col & Sol IDs Page, the Solvent Prg Page and the Peak Picking Page.



New method

Clears out most of the parameters in the program, except the peak-picking and hardware parameters. Basically zeroes the time-dependent and sample-specific parameters.

Open Method File

Read in a method file from disk.

Save Method File

Save the method parameters to the most recently read-in method filename. The method parameters must pass the method check before they can be written to disk.

Save Method File As...

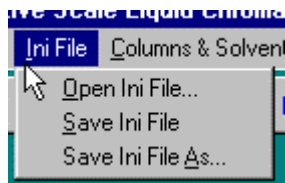
Specify a new filename to save the method parameters to. The method parameters must pass the method check before they can be written to disk.

Exit Program

Exit the program.

Ini File

The hardware initialization parameters are initially read by the program from the DStarLC.ini file and include the scales and zeros to be applied to the various readings.



Open Ini File

Read in a new set of hardware parameters from disk.

Save Ini File

Save the current set of hardware parameters to the current DStarLC.ini file on the disk.

Save Ini file As...

Save the current set of hardware parameters to a new DStarLC.ini file on the disk.

Columns & Solvents

Names of columns and their dead volumes are stored in a file on the disk with extension clm. A typical columns file might be

3.2 C18 5u 4.6mm x 25 cm s/n 341278

3.1 C18 3u 4.6mm x 25 cm

3.0 C8 5u 4.6mm x 25 cm

3.1 C4 5u 4.6mm x 25 cm

3.2 Chirobiotic T

3.3 C18 5u 4.6mm x 25 cm

0 blank

where the dead volume is provided as the first item on each line and is separated from the column name by at least one space. The dead volume is needed when a solvent gradient is being used and the program has to estimate the composition of the solvent mixture at the

detector. Knowing the composition as it leaves the mixer, the dead volumes of the intervening mixer, pump and columns, and the flow rate of the solvent.

A solvents file is merely a list of solvent names, one per line, for example:

10/90 THF/20mM NH4NO3

10/90 MeOH/0.5% NaAc

20/80 ACN/1.0% TEAA pH 4.1

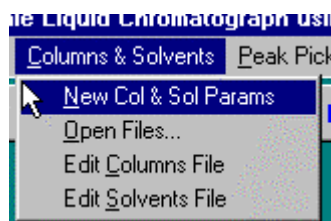
40/60 MeOH/H2O

95/5/0.3/0.2 ACN/MeOH/HOAc/TEA

Ethanol

None

The columns and solvents used in a separation must be documented. The columns and solvents actually used are stored in the Col & Sol IDs page and may be selected from frequently used columns and solvents using the drop-down combo boxes on that page. The choices presented by the boxers are read in from column and solvent files. The column files have extension clm and the solvent files have extension slv. The choices are placed in the drop-down column and solvent boxes by reading in column and solvent files. These files may also be edited using the following menu options.



New Col & Sol Params

Clears the column and solvent names in the program and the interface.

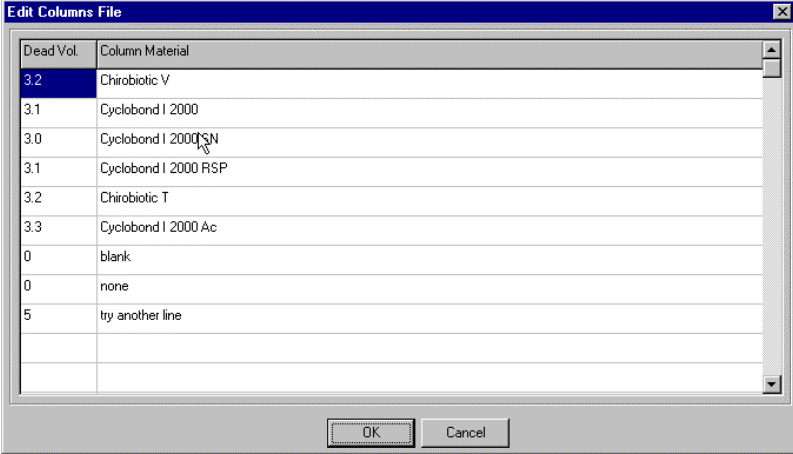
Open Files...

Read new column and solvent names from files on the disk into the program. These names will be put in the

combo boxes on the Col & Sol IDs page. The set being used in the next separation should be selected from the choices in the combo boxes.

Edit Columns File

Selecting this menu option brings up the following form which allows the operator to edit a columns file:

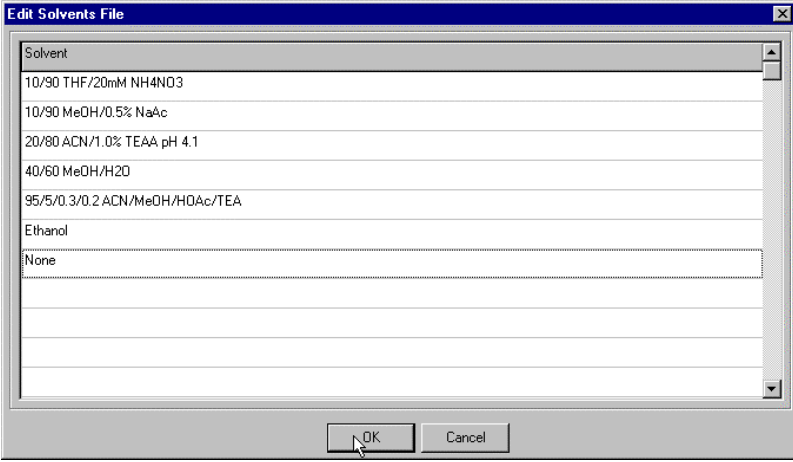


Dead Vol.	Column Material
3.2	Chirobiotic V
3.1	Cyclobond I 2000
3.0	Cyclobond I 2000 RSP
3.1	Cyclobond I 2000 RSP
3.2	Chirobiotic T
3.3	Cyclobond I 2000 Ac
0	blank
0	none
5	try another line

Note that this menu option only edits the file on the disk. If the information is to be used in the program, it must be read into the program using the **Open Files...** option above.

Edit Solvents File

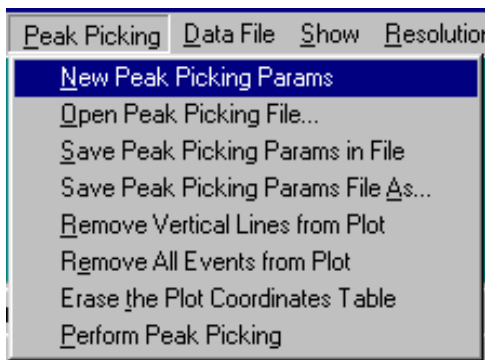
Selecting this menu option brings up the following form which allows the operator to edit a solvents file:



Solvent
10/90 THF/20mM NH4NO3
10/90 MeOH/0.5% NaAc
20/80 ACN/1.0% TEAA pH 4.1
40/60 MeOH/H2O
95/5/0.3/0.2 ACN/MeOH/HOAc/TEA
Ethanol
None

Note that this menu option only edits the file on the disk. If the information is to be used in the program, it must be read into the program using the **Open Files...** option above.

Peak Picking



The first four menu items are concerned with zeroing, reading in and saving peak-picking parameters. The remainder are concerned with testing peak-picking on an existing chromatogram, either newly-measured or read in using the **Data Files** menu option. See the Peak Picking section for a detailed description of what the various peak-picking parameters do.

New Peak Picking Parameters

Clears out the peak-picking page in the program.

Open Peak Picking Parameters File

Read in a peak-picking file from disk.

Save Peak Picking Parameters File

Save the peak-picking parameters to the most recently read-in peak-picking filename.

Save Peak Picking Parameters File As

Specify a new filename to save the peak-picking parameters to.

Remove Vertical Lines from Plot

Putting the coordinates in the grid in the above menu option puts vertical lines on the detector plot where mouse clicks were made. This menu option removes those lines.

Remove All Events from Plot

If tests of the peak-picking scheme and parameters are being made, the peak beginnings, valleys and ends are

shown on the detector plot. Selecting this menu option removes all these marks from the plot so that the results of the latest test can easily be seen.

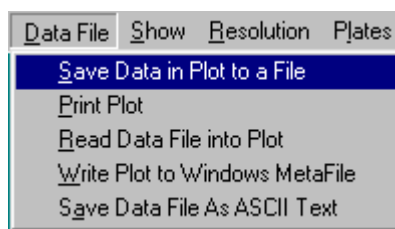
Erase the Plot Coordinates Table

Selecting this menu option erases all the coordinates in the coordinate grid.

Perform Peak Picking

Selecting this menu option forces a test of the current peak-picking parameters on the detector trace currently displayed in the detector plot.

Data File



Save Data in Plot

The normal procedure is to save the data file automatically at the end of a run by checking the "Save data to file on disk after run" check box on the Data Logging Page. If that was not done, the data can still be saved using this menu option.

Print Plot

If the data were not printed out automatically at the end of a run by checking the "Print chromatogram after run" check box on the Data Logging Page, the data can still be printed using this menu option.

Read Data File into Plot

So that methods can be recovered from previously-measured chromatograms and peak-picking can be refined using test cases, it is possible to read chromatograms back into the program using this menu option.

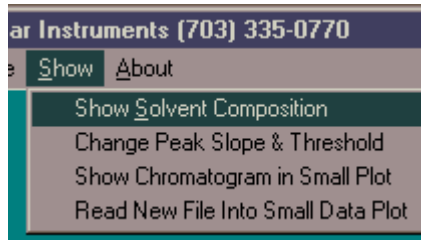
Write Plot to Windows Metafile

A Windows Metafile is a resizable "image". This menu option writes a Windows Metafile of the detector plot to a disk file.

Save Data File as ASCII Text

Exports the current data file to a file in an ASCII text format which can be imported into other programs such as a spreadsheet program.

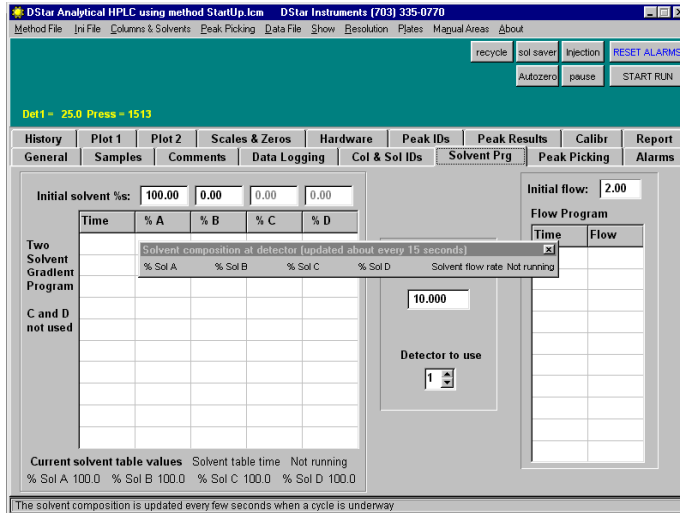
Show



Checking and un-checking these menu options by selecting them and reselecting them brings to the fore various small windows which show some aspect of the current status of the program. These windows are most useful when a separation is underway.

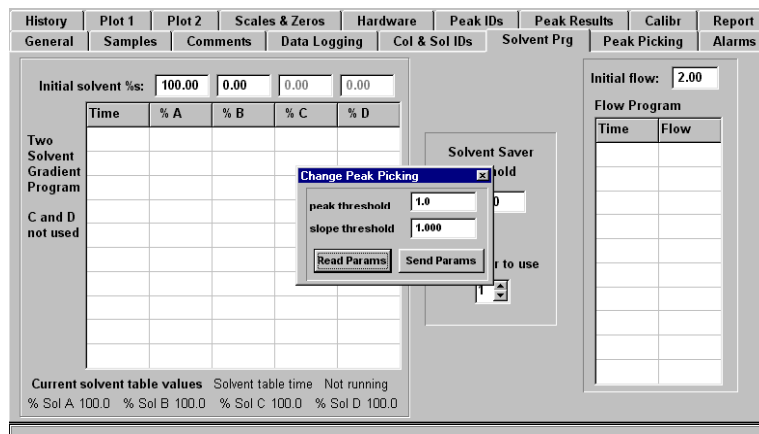
Show Solvent Composition

The solvent composition in a solvent gradient is different in different parts of the chromatograph because the solvent composition mixed at the mixer and pumped through the chromatograph is continuously changing. This pop-up window shows the solvent composition at the detector, estimated using the solvent flow rate and the volume of the chromatograph between the mixer and the detector.

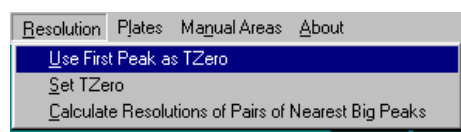


Change Peak Slope and Threshold

Use this menu option to change the peak picking height threshold and slope threshold during a separation. It will be reset to the programmed values at the beginning of the next cycle.



Resolution



Resolution can be calculated using this menu option.

Plates

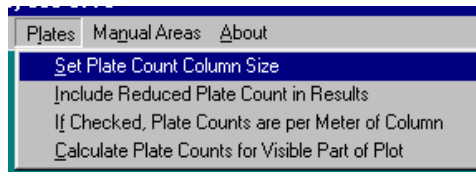
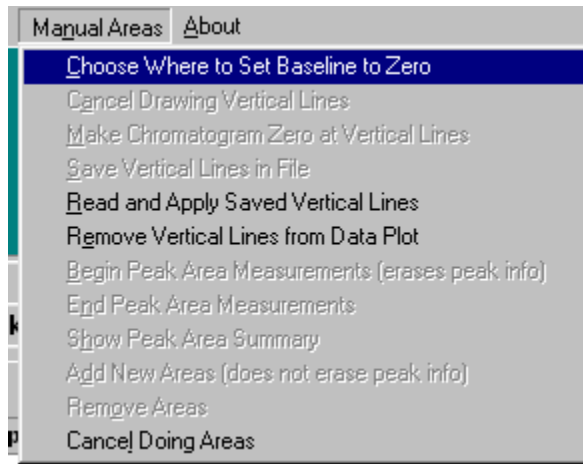


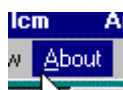
Plate counts can be calculated using this menu option.

Manual Areas

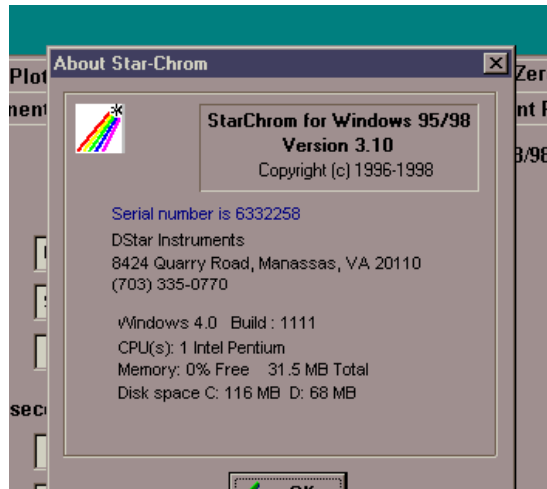


You can manually integrate the areas of peaks using this menu option. You need to first define the baseline. Then you define the time window for integration. The results can replace the Peak Results table or can be added to it.

About

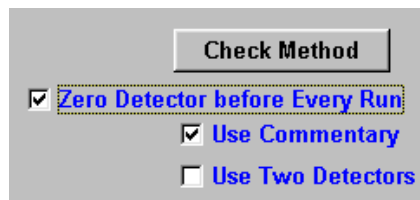


Selecting this menu option brings up an About box.



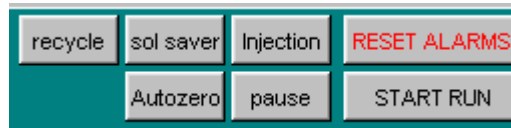
Direct Mode Bar

AutoZero



Auto Zero is selected in the General Page. This event may also be activated under direct control of the operator by clicking on the control button below. When the button is down, the event is "on" and the contact is closed. The status (up or down) is reversed by clicking on the button.

Control Buttons



Recycle

This button shows the current status of recycle valve, down meaning recycling is being used. Recycle is

activated under direct operator control by clicking on the solvent saver button.

Solvent Saver

Solvent Saver is essentially automated recycle controlled by the detector signal. The assumption is that when the detector signal is low the solvent stream is pure and can be re-used. If the solvent system is isocratic, it may therefore be directed back through to the solvent bottle. The threshold detector signal for Solvent Saver is set on the Solvents Page. Solvent Saver may be activated programmatically in the configuration table. The program sets the button when Solvent saver has been activated programmatically. When this button is down, Solvent Saver is being used. Solvent Saver may be activated directly under operator control by clicking on this button.

Injection

When this button is down, an automatic injection is underway. In that case, the button is used as a signal to the operator. Once the run has been "Started" the operator can begin data acquisition by clicking on this button.

Pause

Clicking on this button causes the run to be paused. Clicking on the button when it is down causes the run to be resumed.

Start run

Clicking on this button starts a separation. You must have read in or typed in a method. Perhaps you have already checked the method by clicking on "Check Method" button. The apparatus must be switched on, the sample ready to inject, and the solvent reservoirs filled.

The program will first check the method. If for some reason the hardware parameters have not been read in, you will be prompted for the name and location of the hardware parameter file. If you have specified peak picking and there are no peak picking parameters on the Peak Picking Page, you will be prompted for the name and location of the file containing a set of peak picking parameters. If all these tests are successful, the program

passes to the equilibration stage, followed by conditioning, injection, and separation. The caption of this button changes to Stop Run.

Stop run

If for some reason you do not want to wait until the program comes to a normal stop, clicking on this button will stop the program in its tracks, reset and abandon all remaining injections. If the options 'Save data to disk' and 'Print chromatogram' were selected before the end of the run, these functions are carried out IF more than 30 detector readings have been taken. Otherwise, the run is abandoned without saving or printing any data.

Reset Alarms

The program continuously monitors several alarms (see the Alarms Page). When an alarm has been activated, all program activity stops. An activated alarm must be reset before the run can be resumed. When the cause of the alarm condition has been removed, clicking on this button removes the alarm status (a red message on the alarm page) and signals to the program that it can continue.

Messages

Four messages are used to convey information to the user:

- 1) A continuously updated display of the detector reading(s), the flow and pressure.
 - 2) Status report on the number of the current cycle and the stage in that cycle - equilibration, conditioning, injection, or separation.
 - 3) The latest activity concerning an event - the time it was activated and whether it was switched on or off.
-

Method Setup - General conditions

History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	
Method date & time = 1/29/00 11:54:37 AM								
Conditioning time (min)	1.00					<input checked="" type="checkbox"/> Test Mode		
Separation time (min)	6.00					Check Method		
Time between data points in seconds	1.00					<input checked="" type="checkbox"/> Zero Detector before Every Run		
Detector maximum (mv)	1000.0					<input checked="" type="checkbox"/> Use Commentary		
Det Wavelengths	254		Measured Det Wavelength = 254			<input type="checkbox"/> Use Two Detectors		
Detector ID(s)	DStar Variable Wavelength 7mm path							
Operator	Put your name here							
Running in demo mode								

Conditioning time

The program pumps the initial solvent mixture, as specified in the four edit boxes at the top of the Solvent Program Page, through the columns for the conditioning time. This allows the columns to equilibrate to the initial solvent mixture. It is good practice to use isocratic solvent mixtures as much as possible and, when using a new solvent mixture, to pump several column volumes through the column in the conditioning phase.

Separation time

The separation time is the time from the completion of the conditioning process to the end of the run. The maximum feasible time is determined by the frequency of data collection (see below) and the space allowed in the plot arrays (40,000 points). Thus, at a data collection frequency of once per second (the recommended spacing of data), the maximum run time for which data will be saved is $40000/3600 =$ about 11 hours. At a data frequency of once every 0.2 seconds (5Hz), the maximum run time becomes about 2.2 hours.

The separation time is also used to specify the horizontal axis of the plots. The same axis limits are used for the two stages of separation. The first stage is equilibration and conditioning. The second stage is the injection and separation. The detector maximum sets the limit on the vertical axis of the detector plot. The plot axis lengths can also be changed from the plot page using one of the

buttons over the plot. The two plots can be set to have different limits in that way.

Detector maximum

The purpose of specifying the detector maximum is to set the vertical axis on the detector plot. The plot axis limits can also be changed from the plot page using one of the buttons over the plot. Each plot can be individually set in that way. This does not affect the signal storage. The full dynamic range is stored.

Operator

The operator has to be documented for Good Laboratory Practice. The operator ID is recorded in the data file if one is written to disk.

Method time & date

This is not an editable field. If a method has been read from the disk, this field is used to display the date and time of originally writing the method to disk.

Frequency of data measurements

The spacing of data measurements should normally be near 1 second (the spacing can be specified in fractions of seconds such as 1.1). Data can be collected at up to 5 Hz or 0.2 seconds per point. Using a data collection spacing of two seconds or more, you can collect data for several hours, but large time intervals between the detector readings is not recommended because the peaks will be poorly characterized (they will contain fewer points than normal). This decision will be determined by the peak width.

Detector Wavelength

Enter the desired wavelength into the box and press the enter key. The detector will go to the desired wavelength. Next to it is the actual measured wavelength. You can not change the wavelength once the run is started.

When you first get your system you may need to set the detector zero. To do this enter "0" in the "D-Star detector zero" box found on the Scales & Zeros page. Make sure to press the button at the bottom of the hardware page to

update the value. Next using the up/down arrow keys on the detector go to zero wavelength. Make a note of the “measured det wavelength” value and enter this into the “DStar detector wavelength zero” box found on the Scales & Zeros page. Again, make sure to press the button at the bottom of the hardware page to update the value. Now enter the desired wavelength into the detector wavelength box and press enter. The detector should go to the wavelength within +/- 2 nm. If the wavelength value is +/- 2nm of the value on the front panel of the detector then go to the “INI file” menu and select the “Save INI file”.

Detector ID

This is descriptive information about the detector and is used for documentation.

Test mode

Sometimes one wants to run the program without having the chromatographic hardware attached, either for demo purposes or to become familiar with the program without tying up the apparatus. In normal operation, this box has to be unchecked. When the box is checked the program does not try to control or read from the hardware. If you keep getting the standard test chromatogram, you may have left this box checked!

If the test mode box is checked, the equilibration, conditioning and injection phases will operate “normally” and, in the separation phase, a chromatogram will be synthesized. A pressure profile will also be synthesized.

Check method

Before beginning a run, the program has to check the parameters in the method. It does that when you click on Start Run. You can check the method more innocuously by clicking on Check Method. If a parameter is wrong or inconsistent, the program will tell you in a message box. Clicking the cancel box in the message box stops further testing of the parameters. Selecting OK continues parameter testing. If the method passed inspection, a message box will tell you so.

Zero Detector before Every Run

If this box is checked, the program will send a contact closure to the detector at the beginning of every run. There may be reasons why you should not want to auto-zero the detector. For example if you wanted to monitor the baseline and see if it is changing from run to run.

Use Commentary

If this box is checked, the program announces verbally the general actions it is performing. The announcements are played through speakers attached to a sound card in the computer. This allows the operator to be busy elsewhere in the vicinity and yet follow the progress of the run because the chromatograph is telling what it is doing. The Use Commentary box governs the more general aspects such as start run, stop run and so on. Control over more specific aspects, such as peaks found, or events fired, are located on the pages where those parameters are specified.

Use two detectors

Click on this option if there are two detectors.

Samples

History		Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General		Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	
Click here to read or save the sample list <input type="button" value="Sample Files"/>									
#	Use	Sample ID	Solvent, concentration and injection volume						
1	y	testmix 10uL	1mg/mL, 10 uL						
2	y	testmix 15uL	1mg/mL, 15 uL						
3	y	testmix 20uL	1mg/mL, 20 uL						
4	y	testmix 25uL	1mg/mL, 25 uL						
5									
6									
7									
8									
9									
10									
11									
12									
13									

Use

Enter "Y" or "N". This is used if a series of samples has been run and then one or two must be re-run.

Sample ID

While this label appears to be self-explanatory, it is wise to remember that the information is used to generate the name of the data file (the program uses long file names which contain the sample ID as an important part of the file name). The complete file name consists of A (for analytical HPLC), a number sequentially incremented for each new file, the date and time on the computer's clock when the file was written, the sample ID as specified here, and the extension chr, which allows programs such as the Browse program to present a meaningful set of choices when a file is to be read from the disk.

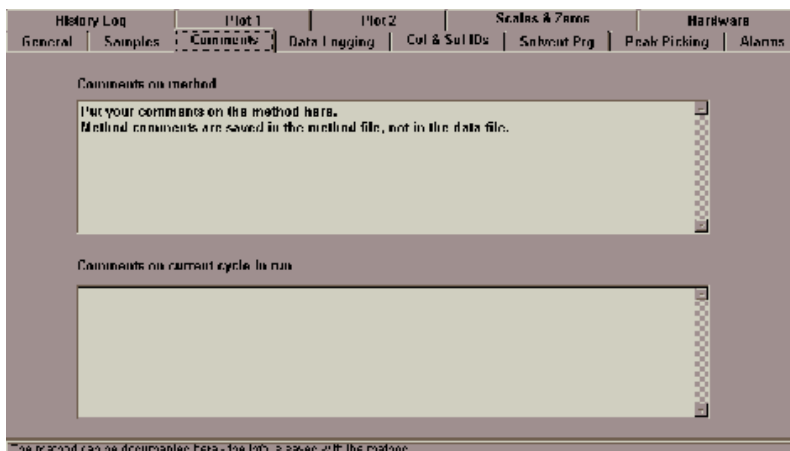
Sample Concentration

The concentration of the sample injected has to be documented for Good Laboratory Practice, so that amount must be provided here, although it is not used by the program other than to record it in the data file if one is written to disk.

Sample dissolved in

The solvent in which the sample is dissolved has to be documented for Good Laboratory Practice, so identification of the solvent must be provided here. The information is not used by the program other than to record it in the data file if one is written to disk.

Comments



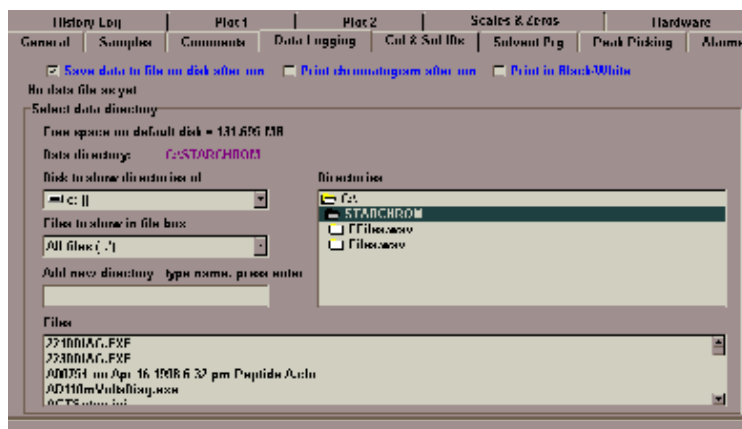
Method Comments

Comments typed into this box remain with the method and document what the method does and what it is intended for. Comments can be changed or added at any time and will be saved with the data if the data are written to disk. The method must be resaved to disk for the comments to reappear the next time the method is read into the program.

Comments for cycle of run

This box is intended for comments which are only relevant to the particular cycle of the run. The comments may be added or edited at any time during the cycle and will be saved with the data if the data are written to disk. The box will be cleared when the next cycle begins.

Data logging



Save data to disk after run

The normal procedure is to save the data to a file on the hard disk. This option gives you a choice. If this box is checked when the program comes to the end of a separation, the program will look for a file called pslcrun.nbr in the directory from which the program was originally started and if the file is found will read from that file a number which it will increment by one and use to synthesize the name of the new data file. All data for a given cycle will be written to the same file.

'All data' consists of

- 1) the sample ID, used as title (and in the name) of the data file
- 2) the method used
- 3) the history log
- 4) any notes you have added to explain this particular run.
- 5) the detector - time traces (up to 6 depending on the detector(s)).
- 6) time traces for the 4 solvents
- 7) the pressure trace
- 8) the actual sequence of event (shown on the detector plot).

Data previously saved to files can be read into the program again using the menu option “read data” under data files in the top menu. The method parameters are displayed in the appropriate boxes and the history window displays what happened at the time of the original run. The “comments” window displays any comment you added during the original run. The method can be edited but the history can not be edited and the data can not be resaved under the original name.

Print chromatogram after run

If the program finds this box is checked at the end of the run, the two plots are plotted in portrait mode on a single page (in color if you have a color printer). Printing then continues on subsequent pages with the run history, your comments for this cycle of the run, and the results of any peak picking and peak areas.

Print in Black/White

If the program finds this box is checked at the end of the run, the two plots are plotted in portrait mode on a single page in B/W regardless of whether you have a color printer or not. The various colors in the plots are translated into combinations of dots and dashes. Depending on the options you selected, printing then continues on subsequent pages with the run history, your comments for this cycle of the run, and the results of any peak picking and peak areas.

No data file as yet

This is merely a label to tell you where the latest set of data was saved. If no data have yet been saved during the current session, the label caption is “No data file as yet”. Otherwise, the caption is “Data saved in file *****” where ***** is the name of the latest file.

Select data directory - current directory

Use the collection of controls in this group box to select the directory where you want the data files to be stored. This label shows the directory where the next file to be written will be stored. The directory name is shown in abbreviated form if it is long. Use the directory selection box to change to a different directory. Use the disk selection box to change to a different disk. To create a

new directory, use the directory selection box to go to the place on the disk where you want the directory to be created (in other words to its parent directory), then use the edit box at the bottom of the screen to tell the program the name of the directory, and press the Enter key to activate the directory generation process. Then select the directory in the directory selection box. If the directory name did not appear in the directory selection box, you did something wrong. Try again. When you have done everything correctly, the new user directory will appear in the label.

Select data directory - disk to show directories of

Use this control to change to a different disk.

Select data directory - files to show in files box

Use this control to specify the file filter which governs which types of file names will be shown in the file list box (see below).

Select data directory - Add new directory...

First, use the directory selection box to go to the directory, which will become the parent directory of the new directory. Then type the name of the new directory in this edit box. When the name is complete, press the Enter key. The directory will be created and will appear as a choice in the directory selection box. Not until you select the directory in the directory selection box will it become the active directory!

Select data directory - directories

Use this control to select the active directory - where future data files will be written. Only existing directories can be selected. If you want to create a new directory, you have to generate it using the "Add new directory" control and then select it using this control to make it the active directory.

Select data directory - files

This control merely shows the contents of the currently selected directory. There is no direct user input.

Mtls - Cols & Sols

History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	

column 1

Dead volume of column 1 = 3.2

Solvent Bottle 1

Solvent Bottle 2

Select the description of column 1

Column 1

The columns are specified using selections from lists. The lists are contained in files of the type *.clm. When these files are read in, the contents are used, one line per column, to populate the list box. Note that the first parameter on each line in the file is the column dead volume. The dead volume is separated from the rest of the column description by at least one space.

Solvent Bottle 1-2

The contents are specified using selections from lists. The lists are contained in files of the type *.slv. When these files are read in, the contents are used, one line per solvent bottle contents (which can be a pure solvent or a mixture), to populate the list box.

When the input focus shifts from the solvent table, the program assumes that all entries have been made and automatically re-arranges the entries into chronological order and checks them for 100% solvent being specified in each line. **Only when you leave the table (this is true for all tables) will the program read what you entered into the table - this is to prevent the program from doing the checking before you have finished the editing.**

When using high pressure mixing these times are indistinguishable from the run time.

Time

The time in the solvent time system, as described above under Solvent Program, at which the solvent mixture is to have the composition in the right hand columns of the solvent table.

%A, %B,

The two percentage compositions used to specify the starting solvent mixture. The total percentage must add up to 100% before the user is allowed to leave the solvent editing region. When the solvent table is empty, the starting composition is used throughout the run and the solvent system is isocratic. When the solvent table contains valid entries, the solvent composition changes linearly with time so that it has the specified composition at the times in the table.

If the user leaves the solvent area or jumps to the next line without completing a line in the solvent table and the total composition is less than 100%, the missing percentage is given to solvent B. The program will not allow %A + %B to be greater than 100%. A total of greater than 100% will display an error message, but only after leaving the solvent area. Until then the operator may make changes at will.

Current Solvent Table Values

Solvent Table Time

As described under Solvent Program, if the solvent mixture is not isocratic, i.e., it varies with time, then it is necessary to know the current time in that time system if the solvent table is to be edited. The current solvent time is displayed here.

Isocratic

A message is displayed here to show whether the solvent system has been perceived by the program to be isocratic or not. For example, the starting composition could have been specified as 50%A, 50%B and the solvent table could contain an entry 50.1%A and 49.9%B. Is this supposed to be isocratic or not? The program considers only those compositions which differ by 0.1% in composition to be different from one another. Therefore, in the example given, the solvent system would be considered to be isocratic, even though two solvent compositions had been specified. In particular, this would mean that no time would be spent waiting for the solvent front to go from the mixer to the injector and the run would be over more quickly, less total solvent would be consumed, Solvent Saver could be used, and so on.

%A, %B

The current solvent composition at the mixer is displayed here. Among other things, this provides a useful check on what the mixing valves (low pressure) or pump flow rates (high pressure) should be doing - you can hear them clicking if the solvent composition is being mixed in real time.

Solvent saver threshold

Solvent Saver is the automatic recycling of solvent through the pump. This value is based on signal level around the zero axis that means that the mobile phase is recycled when the signal is between the +/- of the entered value. Two blue dotted lines indicate these boundaries on the screen. The threshold detector signal at which to cut off Solvent Saver is specified here. It is worthwhile expending some considerable effort to develop an isocratic method so that Solvent Saver can be used - the cost savings are often substantial. The only solvent loss from the system then basically becomes that area of the baseline where there are peaks.

Detector to use

If more than one detector is in use, only one of the detectors can be used to control Solvent Saver. That detector must be specified here.

Flow Program

The flow program is merely a series of flow rates and the times at which to implement these flow rates. Because a new flow may be implemented at any time, the time of the flow system is the same as the normal run time and is based on time zero being the moment of injection.

Initial Flow

The initial flow rate must be specified here in mLs/min.

Flow Program Table

Time

The time, based on time zero being the moment of injection, when the flow in the right hand column is to be implemented.

Flow

The flow in mLs/min to be implemented at the time in the left hand column.

Peak Picking

Peak detection is somewhat of an art, but we have tried to make the procedure robust. The various parameters are explained below. The accompanying Browse program allows you to test your choice of peak-picking parameters on a chromatogram you have already collected.

History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	

Use Peak Picking Pick Peaks on Current Chromatogram

Detector to use: 1 Show Peak Picking Results In History

Selecting peak picking parameter values

Peak Detection Parameters	
1.0	1: Min Threshold
<input type="checkbox"/> Use PeakThreshold	
1.0	2: Min Slope
3000.0	3: Peak Max
2	4: Begin
2	5: Valley
2	6: End
2	7: # Points in Slopes
10.0	8: Min Area %

Peak Picking Parameters

Peak detection is somewhat of an art, but we have tried to make the procedure robust. The various parameters are explained below.

Peak threshold

The detector reading must be above this level for a peak to be possible. It is possible to operate with a reasonable value for the threshold and no slope criterion if the chromatogram baseline is well-behaved. The most difficulty is found in liquid-liquid chromatography, where the mobile phase itself can exert a significant signal in the detector and this signal will change if and when the composition of the mobile phase changes. For gas chromatography, peak detection is simpler.

A dotted red line corresponding to the current peak threshold is drawn on the plot.

Specify the peak detection parameters

Peak Picking Parameters

The various parameters are explained below.

Peak threshold

The detector reading must be above this level for a peak to be possible. It is possible to operate with a reasonable value for the threshold and no slope criterion if the chromatogram baseline is well-behaved. The most difficulty is found in liquid-liquid chromatography, where the mobile phase itself can exert a significant signal in the detector and this signal will change if and when the composition of the mobile phase changes.

Note1:

In assessing chromatogram quality in the Browse program, the Peak Threshold is kept constant during evaluation of the chromatogram.

Note2:

The threshold is only active if the “Use Peak Threshold” box is checked.

Minimum slope

A peak can only begin when the detector reading is above the peak threshold level as described above under ***Peak Threshold***. The second condition is that the **slope of the detector reading with time must be greater than some threshold slope value**. That threshold slope value is specified here. If you specify zero, all crossings of the detector reading from below to above the minimum threshold discussed above will result in a peak beginning being found.

However, it could be that what is occurring is only a slow drift in detector reading which is meaningless in terms of peaks and eluents. Also, you can not find a valley in a compound peak.

For those reasons, it is a good idea to use the minimum slope parameter. In that way, you specify the detector trace must have at least a given trend upwards with respect to time before the program will declare it has found a peak.

Generally, decrease the minimum slope value to increase the sensitivity (include more of the peak). Similarly, increasing the value will decrease the area of the peak. Changing the minimum slope value may also be used to include or omit some small peaks.

The slope threshold is used to find valleys in compound peaks. A valley is considered to have occurred if the program is already on a peak and the peak slope has “*recently*” been above the slope threshold but with a negative sign (i.e., coming down, not going up) and is now also above the slope threshold but with a positive sign (i.e., going up, not coming down). “*Recently*” means within the number of data points specified under **Range for Valley**.

A good way to choose reasonable values for the slope parameter is to use the Browse program to run peak-picking on several chromatograms similar to the one you expect.

Note:

In assessing chromatogram quality in the Browse program, the Peak Minimum Slope is kept constant during evaluation of the chromatogram.

Peak maximum

If the detector reading is very high, it may be unsteady, for example in the case of a UV detector as it fluctuates between (say) 99.0 and 99.9% absorption (absorbances of 2 to 3). To avoid the spurious generation of valleys, **any detector reading above the value chosen here as the detector maximum will be made equal to the maximum for peak-picking purposes (the original reading will be retained for the data file)** and, if the detector maximum has been chosen wisely, this will **suppress the false valleys** generated by apparent spikes or raggedness in the tops of peaks.

When the program has a value for the peak maximum parameter, a dotted blue line is drawn on the detector plot.

Range for Begin

When the beginning of a peak is found, the detector trace has just passed the criteria of height and slope threshold. However, since the peak is only just beginning,

the signal is very small and easily affected by impurities, small glitches and noise. To stop spurious beginnings and ends of peaks being reported immediately after a peak beginning has been discerned, no further checks of peak activity are made until the **Range for Begin** number of data points have been measured. By that time, the detector signal will probably be significantly above baseline and less affected by noise, etc. and peak checking for valley and peak end can be resumed. A good number **Range for Begin** is 4 to 10 data points.

Range for Valley

The slope threshold is used to find valleys in compound peaks. A valley is considered to occur if the program is already on a peak and the peak slope has “recently” been above the slope threshold but with a negative sign (i.e., coming down, not going up) and is now again above the slope threshold but with a positive sign (i.e., going up, not coming down). “Recently” means within the number of data points specified under **Range for Valley**. A good number to start with is 10 or so data points. For some chromatograms, larger numbers are required to bridge a wide valley. Generally, this value needs to be increased when the valley is very U-shaped (approx. 20-25) versus a sharp V-shaped valley (approx. 5-8). All these peak parameters can be tested and refined by reading a previously collected data file into the Browse program.

Range for End

When the end of a peak is found, the detector trace has just “failed” all the criteria of height and slope threshold, etc., after being on a peak. However, since the signal is very near to the peak thresholds and is therefore easily affected by noise, a very small excursion could make the program think it has found a new peak. To stop spurious beginnings of peaks being reported immediately after a peak end has been discerned, no further checks of peak activity are made until the **Range for End** number of data points have been measured. By that time, the detector signal will probably be significantly below the thresholds and peak checking for peak beginnings can be resumed. A good number for **Range for End** is 4 to 10 data points.

Points in Slopes

If a chromatogram is noisy and peak detection is triggering too frequently you can increase this value. This

will change the number of points, which are averaged together.

Increasing the number of points used in estimating the slope of a curve increases the certainty that a particular slope has been attained in peak picking but doesn't detect the increase in slope as early/low down on the peak as using a smaller number of points would do. Some chromatograms require that as many as 10 points be used in estimating slope. Chromatograms with less scatter in the data can use 4 or 6.

Min Area %

This will determine which peaks will be displayed and used in the Peak Results table.

Use peak picking

When this option is checked, the program continuously monitors the detector signal for the appearance of a peak feature (begin, valley or end) as judged from the peak-picking parameters supplied. Peak-picking can be carried out even when the fraction collectors are operated on time rather than on peak-picking. Markings showing the peak features found are written to the detector plot.

Use peak picking sounds

When this option is checked, the program announces the appearance of any peaks via verbal messages through the speaker system attached to the sound card in the computer.

Detector to use

If more than one detector is being used, the detector signal to be used in looking for peaks must be specified here.

Pick Peaks in Current Chromatogram

Pressing this button causes a re-integration of the chromatogram using the current peak picking parameters.

Alarms

History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report
General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	

Alarm Limits

low	high	
<input style="width: 50px;" type="text" value="0"/>	<input style="width: 50px;" type="text" value="2500"/>	Pump pressure

Alarm Status

Pump pressure high

Pump pressure low

Alarm Control

Audible alarms

Alarm limits

Various hardware and software alarms are supplied with the apparatus and program. The software alarms are generated by the software from the appropriate transducer readings. The alarms are triggered when the transducer signal is below (for the low limits) or above (for the high limits) the limits given in the boxes below.

Pump pressure - low

The pump pressure must be above this limit. However, because the pump pressure is zero when pumping first begins, the test is not carried out during the equilibration phase, the injection phase or during the first 30 seconds of the separation phase.

Pump pressure - high

The pump pressure must be below this limit, no exceptions.

Alarms status

Some of the alarms below are generated by hardware sensors, the others are generated by the software when the appropriate transducer signal exceeds the alarm limits specified above. The cause of all alarms must be corrected before execution of the program can continue. When you have corrected the cause of the alarm, click on Alarm reset to allow the program to continue. Unless you have really corrected the cause, the program will of

course immediately stop again on the same alarm condition.

When an alarm is set, the background of the corresponding text below becomes red; when all is well, the background is green.

Pump pressure low

and

Pump pressure high

are set by the software comparing the latest pump pressure reading with the user-chosen limits on the Alarms Page.

Allow check of alarms

Alarm checking is an important part of normal operation. Sometimes, however, one has to over-ride the alarm checking in order to diagnose what is wrong. The alarms are CHECKED ONLY when this button is DEPRESSED. Having the button depressed is the normal state of operation. Use the program and apparatus with this button up only in very rare cases when you MUST for some reason over-ride the alarm system in order to diagnose what is wrong. DO NOT OPERATE THE APPARATUS ROUTINELY WITH THIS BUTTON DEPRESSED. TO DO SO COULD BE DANGEROUS.

Alarm reset

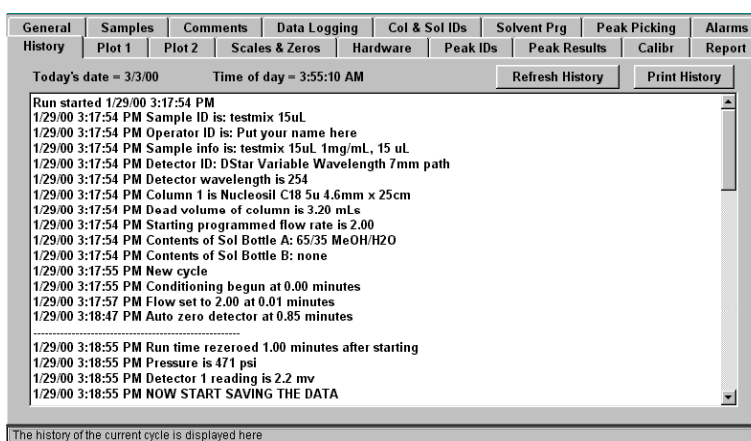
Clicking on this button resets the alarm condition to be set to off and continues all program operations, including further checks on alarms, unless the above button, Allow Check of Alarms, is NOT DEPRESSED.

Audible alarms

When this option is checked, the program announces all alarm conditions through the speakers attached to the sound card.

History log

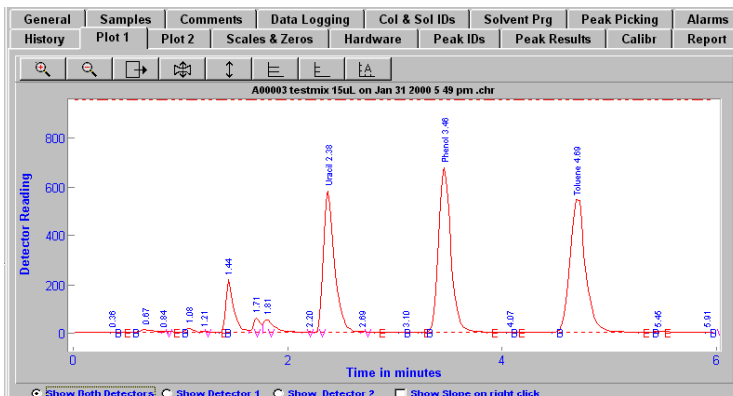
All events (such as injection time, contact closures, column configurations, new flows) and status reports generated during a cycle are posted to the operator in the history log. The log is regenerated for each cycle and begins with a description of the columns and solvents used. This is a read-only window, with the date, time and a description of the event or status on each line. The information is written to the data file if the data are saved in a file and can be re-read by reading in old data files. The history log is printed when the chromatogram is printed.



Plot (1) of detector(s), events fired, peaks found

This plot goes through two stages in each cycle. The first stage is from the beginning of the cycle through the equilibration phase, and the conditioning phase. The plot window is then wiped clean and the second stage begins with the injection phase. The second stage is a plot of the detector response during the separation phase, with any events and peak picking marks superimposed on the plot.

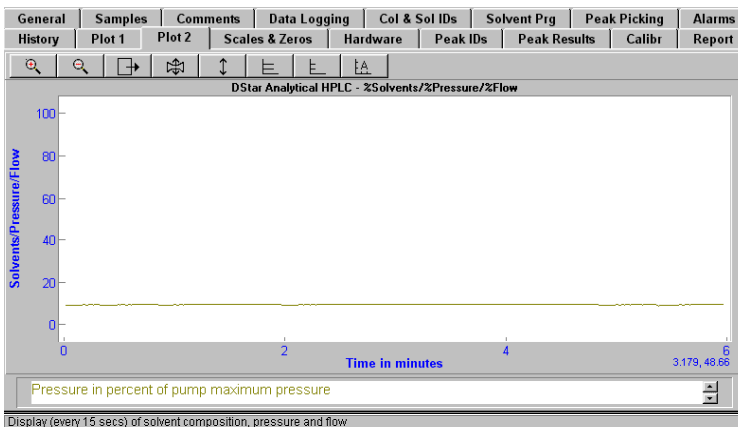
If the data are printed, this is the first of two plots to be printed and is to be found at the top of the page. The bottom half of the first page contains a plot of the solvent program and pressure. A key to the colors and symbols used in the plots is given at the top of page 2 of the output.



Plot (2) solvent program and pressure

This plot shows visually the solvent program and the course of the pressure of the mobile phase as measured at the pump. Thus, you can see at a glance if the apparatus is behaving itself.

If the data are printed, this is the second of two plots to be printed and is to be found at the bottom of the first page. A key to the colors and symbols used in the plots is given at the top of page 2 of the output.



Right clicking on the plot gives the solvent composition at the run time clicked.

The various curves in the plot are identified in the box below the plot in the colors used for plotting the curves.

Scales & Zeros

General	Samples	Comments	Data Logging	Col & Sol IDs	Solvent Prg	Peak Picking	Alarms	
History	Plot 1	Plot 2	Scales & Zeros	Hardware	Peak IDs	Peak Results	Calibr	Report

Scales and zeros	
<input type="text" value="1.000"/>	detector 1 scale - applied to readings
<input type="text" value="0.000"/>	detector 1 zero - applied to readings
<input type="text" value="1.000"/>	detector 2 scale - applied to readings
<input type="text" value="0.000"/>	detector 2 zero - applied to readings
<input type="text" value="1.000"/>	pressure scale - applied to readings
<input type="text" value="0.000"/>	pressure zero - applied to readings
<input type="text" value="1.000"/>	DStar detector wavelength scale
<input type="text" value="0.000"/>	DStar detector wavelength zero

Read parameters from screen using button at bottom of Hardware Screen

directory for sound files for notifying operator

Scales and zeros

The following quantities are all very similar in function. First the zero is subtracted from the readings. Then the scale is applied. This may serve several purposes, but a typical use is when it is desirable to transform mV to some other unit of measurement.

detector 1 scale - applied to readings

detector 1 zero - applied to readings

detector 2 scale - applied to readings

detector 2 zero - applied to readings

pressure scale - applied to readings

pressure zero - applied to readings

Dstar Detector Wavelength scale - applied to readings

Dstar Detector Wavelength zero - applied to readings

ANY TIME A CHANGE IS MADE IN THIS PAGE OR THE HARDWARE PAGE YOU NEED TO PRESS THE "CLICK HERE TO READ..." BUTTON ON THE BOTTOM OF THE HARDWARE PAGE. IF THESE CHANGES WILL BE PERMANENT YOU MUST ALSO SELECT "INI FILE" IN THE

Calibr

Calibr DB table is C:\STARCHROM\Startup\CalibrDB.dbr
 Q.C. Test Mix

Calibration DB Files

Send Record to Peak IDs

CoefFs: -6.640675E-16 2.920688E-3 0.000000E+0
 0.000000E+0 0.000000E+0 0.000000E+0

Offsets: 4273.91 12.50 Range: 1542.74 to 6568.15

Sort on... Class, Cmpd ID, Detector, Detector number, Date Show All Records Filter records according to line last clicked in Peak ID Info table
 Date, Class, Cmpd ID 1/30/00 6:43:55 PM Operator's name goes here

CLASS	COMPOUND	DETECT_ID	DET_NUM
Put class of compound here	Uracil	Var UV 7mm path	254.0 nm
Put class of compound here	Phenol	Var UV 7mm path	254.0 nm
Put class of compound here	Toluene	Var UV 7mm path	254.0 nm

The calibration file

This page shows which database is open and which calibration records are available. A “Startup...” database is automatically opened on startup. You can open a different database using the “Calibration DB” button.

Calibration DB Files

Pressing this button allows you to select a database to use.

Send Record to Peak IDs

After selecting a record from the table pressing this button will send the record info to the Peak IDs page.

Report

This page lets you select the type of report that will be printed.

Description of auxiliary files

Solvent File

The solvent file has extension slv (i.e., is *.slv) and is a list of solvent descriptions, one per line, in an ordinary text file.

For those cases where a solvent bottle contains no solvent, you might want to include at the end of the list a line such as :

none.

Columns file

The columns file has extension clm (i.e., is *.clm) and is a text list of column descriptions, one per line, WITH the column dead volume in mLs as a number at the beginning of each line and separated from the rest of the line by at least one space.

For example, 2.5 Column type 252 from Consolidated Widgits.

You might want to include the following line at the end of the list:

0 none

DStarLCMicro.ini

The DStarLCMicro.ini file is used to provide needed parameters to the program. The parameters are provided in a separate file so that they can be modified without needing a new version of the program. A typical DStarLCMicro.ini file might contain:

10 maximum flow of solvent pump in mLs/min

7500 maximum pressure reading for pressure transducer

1 is the comm port for hardware control

1.000 detector 1 scale

0.000 detector 1 zero

1.000 detector 2 scale

0.000 detector 2 zero

7.46 pressure scale

0.000 pressure zero

0 limit for pump pressure too low

2500 limit for pump pressure too high

0.0 dead vol in mLs from mixer to injector

y use dstar detector

1.000 detector wavelength scale

0.000 detector wavelength zero

\starchrom\wav files\ directory for wav files for notifying operator using sound.

Each line is a parameter, followed by descriptive text. Most of these values can be edited in the Scales & Zeros and Hardware Pages. If you are tempted to edit the file

using a text editor rather than using the program page, note that the program uses the text after the parameter to discern which parameter is at the beginning of the line, so do not edit/change the text part.

Peak-picking parameter file

A peak-picking parameter file has the extension val (i.e., is *.val) and contains one text line, for example

```
.013    .001    4      10     4      4
```

which is the six peak-picking parameters slope, threshold, begin, valley, end, number of point in the derivative. You would only edit such a file using the program.

Psicrun.nbr

Each data file begins with a unique run number which is sequentially increased with each cycle. The current run number is kept in this text file.

NOTICE

Supplement to the

Star-Chrom[™]

Star-Chrom LITE[™]

&

Star-Chrom LITE PLUS[™]

SOFTWARE MANUALS

*New Features for the **Star-Chrom**[™] family of software (V4.0.5.2)*

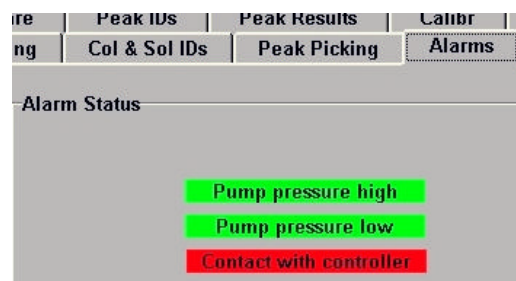
1. COMMUNICATIONS ERROR ALERT

A new feature of the *Star-Chrom*[™] and *Star-Chrom LITE/PLUS*[™] HPLC Management System is an alert message for a communications failure.

A communications failure may occur when the *Star-Chrom*[™] software loses contact with the *Star-Chrom*[™] Interface Box. This may be caused by a loose connection in the RS-232 cable attached to the PC or to the Interface Box (“*controller*”). It may also occur when the Interface Box loses power. Normally this is detected during start up procedures. You are alerted by the message in the lower right of the General page showing “*Running in demo mode.*” You may also be alerted by a voice message. The event is recorded on the History Log.

These warnings may not be noticed if the unit is left unattended during a *Run*. In order to ensure that a communications fault is detected, the software now activates an *alarm* condition. When the software loses contact with the Box:

- a. The Run is paused
- b. A voice message repeats until the alarm is reset
- c. The “Contact with controller” bar on the Alarms page is displayed, turns red, and flashes



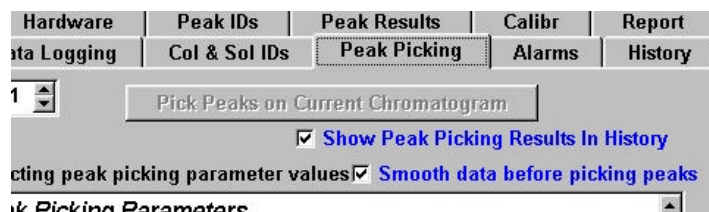
2. SMOOTHING

A feature has been added to the *Peak Picking* function that smoothes data to provide more robust peak integration. This helps to improve the reliability of the peak integration by reducing the amount of noise with minimal change in peak shape. The process is accomplished with a smoothing algorithm.

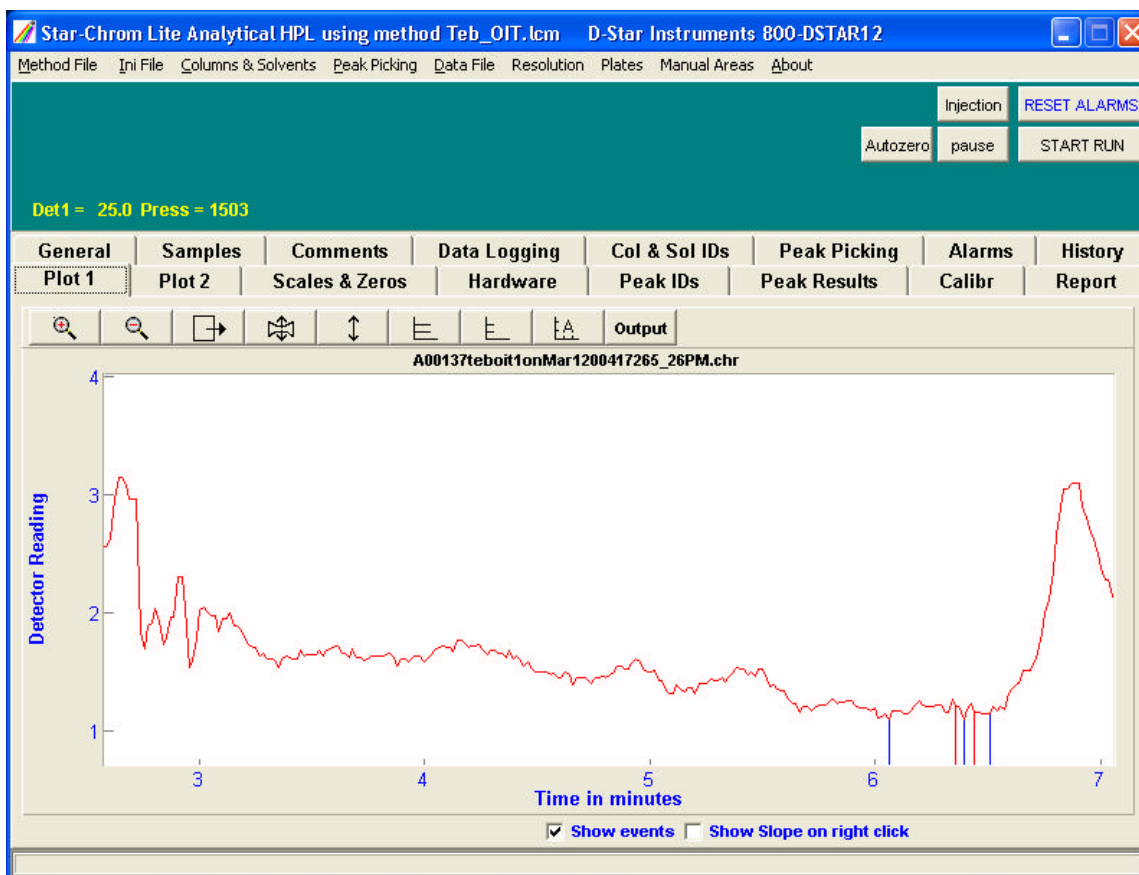
The smoothing does not affect the raw data stored in the original chromatographic run file (.chr). The smoothing function is not stored in the method file. The default value for the function is ON (checked).

There are two ways to select Smoothing.

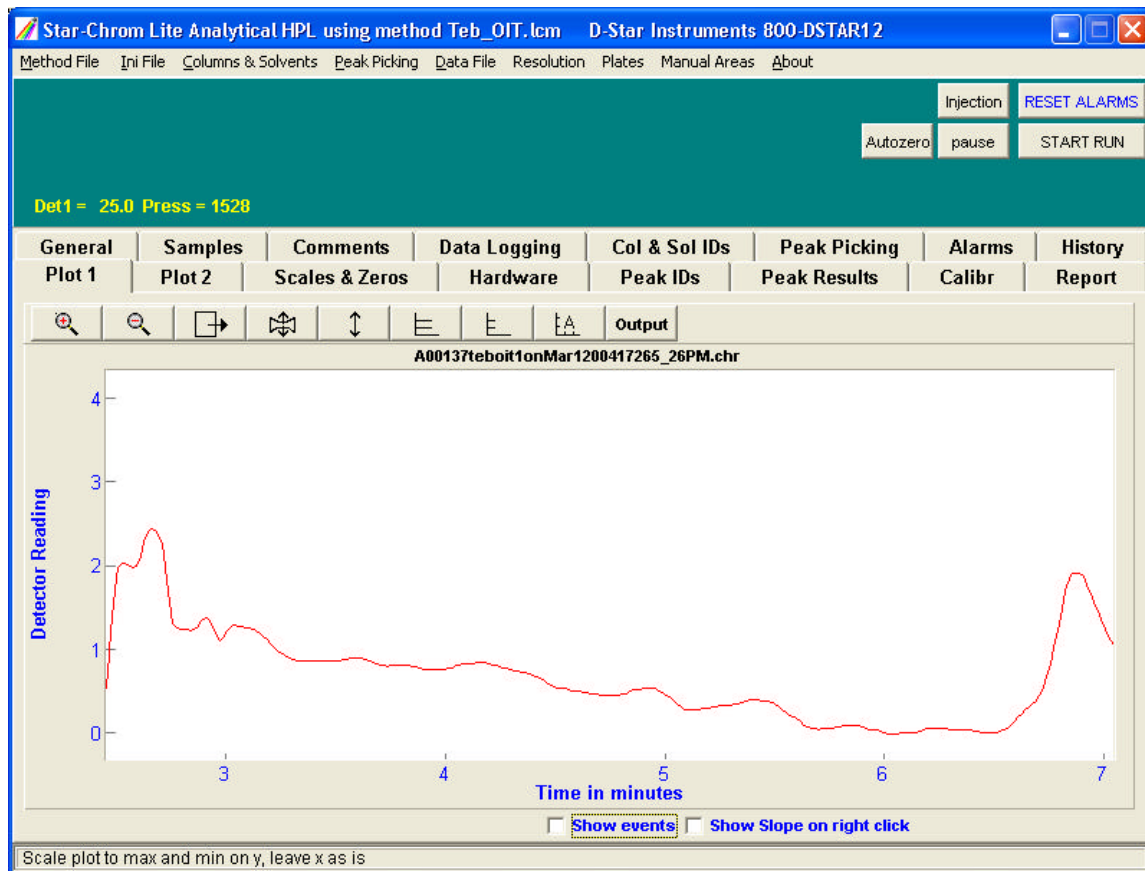
- a. Select the *Smooth Detector Curves* in the Data File drop down menu;
or
- b. Check (select) the Smooth data before picking peaks box on the Peak Picking page.
Then click on the Pick Peaks on Current Chromatogram to view the peaks that have been selected after smoothing.



Examples of the a plot without and with smoothing are shown below



Without Smoothing



With Smoothing

NOTICE

Supplement to the

Star-Chrom™

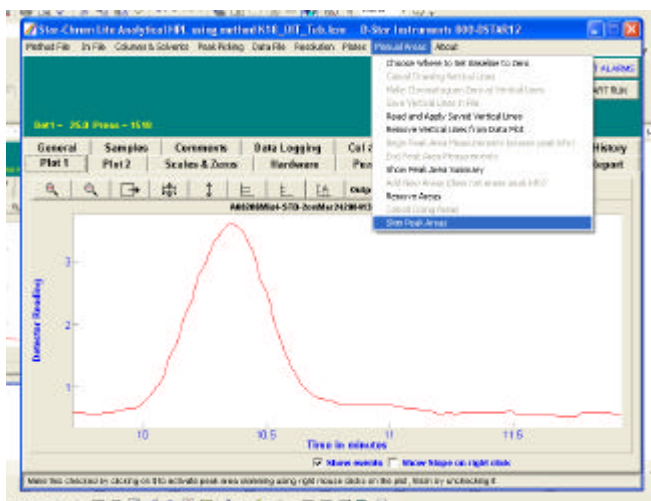
Star-Chrom LITE™

&

Star-Chrom LITE PLUS™

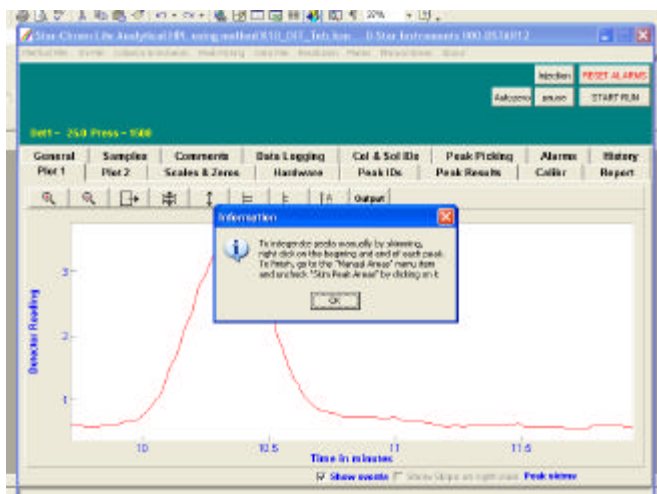
SOFTWARE MANUALS

New Features for the ***Star-Chrom™*** family of software (V4.0.5.3)

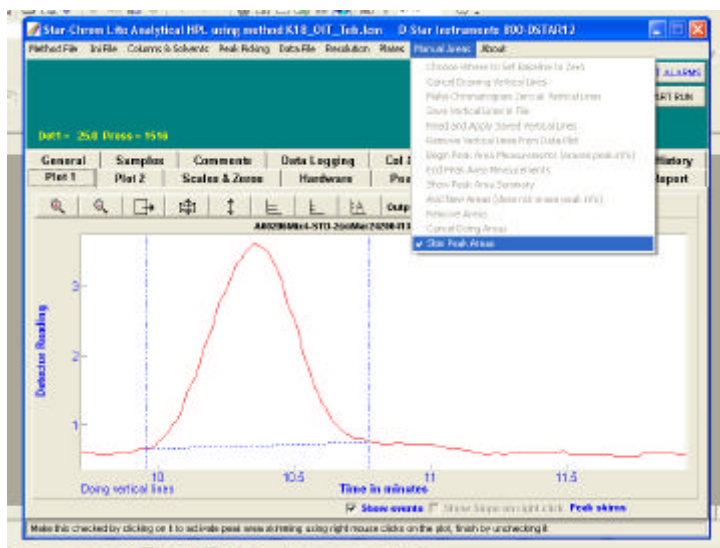


Some times it is hard to automatically integrate peak areas especially of peaks that are near the limits of detection. You can use manual peak picking to get these areas.

Select “Manual Areas” from the menu bar. Then click on “Skim Peak Areas”.



You will then get an information box that tells you how to proceed to select the begin and end points for the manual integration of the peak areas.



You can zoom the plot at any time to get a better view of the base of the peak.

When you are done select “Manual Areas” from the menu bar.

Then click on “Skin Peak Areas”.

This will end the manual peak picking session and the peak areas will be shown and will appear in the “Peak Results” table.

Guide to Using *Star-Chrom's*™ Calibration Module

Calibration Module



D-Star Instruments
8424 Quarry Rd.
Manassas, VA 20110-5326
Phone: 703-335-0770
Fax: 703-335-9952
Toll-Free (USA): 800-dstar12
E-Mail: dstarinstr@aol.com
Web-site: <http://www.d-star.com>

May 21, 2000

CHAPTER 1 - INTRODUCTION AND INSTALLATION	3
INTRODUCTION	3
CHAPTER 2 - MENUS	4
MENU OPTIONS	4
<i>Chromatogram</i>	4
Clear Plot.....	4
Choose Detector to Read (now #1).....	4
Read in chromatogram(s).....	5
Remove curves from plot.....	5
Read Set of Curves	5
Save Set of Curves.....	5
<i>Curve Zeros</i>	6
Clear Zero Places.....	6
Read Zero Places from File	6
Save Zero Places to File	6
Show Zero Places	6
Zero the Curves	6
<i>Peak Areas</i>	7
Select Peak Limits	7
Get Peak Area Measurements.....	7
Show Peak Area Summary	7
Show Peak Info Table.....	7
Cancel Doing Areas.....	7
Remove Lines From Plot.....	7
<i>Data Tables</i>	8
Clear the Data Table.....	8
Read in New Data From a File	8
Save the Data Table to a File.....	8
Save the Data Table to a New File as.....	8
<i>Calibration DB</i>	9
Put Calibration in DB	9
Get Calibration from DB	9
Show Calibration DB Window.....	9
Open an existing calibration database	9
Close an existing calibration database.....	9
CHAPTER 3 – TABBED SCREENS	10
TABBED SCREENS.....	10
<i>Chromatograms</i>	10
Button Bar	10
Cursor Coordinates.....	11
<i>Data Table</i>	11
Title	11
Class of Compound	11
Compound ID.....	12
Detector ID	12
Detector “Number”	12
Operator.....	12
Begin peak at	12
End peak at	12
Baseline / Tangent	12
Quantity / Area table.....	12
Read Info From DB Record.....	12
Save to Database Record	13
<i>Get Polynomial</i>	14
<i>Fit Details</i>	15
<i>Calibration Plot</i>	16

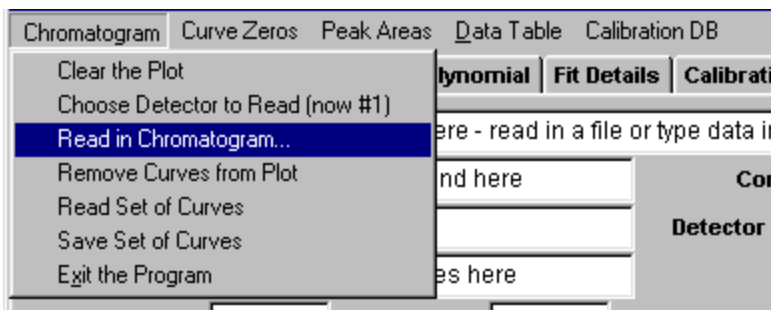
CHAPTER 4 – DISCUSSION ON CURVE FITTING..... 17
DISCUSSION ON CURVE FITTING 17
EXAMPLE OF MAKING A CALIBRATION CURVE..... 23

CHAPTER 6 – EXAMPLE PART TWO 31
EXAMPLE OF APPLYING A CALIBRATION CURVE 31

Chapter 2 - Menus

Menu Options

Chromatogram

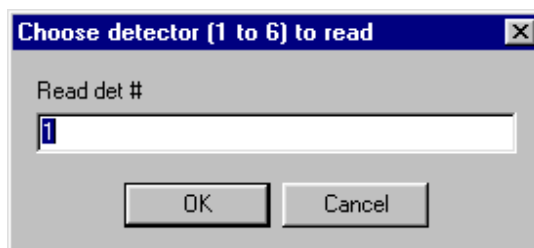


Clear Plot

This menu option removes all curves from the plot.

Choose Detector to Read (now #1)

Each chromatographic file contains information for up to 6 detectors. If the file contains information on multiple detectors, this menu option tells the program which detector to use in reading in the chromatogram. Selecting the Choose Detector to Read menu option brings up the following dialog box, which allows the operator to specify another detector. If OK is clicked, the new detector number will be shown in the Choose Detector menu option.



Read in chromatogram(s)

Read in one or more chromatograms which must be the *.chr type, written by all the Star-Chrom control programs. This file is the default option for types of files to be read into the Calibration program. It contains the detector readings from a chromatographic run.

Remove curves from plot

Allows the user to remove one or more curves from the plot.

Read Set of Curves

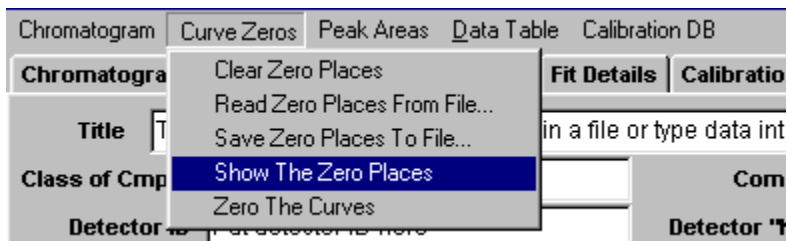
This option opens a file dialog box. You need to find the file you want to open then click on the file name, which highlights it. Then press the "open" button. There will only be a file available if you have previously saved a set (see below).

Save Set of Curves

After multiple curves have been read into the program, the operator can save the file names making up that particular collection as curves in a "set" file using this menu option. The overlaid plot can then be regenerated easily by reading in the set file. If you move the files, or transfer the set files to another computer, the set file will not work unless the directory names mentioned in the set file are the same as those on the original computer.

This is useful for grouping a set of related files. For example, a blank run, sample run and duplicate could be overlaid and saved as a set.

Curve Zeros



Clear Zero Places

This menu option removes all zero points from the show zeros table.

Read Zero Places from File

This menu option allows the user to open a file in which a list of zero places has been stored previously. This is a useful feature when processing the same compounds on a routine basis such as in a q.c. lab.

Save Zero Places to File

This menu option allows the user to save all zero points from the show zeros table to a file. This is a useful feature when processing the same compounds on a routine basis such as in a q.c. lab.

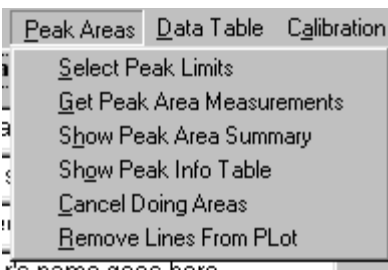
Show Zero Places

This menu option makes the zero table visible. If the table is already visible selecting this option again removes the table from the screen. While this table is visible you can select points on the baseline using the right mouse button the retention time at that point is now filled in the table. The program will zero the baseline at these points. This is necessary to get accurate and reproducible results.

Zero the Curves

This menu option zeros the baseline using all of the zero points from the show zeros table. You must zero the baseline before trying to construct the calibration curve.

Peak Areas



Select Peak Limits

Selecting this menu option toggles the feature on and off. If the feature is active then a check mark will be visible. When active, this feature allows the user to click on the plot with the right mouse button and the selected retention times will be filled into the "Data Table page". You can only select one retention time range per calibration curve.

Get Peak Area Measurements

Once you have selected the peak limits or retention time range this option will calculate the area for the specified peak for all curves on the plot. This information is displayed in a table. The areas can then be entered or dragged to the "Area" column on the data table page.

Show Peak Area Summary

This option makes the Peak Area Summary screen visible.

Show Peak Info Table

This option makes the Peak Info Table visible.

Cancel Doing Areas

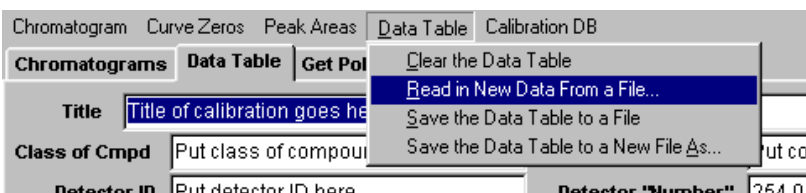
Cancels the get area operation.

Remove Lines From Plot

Removes the vertical lines on the plot.

Data Tables

Clear the Data Table



Selecting this menu option toggles the feature on and off. If the feature is active then a check mark will be visible. When active, this feature allows the user to click on the plot with the right mouse button and the selected retention times will be filled into the “Data Table page”. You can only select one retention time range per calibration curve.

Read in New Data From a File

Selecting this menu option toggles the feature on and off. If the feature is active then a check mark will be visible. When active, this feature allows the user to click on the plot with the right mouse button and the selected retention times will be filled into the “Data Table page”. You can only select one retention time range per calibration curve.

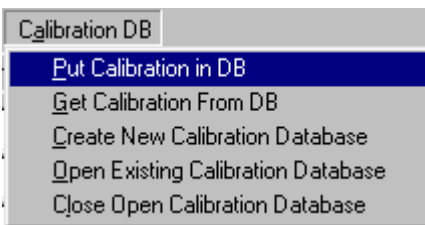
Save the Data Table to a File

Selecting this menu option toggles the feature on and off. If the feature is active then a check mark will be visible. When active, this feature allows the user to click on the plot with the right mouse button and the selected retention times will be filled into the “Data Table page”. You can only select one retention time range per calibration curve.

Save the Data Table to a New File as..

Selecting this menu option toggles the feature on and off. If the feature is active then a check mark will be visible. When active, this feature allows the user to click on the plot with the right mouse button and the selected retention times will be filled into the “Data Table page”. You can only select one retention time range per calibration curve.

Calibration DB



Put Calibration in DB

After you have completed all the information and calculated the calibration curve you can add the record to the Calibration Database (DB). Once you add the record you can not change the information in the record.

Get Calibration from DB

Selecting this option will make visible the Calibration DB Window. When you press the "Database" button on this window you can open a database and select a record to read in. When you read in this record all the information is entered into the Data Table page. This saves time re-entering information if you are processing a new calibration curve for a compound that you routinely are running.

Show Calibration DB Window

Selecting this menu option makes the Calibration DB Window visible.

Open an existing calibration database

Selecting this menu option opens a Calibration DB . This is needed in order to save a newly created record.

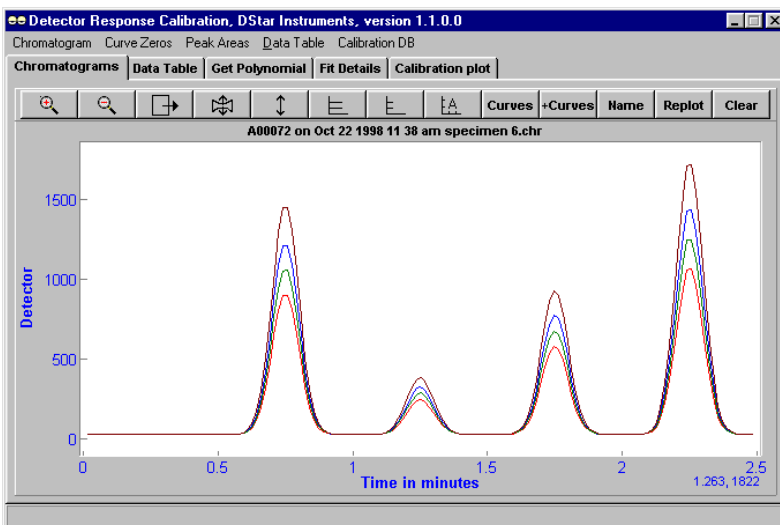
Close an existing calibration database

Selecting this menu option closes the Calibration DB.

Chapter 3 – Tabbed Screens

Tabbed Screens

Chromatograms



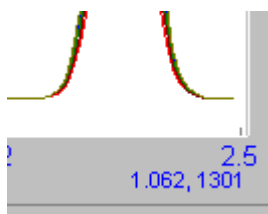
This screen displays the chromatograms selected for use in creating a Calibration DB record.

Button Bar



These buttons act on the plot display.

Cursor Coordinates



The lower right corner shows the current cursor coordinates in Time, Detector Signal units.

Data Table

This screen displays the information that will be used to define a record for the Calibration DB.

Detector Response Calibration, DStar Instruments, version 1.2.0.4

Chromatogram Curve Zeros Peak Areas Data Table Calibration DB

Chromatograms Data Table Get Polynomial Fit Details Calibration plot DB Records Plot of Calibrations in DB

Title Title of calibration goes here - read in a file or type data into table

Class of Cmpd Put class of compound here Compound ID Put compound ID here

Detector ID Put detector ID here Detector Wavelength 254.0 nm

Operator Operator's name goes here Show Clipboard - put peak data on clipboard using Peak Areas menu

Begin peak at 0.00 End peak at 0.00 Baseline/Tangent B

Put the sample quantity/peak area data here

Quantity	Area	Use? (y/n)

Read Info From DB Record

Save to Database Record

Title

This is a descriptive name used to identify the calibration curve being generated.

Class of Compound

This information is used as a field or keyword used by the database to help identify the calibration curve being generated.

Compound ID

This information is used as a field or keyword used by the database to help identify the peak.

Detector ID

This field should be used to identify the actual detector used. A descriptive name and serial number or station number is appropriate.

Detector "Number"

Used as an identifier of how the detector was operated. For example the wavelength setting on a UV-VIS detector.

Operator

This is used to identify the person who developed the calibration curve.

Begin peak at

Defines the beginning time for a retention time window where the peak will be found.

End peak at

Defines the ending time for a retention time window where the peak will be found.

Baseline / Tangent

Defines how the area will be calculated.

Quantity / Area table

Allows the user to define the relationship between the area of a peak and its relation to the actual injected quantity.

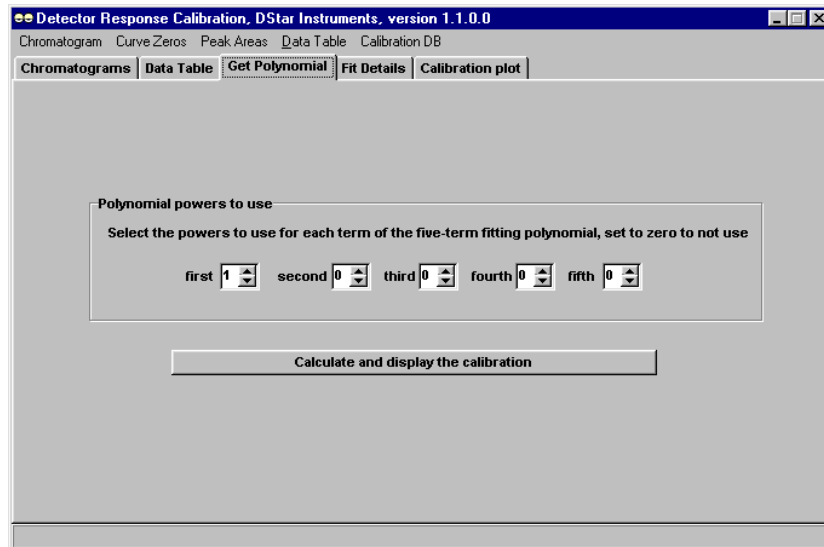
Read Info From DB Record

When pressed, will fill in this page with the information from the record selected from the open calibration database.

Save to Database Record

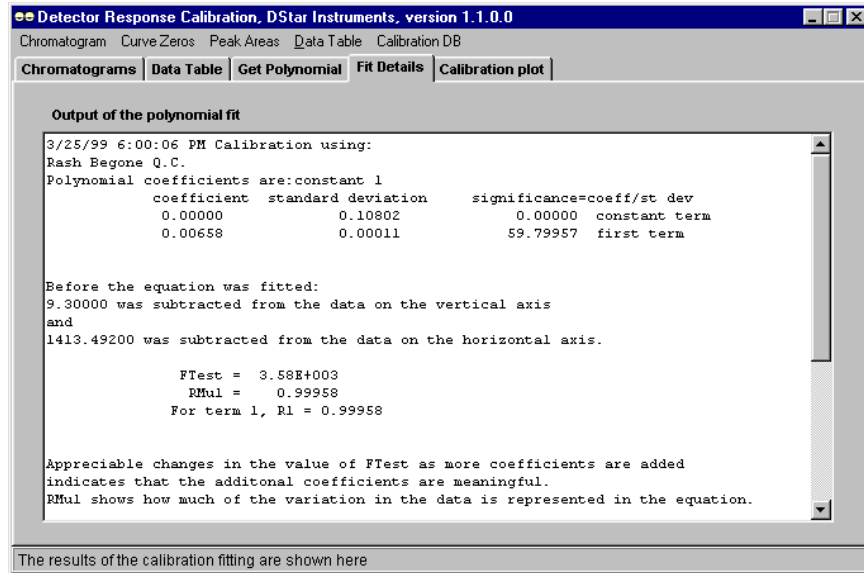
If a previous calibration database has been loaded or opened this button, when pressed, will save this current record to the database selected.

Get Polynomial



This screen is used to define the equation that will be fitted to the data. Once the equation is defined, pressing the “Calculate and display the calibration” button will generate the curve fit information and display the plot of the calibration.

Fit Details



Detector Response Calibration, DStar Instruments, version 1.1.0.0

Chromatogram CurveZeros Peak Areas Data Table Calibration DB

Chromatograms Data Table Get Polynomial **Fit Details** Calibration plot

Output of the polynomial fit

3/25/99 6:00:06 PM Calibration using:
Rash Begone Q.C.

Polynomial coefficients are: constant 1

coefficient	standard deviation	significance=coeff/st dev	
0.00000	0.10802	0.00000	constant term
0.00658	0.00011	59.79957	first term

Before the equation was fitted:
9.30000 was subtracted from the data on the vertical axis
and
1413.49200 was subtracted from the data on the horizontal axis.

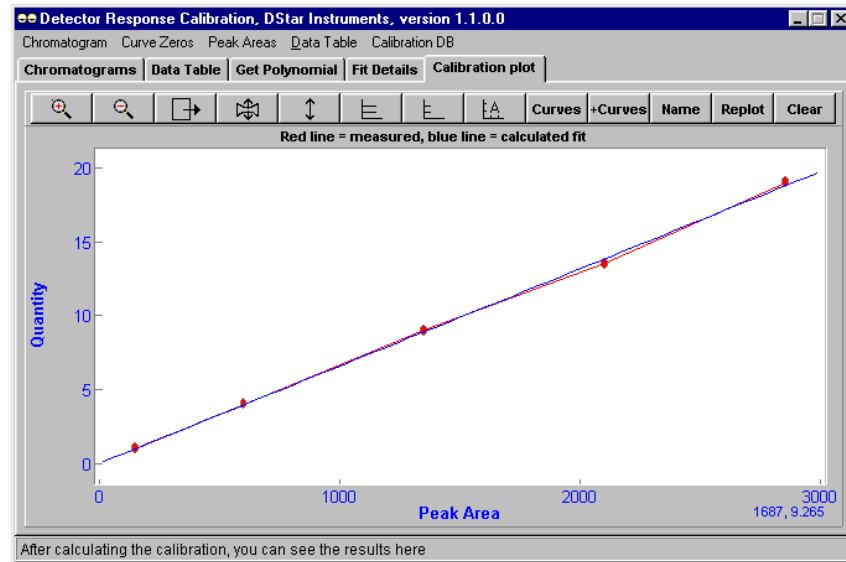
FTest = 3.58E+003
RMul = 0.99958
For term 1, R1 = 0.99958

Appreciable changes in the value of FTest as more coefficients are added
indicates that the additional coefficients are meaningful.
RMul shows how much of the variation in the data is represented in the equation.

The results of the calibration fitting are shown here

This screen is used to display the statistical information generated when the equation was fitted to the data.

Calibration Plot



This screen is used to display the data used and overlaid on this data is the curve that was fitted using the equation defined.

Chapter 4 – Discussion on Curve Fitting

Discussion on Curve Fitting

Before the fit of the injected quantity to the area of the peak is calculated, the origin of the data is moved to the “centroid” of the data. This means that the average of the x values is subtracted from all x values and the average of the y values is subtracted from all y values. The reason for this manipulation is that the error in the fit, and hence in the values estimated from the fit, increases as the point being estimated is moved away from the origin of the data. Putting the origin of the data at the centroid of the data ensures that the minimum error in the fit is in the most useful place, i.e., in the center of the range of the data, rather than at the original origin (where $x=0$, $y=0$).

The equation fitted is of the form

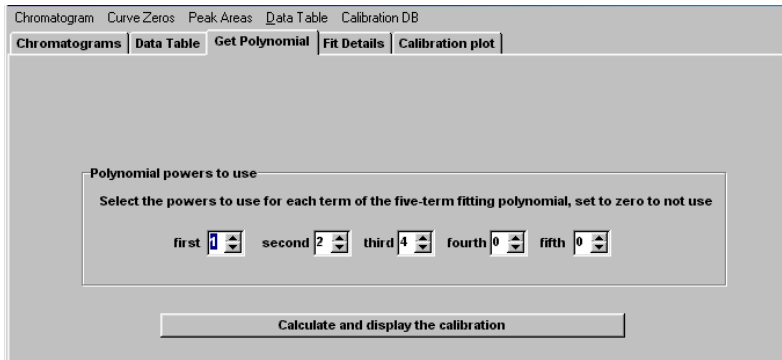
$$\text{Concentration} = c_0 + c_1 \cdot \text{Area} + c_2 \cdot \text{Area}^2 + c_3 \cdot \text{Area}^3 + c_4 \cdot \text{Area}^4 \dots$$

where various values of concentration and the areas of the resultant peaks are input to the program. Actually, the area is typically measured from a series of chromatograms using one of the features of the program. The program estimates the values of c_0 , c_1 , c_2 , etc. Because the areas are raised to the n^{th} power in the equation, the fit is known as a polynomial fit and the coefficients c_n are known as the polynomial coefficients.

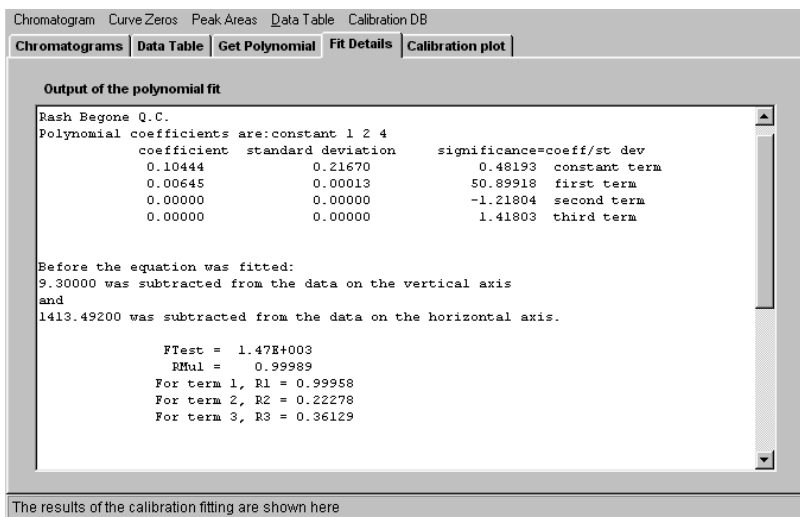
In the above equation, all powers of the area between 0 and 4 are shown. (A standard rule of arithmetic defines Area^0 as 1, so c_0 appears to stand alone.) However, not all powers need be used. For example, one could just as easily calculate the fit of the equation

$$\text{Concentration} = c_0 + c_1 \cdot \text{Area} + c_2 \cdot \text{Area}^2 + c_4 \cdot \text{Area}^4 \dots$$

Simply by not having a power of 3, as in the screen below, we will calculate the fit for powers of 1,2 and 4.



Are all the coefficients which result from this fit real (non-



zero)? The statistical way to tell is to look at F Test values, as on the Fit Details page.

The second line of the text shown in the screen shot reports that coefficients were determined for a polynomial containing powers of 1, 2 and 4, which were the numbers chosen using the spin buttons on the Get Polynomial page. Below that line, the estimated values of the coefficients are shown, together with the estimates of their standard deviations and the value of the coefficient divided by its standard deviation. If the coefficient is really non-zero, presumably it will be more than (say) 2 standard deviations from zero and the absolute value of the value/standard deviation quotient will therefore be greater than 2.

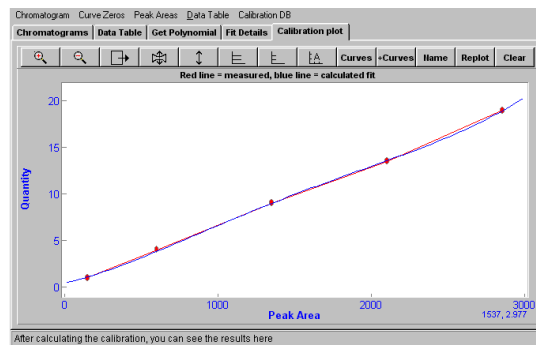
According to the text in the screen shot, 9.3 was subtracted from the concentration values (the vertical axis

in the plot) and 1413.492 was subtracted from the area values. These are the averages of the concentrations and areas, respectively.

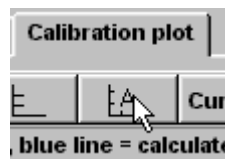
The FTest for this fit is 1.47×10^3 . The FTest is used to compare the effect of changing the numbers of coefficients estimated. In a moment, we will compare this value with a value calculated for a fit with fewer coefficients.

The “multiple” R value for this fit, the amount of the variation in the data which is covered by the equation, is very high, 0.99989. Almost all of this variation is accounted for by the first coefficient ($R_1 = 0.99958$). The remaining coefficients (or terms) contribute relatively little to the fit and therefore the fit should be redone with some or all of these coefficients removed.

The plot resulting from this fit is shown below.

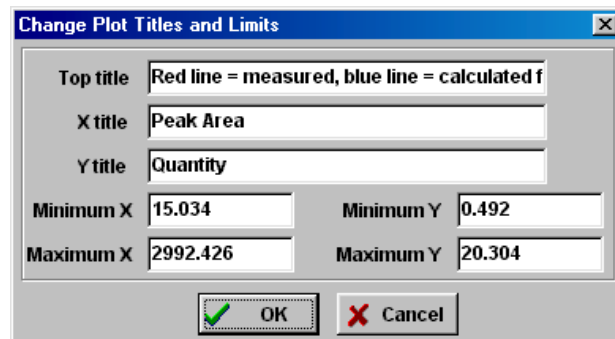


The original data are plotted as the red line and the fit is plotted as the blue line. The blue line seems suspiciously wavy. If the plot is examined in regions where the fit is not constrained by data (i.e., it is being extrapolated) by changing the plot limits (click on the plot button as shown



and changing the plot limits appreciably, as from

to



Change Plot Titles and Limits

Top title: Red line = measured, blue line = calculated f

X title: Peak Area

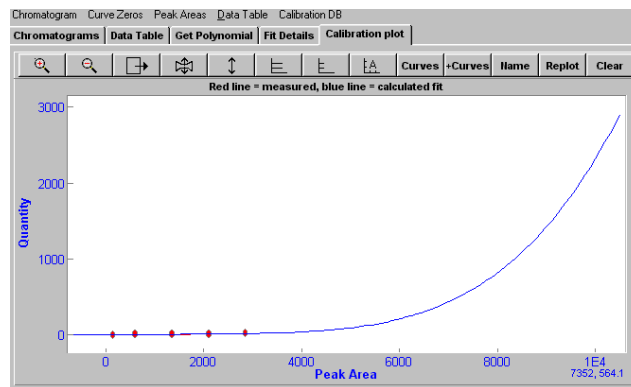
Y title: Quantity

Minimum X: 15.034 Minimum Y: 0.492

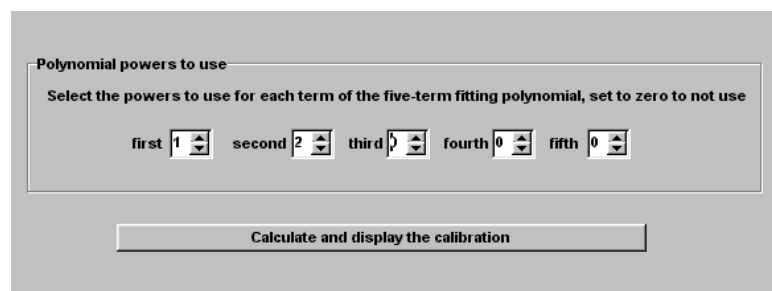
Maximum X: 2992.426 Maximum Y: 20.304

OK Cancel

we see



which suggests that a better fit can be found and shows the dangers of extrapolating polynomials. In brief, **DON'T EXTRAPOLATE!** Make sure your data cover the range over which you want to estimate concentrations.



Polynomial powers to use

Select the powers to use for each term of the five-term fitting polynomial, set to zero to not use

first 1 second 2 third 3 fourth 0 fifth 0

Calculate and display the calibration

Let's go back and reduce the number of terms, as shown in the next 3 figures:

```

Output of the polynomial fit
3/3/99 11:39:00 PM Calibration using:
Rash Begone O.C.
Polynomial coefficients are:constant 1 2
      coefficient  standard deviation  significance=coeff/st dev
      -0.10392      0.19543          -0.53172  constant term
      0.00656       0.00012          52.70196  first term
      0.00000       0.00000          0.67195   second term

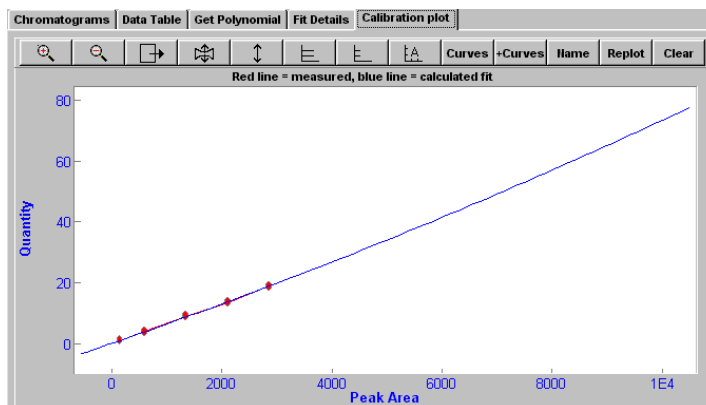
Before the equation was fitted:
9.30000 was subtracted from the data on the vertical axis
and
1413.49200 was subtracted from the data on the horizontal axis.

      FTest = 1.46E+003
      RMul = 0.99966
      For term 1, R1 = 0.99958
      For term 2, R2 = 0.22278

Appreciable changes in the value of FTest as more coefficients are added

```

This result is visually a lot better, but the statistics still suggest that the second term is not needed. Among other indicators, the FTest is still 1.46×10^3 .



We try again with one term, the linear term of $n = 1$:

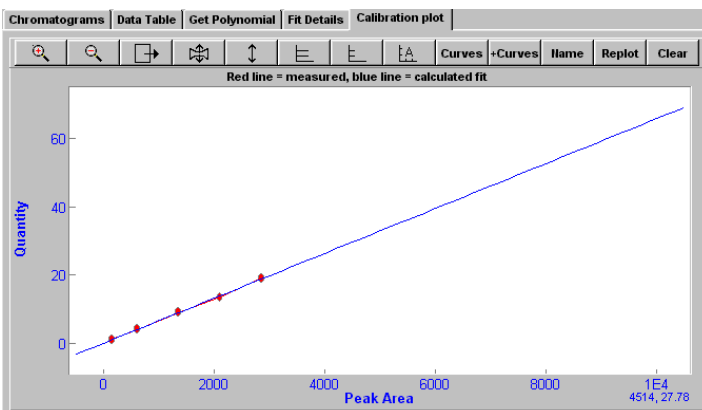
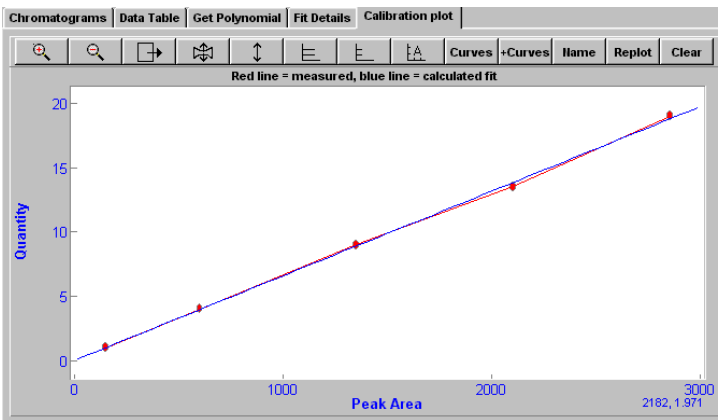
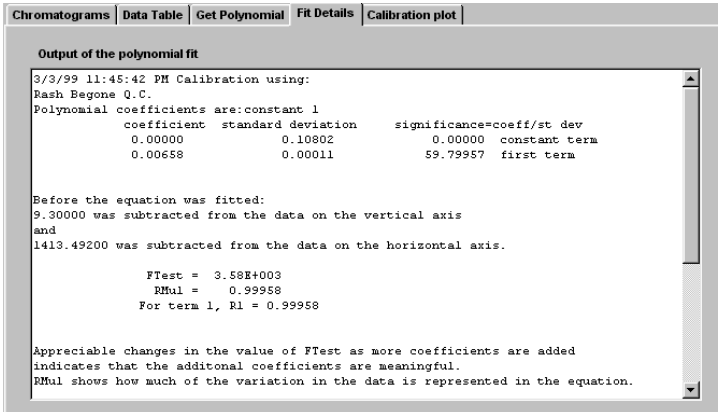
Polynomial powers to use

Select the powers to use for each of the five-term fitting polynomial, set to zero to not use

first second third fourth fifth

Calculate and display the calibration

The value of the FTest is now considerably larger than it was when 2 and 3 coefficients were estimated. This tells us that fitting a polynomial with one term (the linear term) is better than fitting a polynomial with a linear and a higher term. This finding is confirmed by the R values, which are now all high (near 1). Extrapolation does not produce disturbing skids to the side of the line through the data, as expected here because we have not allowed any curvature (terms higher than 1).

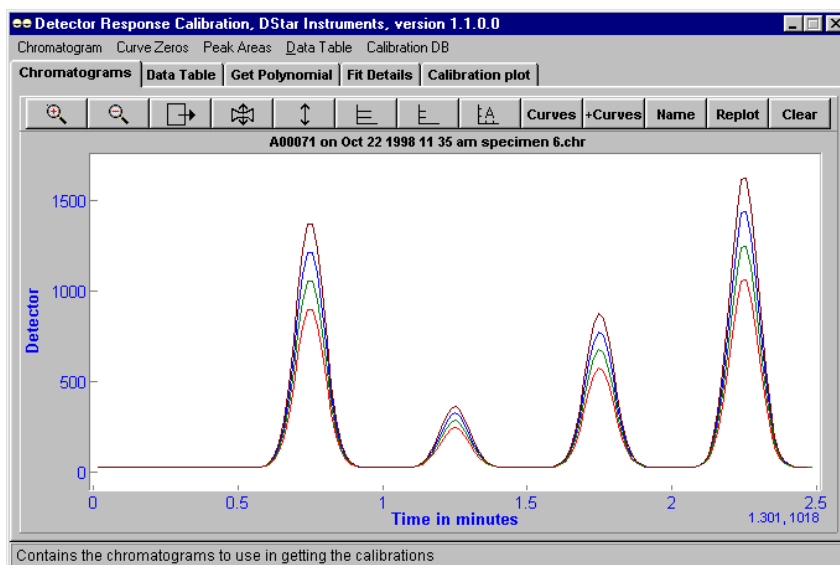


Example of Making a Calibration Curve

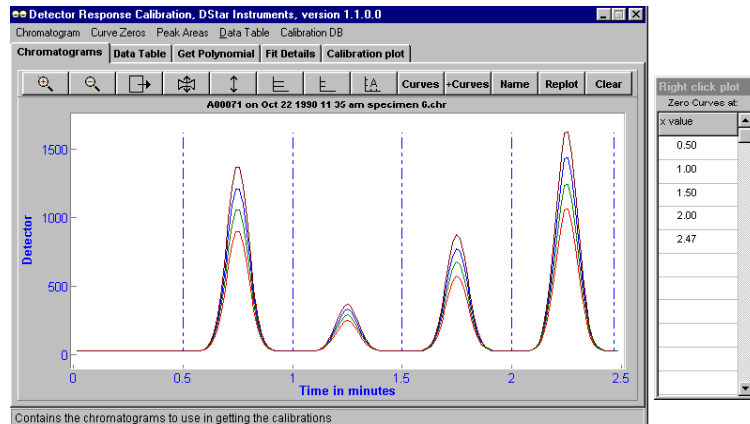
Before you can calculate a calibration curve and create a record you need to inject a set of standards and save the data using the Star-Chrom HPLC Management System.

Once this has been done you then need to load the chromatograms into the plot screen. This is accomplished by selecting the menu item “Chromatogram”. You next need to select the data files to load. You can select more than one file at a time.

The next action you will take is to establish where to zero the baseline. First select the menu item “Curve Zeros”. Next select the item “Show the Zero Places”. Right click the mouse button at selected places along the baseline. The number and placement will depend on how flat the baseline is.



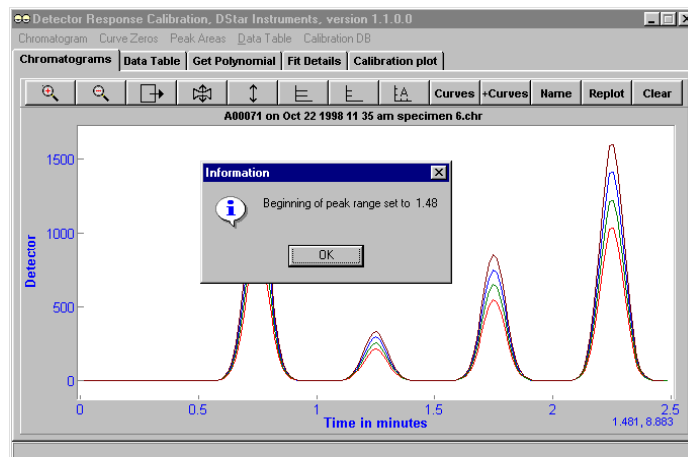
You should now see a screen similar to the one below



Now zero the curves using the menu item “Zero the Curves” found in the menu item “Curve Zeros”. Turn off the zero table by clicking on the close box button at the upper right corner of the zero table. This also will zero the curves

Next you need to identify which peak or retention time window will be used to generate the record. You can only select one peak per record. Select the menu item “Peak Areas” and left click on the item ”Select Peak Limits”.

Now right click the mouse on the baseline where you want to start looking for the target peak. You will see the following.



Press the “OK” button and select the point on the baseline that marks the end of the retention time window. Right click the mouse and press the “OK” button.

Now select the “Data Table” tab.

Detector Response Calibration, DStar Instruments, version 1.1.0.0

Chromatogram Curve Zeros Peak Areas Data Table Calibration DB

Chromatograms Data Table Get Polynomial Fit Details Calibration plot

Title

Class of Cmpd

Detector ID

Operator

Begin peak at 1.48 End peak at 1.96 Baseline/Tangent

Compound ID

Detector "Number"

Show Clipboard - put peak data on clipboard using Peak Areas menu

Put the sample quantity/peak area data here

Quantity	Area	Use? (y/n)

Read Info From DB Record

You can see that the “Begin peak at” and “End peak at” are already filled in. Continue filling in the rest of information.

Detector Response Calibration, DStar Instruments, version 1.1.0.0

Chromatogram Curve Zeros Peak Areas Data Table Calibration DB

Chromatograms Data Table Get Polynomial Fit Details Calibration plot

Title Rash Begone Q.C.

Class of Cmpd Steroids

Detector ID DStar var uv s/n 54321

Operator Tom Finn

Begin peak at 1.49 End peak at 1.96 Baseline/Tangent

Compound ID hydrocortisone

Detector "Number" 215 nm

Show Clipboard - put peak data on clipboard using Peak Areas menu

Put the sample quantity/peak area data here

Quantity	Area	Use? (y/n)

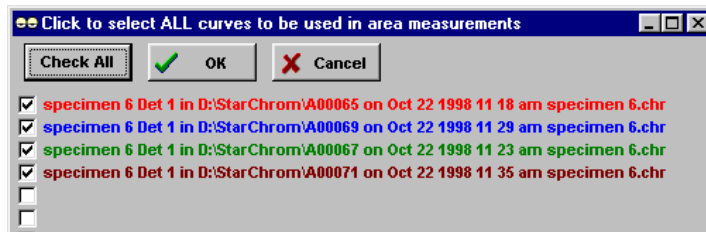
Read Info From DB Record

Peak area for standard 0 - can not be edited

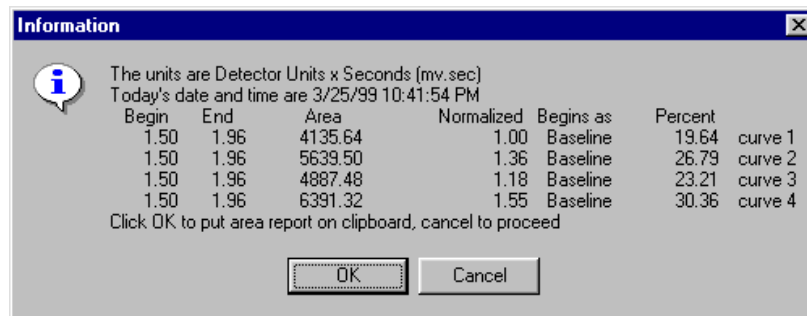
Next you will need to calculate the areas for the target peak in each curve.

Select the menu item “Peak Areas” and then left mouse click on “Get Peak Area Measurement.”

Press the “Check All” button and then the “OK” button.



You will see the following screen. Press the “OK” button.



Now you will see the following window. You can move the window by putting the cursor on the header bar. You will see a 4-way arrow. Left click and hold the mouse button and drag the window to a new position.

Area	Begin	End	B or T	Curve Name
4135.645	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00065 on Oct 22 1998 11 18 am specimen 6.ch
5639.504	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00069 on Oct 22 1998 11 29 am specimen 6.ch
4887.477	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00067 on Oct 22 1998 11 23 am specimen 6.ch
6391.315	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00071 on Oct 22 1998 11 35 am specimen 6.ch

Arrange the two windows as shown. Make sure that the “Data Table” tab screen is visible.

The image shows two overlapping windows from the 'Detector Response Calibration' software. The top window, titled 'Peak Areas As Found', contains a table with the following data:

Area	Begin	End	B or T	Curve Name
4135.645	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00065 on Oct 22 1998 11 18 am specimen 6.ch
5639.504	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00069 on Oct 22 1998 11 29 am specimen 6.ch
4887.477	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00067 on Oct 22 1998 11 23 am specimen 6.ch
6391.315	1.50	1.96	B	specimen 6 Det 1 in D:\StarChrom\A00071 on Oct 22 1998 11 35 am specimen 6.ch

The bottom window is the 'Detector Response Calibration' software, version 1.1.0.0. It has tabs for 'Chromatograms', 'Data Table', 'Get Polynomial', 'Fit Details', and 'Calibration plot'. The 'Data Table' tab is active. The title is 'Rash Begone Q.C.'. The 'Class of Cmpd' is 'Steroids', 'Compound ID' is 'hydrocortisone', 'Detector ID' is 'DStar var uv s/n 54321', and 'Detector "Number"' is '215 nm'. The 'Operator' is 'Tom Finn'. The 'Begin peak at' is 1.49 and 'End peak at' is 1.96. Below this is a table with the following data:

Quantity	Area	Use? (y/n)
	4135.645	y

A 'Read Info From DB Record' button is visible to the right of the table. At the bottom of the window, it says 'Peak area for standard 1 - can not be edited'.

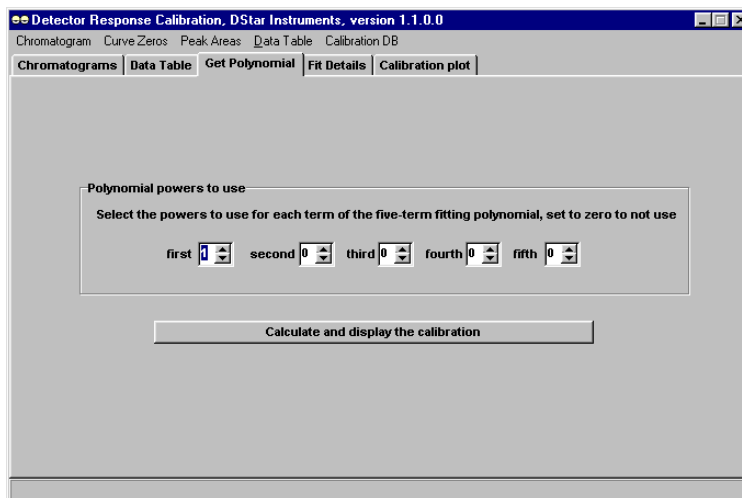
Put the cursor in the “Peak areas as found” table in the “Areas” column. Left-click and hold the mouse button down. Now drag the cursor to the area column on the “Data Table” page and release the mouse button. You can see that the area is moved into the table. Continue this until all areas are transferred. Next you will need to enter the actual quantity injected next to each area.

The image shows the 'Detector Response Calibration' software, version 1.1.0.0, with the 'Data Table' tab active. The title is 'Rash Begone Q.C.'. The 'Class of Cmpd' is 'Steroids', 'Compound ID' is 'hydrocortisone', 'Detector ID' is 'DStar var uv s/n 54321', and 'Detector "Number"' is '215 nm'. The 'Operator' is 'Tom Finn'. The 'Begin peak at' is 1.49 and 'End peak at' is 1.96. Below this is a table with the following data:

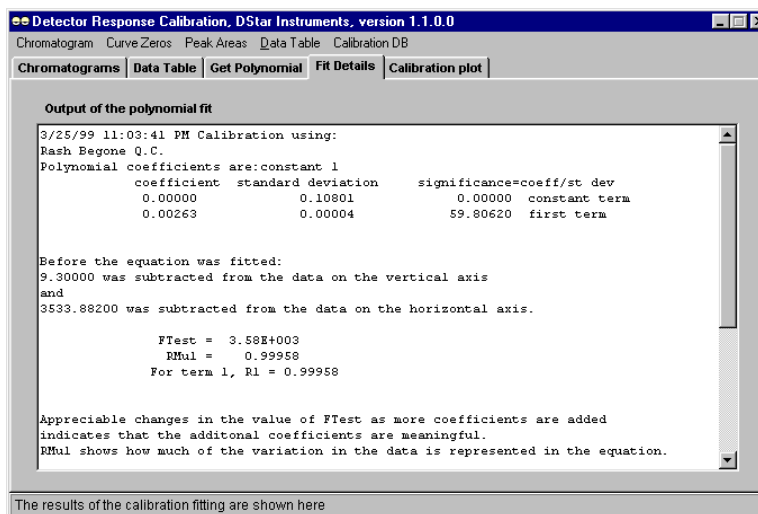
Quantity	Area	Use? (y/n)
1.000	375.95	y
4.000	1503.77	y
9.000	3383.50	y
14.000	5263.16	y
19.000	7143.03	y

A 'Read Info From DB Record' button is visible to the right of the table. At the bottom of the window, it says 'An identifier for the compound so you can find the right line (record) in the Calibration DB'.

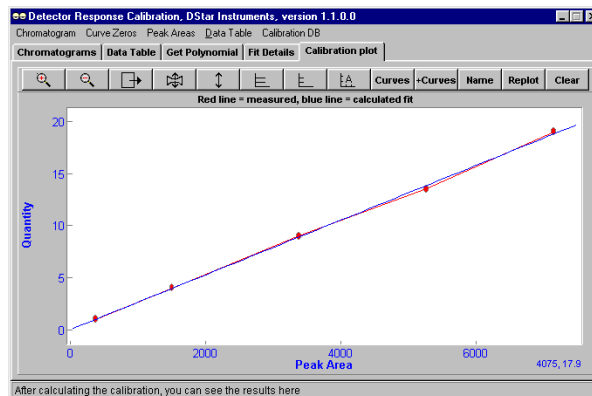
Now go to the “Get Polynomial” tab.



Start with the simplest fit, which would be a straight line. This is the default equation so just press the “Calculate and display the calibration” button. The “Fit Details” tab will display the statistics on how good a fit you got.

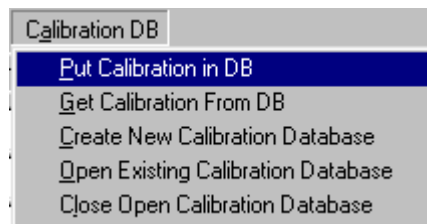


The “Calibration Plot” tab will show you the visual plot of how well the calculated curve fits the actual data.



Finally you will want to create a database file and save the record that you just finished generating.

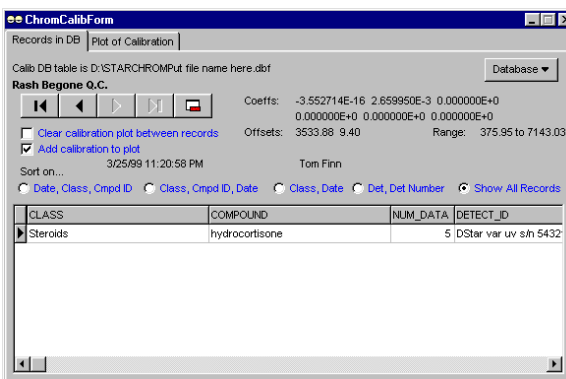
Select the menu item “Calibration DB” and then select “Create Calibration DB”.



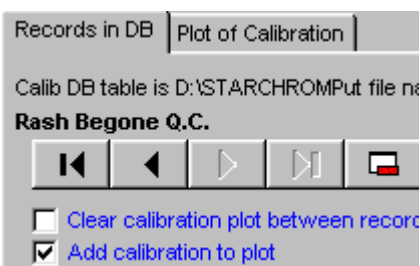
Now you need to make up a name for your database.

Press the “OK” button.

Now you need to put the record into the database. Select the menu item “Calibration DB” and then select “Put Calibration in DB” item. Show the Calibration DB window and you can see that your new record has been loaded into the database that you created.



Put a check mark into the “Add calibration to plot” box and press the VCR style button. This will select the current record and you will be able to see the curve on the ‘Plot of Calibration’ tab.



You can now close the Calibration program or select a new peak window and create a second record for the database.

Now you will want to apply the calibration curve to a real chromatogram and calculate the quantity of the target peak. This is done in the “Star-Chrom” program.

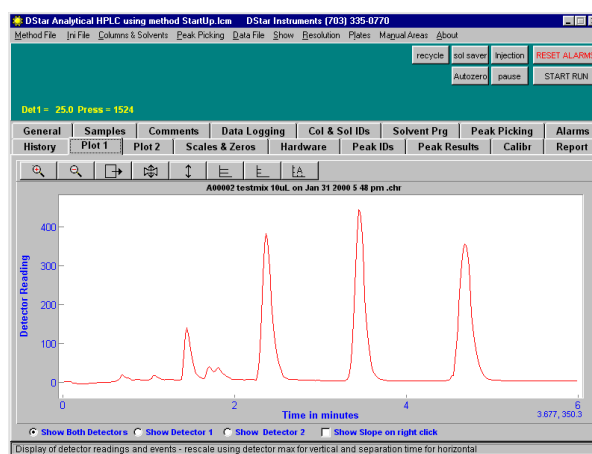
Chapter 6 – Example Part Two

Example of Applying a Calibration Curve

You need to close the Calibrations program first.

Next you will need to start the Star-Chrom program.

Then you will need to load a chromatogram from a run of the target assay.

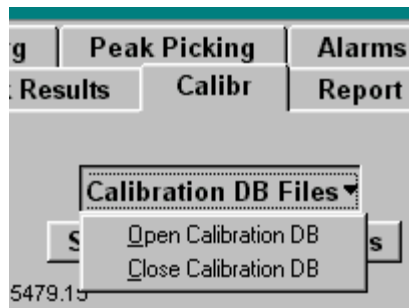


Go to the “Peak Ids” page and using the “Peak ID’s Parameters” button, erase the peak id’s.

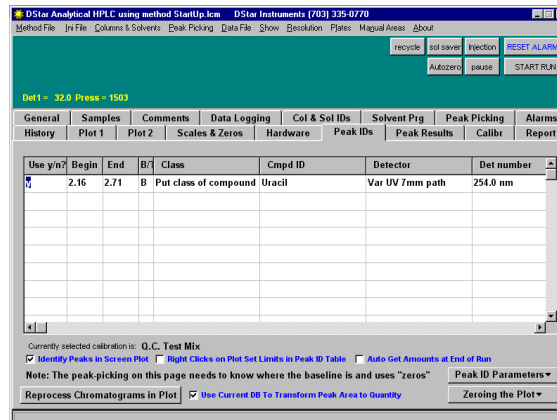
Use y/n?	Begin	End	B/I	Class	Cmpd ID	Detector	Det number
y	2.24	2.60	B	Put class of compound	Uracil	Var UV 7mm path	254.0 nm
y	3.26	3.66	B	Put class of compound	Phenol	Var UV 7mm path	254.0 nm
y	4.39	4.90	B	Put class of compound	Toluene	Var UV 7mm path	254.0 nm

Next select the “Calibr” tab. This will allow you to open a calibration database and record to apply to the chromatogram that you just loaded.

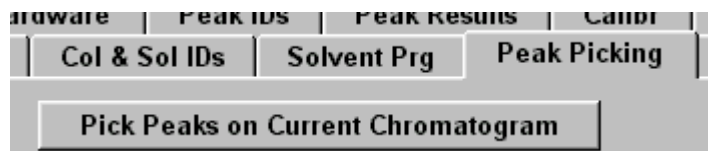
Press the “Calibration DB Files” button



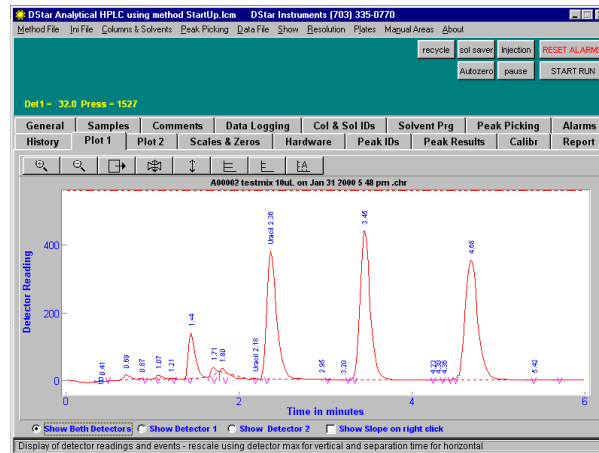
Select the calibration database. Now Select the record you want to use. Press the “Send Record to Peak ID’s” button. This will transfer the information needed to identify and quantitate the target peak.



Next press the “Pick Peaks on Current Chromatogram” button.



The “Plot” will appear with the target identified.



The “Peak Results” table shows the identified peak and its quantitation.

Peak #	Begin	End	Peak Area	Maximum	Time of max	Area %	Begin as	Quantity	Name
1	2.25	2.95	2943	383.55	2.36	28.9	Valley	10.471	Uracil
2	3.27	4.17	3605	445.41	3.45	35.4	Valley		
3	4.43	5.33	3634	356.84	4.68	35.7	Valley		

Area of all peaks found = 11190.34, 90.99% kept as peaks above minimum area % of 10.00

Finally you will want to print a report complete with your company logo. Go to the “Report” tab and press the button named “Print Plot”.

Short Reports to Printer

- Make All Reports Short
- Show Short Report as Preview
- Scale Plot To Individual Curve
- Print Color
- Print B/W

Print Report Choices

- Show Peak Picking Results
- Show Peak Resolution
- Show Plate Counts
- Show Feeds

Print Details Files

- Write Windows MetaFile File
- Write Bitmap (BMP) File

Report Names

Reporter name:

Put your name here

Organization name:

First line of organization's address:

Second line of organization's address:

Report Icon:

Change Report Icon

The report page

Here is an example report output.

