ARTISAN®
TECHNOLOGY GROUP

Your **definitive** source for quality pre-owned equipment.

Artisan Technology Group
(217) 352-9330 | sales@artisantg.com | artisantg.com

Full-service, independent repair center
with experienced engineers and technicians on staff.

We buy your excess, underutilized, and idle equipment
along with credit for buybacks and trade-ins.

Custom engineering
so your equipment works exactly as you specify.

- Critical and expedited services
- In stock / Ready-to-ship
- Leasing / Rentals / Demos
- ITAR-certified secure asset solutions

Expert team | Trust guarantee | 100% satisfaction

All trademarks, brand names, and brands appearing herein are the property of their respective owners.

Find the *Matec CHDF 2000* at our website: ***Click HERE***

Table of Contents

Frequently Asked Questions I-2

Chapter 1: Introduction to the CHDF 2000 I-6

1.1 CHDF 2000 Components I-7

1.2 CHDF 2000 Operation I-10

1.3 Using the Control Panel I-11

Chapter 2: Theory and Sample Preparation

2.1 Scope II-1

2.2 Safety Precautions II-1

2.3 Intended Use II-2

2.4 Description and Specifications II-3

2.5 Theory of Operation II-7

2.6 Data Analysis II-8

2.7 Sample Preparation II-8

2.8 Eluant II-9

Chapter 3 Setting Up the CHDF 2000

3.1 Performing Power-On Procedures III-2

3.2 Reviewing and Editing Operating Parameters III-7

Matec Applied Sciences

56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476

www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Chapter 4 Operating the CHDF 2000

4.1 **Priming the Pump** IV-1

4.2 **Analyzing Samples** IV-4

Chapter 5 Troubleshooting the CHDF 2000

5.1 **Responding to Error Messages** V-1

5.2 **Quick Troubleshooting Checklists** V-4

5.3 **Running CHDF 2000 Diagnostics** V-8

Chapter 6 Maintaining the CHDF 2000

6.1 **Maintaining the Pump** VI-2

6.2 **Maintaining the Manual Injector Valve** VI-12

6.3 **Maintaining the Priming Valves** VI-16

6.4 **Maintaining the Detector** VI-17

6.5 **Adjusting the Control Panel Display** VI-24

6.6 **Dealing with Plugged Cartridges** VI-25

Matec Applied Sciences

56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476

www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Matec Applied Sciences

CHDF 2000 Hardware Users Guide

Version 3.8

April 26, 2006

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

FREQUENTLY-ASKED QUESTIONS ABOUT CHDF OPERATION

1. *How frequently should the CHDF GR500 eluant be replaced?*

   The CHDF GR500 eluant degrades with time. It should be replaced every two to three days. Check the conductivity of the eluant in order to determine if it is suitable. The conductivity ranges for the different GR500 types are as follows (in microSiemens per centimeter): 1X-GR500: 7-10; 2X-GR500: 22-27; 2X-GR500-LA: 14-18; 1X-GR500 pH9: 13-18. The GR500 eluant absorbs Carbon Dioxide from the ambient which causes its pH to drop, and its conductivity to increase; this results in measured particle sizes smaller than expected. The 1X-GR500 pH9 eluant is especially sensitive to CO2 absorption. Bacterial growth also takes place as the GR500 ages.

2. *I obtain smaller than expected CHDF particle size results when I inject calibration standards.*

Most likely, your GR500 eluant is either degraded or contaminated. See Question 1 above.

3. *How is the GR500 eluant prepared?*

Add 950 ml of deionized water (conductivity should be less than 2 micro Siemens per centimeter) to 50 ml of GR500 concentrate. Eluant degassing by either sonication or vacuum filtration is recommended.

4. *How long does the GR500 concentrate last, and how should it be stored?*

The GR500 lasts about one month. It should be kept refrigerated.

5. *Does the Marker fluid need to be injected at an exact time for every run?*

No. In both manual, and auto-sampler operation, the software detects when the marker is injected. The injection time is recorded; the marker elution time is calculated as the marker exit time minus the injection time. A marker delay time of zero seconds is recommended for auto-sampler operation (this is set under the current method file).

6. *How should the marker fluid be prepared?*

The marker fluid consists of a solution of sodium benzoate in deionized water. Its concentration varies depending on the type of fractionation cartridge used. Typical concentrations are in the range 0.1% to 0.5%. This concentration should be adjusted in order to obtain a reasonable marker peak height (displayed on the CHDF software Raw Data graph) upon marker injection into the CHDF. Marker peaks heights of 10 to 40 mAU's (milli Absorbance Units) for the CHDF-2000 instrument, and 5,000 to 15,000 counts for the CHDF-1100 are acceptable.

7. *Why is the pump pressure fluctuating more than 200 psi during a run?*

Air bubbles are lodged in the pump pistons. Stop the pump; open the pump purge valve (located on the lower right- hand corner of the CHDF-2000; or on the CHDF-1100 pump front panel); insert a 5-ml plastic syringe into the purge valve, and slowly draw fluid. Air bubbles in the pump and pump-inlet tubing should be removed. Tighten the purge valve and start the pump. If the air bubbles re-appear, the Whatman de-gassing filter on the pump-inlet tubing should be replaced. This filter lasts about one month. Cavitation, which results in air bubble generation, occurs if the Whatman filter needs

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

replacement. For the CHDF-2000, this filter is optional. You may operate your CHDF-2000 without this filter if you so prefer.

8. *Noise and drift are present in the Raw Data baseline*.

This is probably due to a defective Whatman filter. Replace this filter and observe the baseline. This filter must be filled with eluant when in operation. Use the purge knob on the filter is order to draw any air from the filter. Another possibility is that the UV lamp may need replacement if it has been used for more than 1000 hours (see the troubleshooting section of the hardware manual).

9. *How should samples be prepared for CHDF analysis?*

Only aqueous samples can be analyzed on the current CHDF setup. Dry powders must be dispersed in GR500. Sonication should be applied in order to help suspend the particles in the liquid. Concentrated samples should be diluted in GR500 eluant. The solids contents are typically in the range 0.1% to 5% by volume. UV-absorbing samples can be analyzed at lower concentrations while non-UV absorbing samples (such as colloidal silica) must be run at higher concentrations. Sonication may be used in order to break-up particle aggregates. Reasonable sample concentrations should produce peak heights of 20 to 50 mAU's for the CHDF-2000, and 5,000 to 15,000 counts for the CHDF-1100. A syringe filter may be used on the sample syringe in order to remove aggregates from samples prior to injection. Syringe filters may only be used once per sample. Marker, as well as, Rinse filters may be reused until leaks are observed on the filters.

10. *No peaks are detected in the Raw Data graph upon sample, and marker injection*.

The fractionation cartridge is likely plugged. Refer to the troubleshooting section of the hardware manual for cartridge cleaning instructions.

11. *The pump pressure is higher than normal*.

The filter element in the cartridge in-line filter must be either cleaned or replaced. Refer to the troubleshooting section of the hardware manual. The CHDF-1100 is affected by ambient temperature. Low ambient temperatures result in high CHDF-1100 pressures.

12. *What Noise Level should be used under Configure System in the CHDF software?*

30-40 for the CHDF-2000; 400 for the CHDF-1100.

13. *Can any wavelength be used on the CHDF UV-detector?*

The CHDF UV-detector wavelength can be varied from 190 to 380 nm. However, the CHDF PC software only contains particle extinction cross section curves for 200 nm, and 220 nm. One of these values must be selected in the active Method file. Inaccurate results are obtained if the UV-detector, and the software wavelengths differ.

14. *What is the sharp peak in the Raw Data between the sample peak and the marker peak?*

This peak corresponds to UV-absorbing molecular species present in the sample. Molecular species include surfactants, electrolytes, acids, or bases. These molecules have a smaller size than the particles themselves, and therefore exit the capillary after the particles.

15. *A broad peak after the marker peak is displayed in the Raw Data*.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

This peak is likely due to non-ionic surfactant. Non-ionic surfactants can continuously adsorb and desorb from the capillary wall as they travel down the fractionation capillary. This adsorption/desorption retards the non-ionic surfactant from exiting the capillary.

16. *Can the marker be blended with the sample so that only one injection is made per sample?*

Yes, in manual operation only. Simply add maker fluid to your samples, and make only the sample injection (do not inject the marker).

17. How long should the instrument be allowed to equilibrate (or warm up) at the beginning of the day before starting sample analysis?

Approximately 30 to 45 minutes.

18. Can the CHDF be turned off at the end of the day, or should it be left running overnight?

The CHDF can be operated in either mode. We prefer to turn it off at the end of the day.

19. How much sample, and marker, should be Loaded into the injection valve?

100 microliters for manual operation. It is recommended that at least 400 microliters be drawn into the syringe in order to minimize the chances of loading air bubbles into the injection valve. If using a syringe filter make sure the filter is filled with sample before loading sample into the injection valve. 80-90 microliters is recommended for auto-sampler operation. This is set under System/Configure Auto-Sampler.

20. Are samples loaded directly into the fractionation capillary?

No. They are loaded into a sample loop (a piece of stainless steel tubing) which is connected to a vent. As sample is pushed into the injection valve, the sample loop becomes filled with sample. Sample exits from the sample loop vent as more sample is loaded. The sample loop becomes connected to the pump, and the fractionation capillary upon switching to Inject.

21. Can the CHDF software be run under Windows9X, NT, and 2000?

Yes, the CHDF2000 software can be run on any Windows operating system software.

22. What solids concentration should be used for calibration standards?

Calibration standards should not be overly concentrated. Solids concentrations should be such that Raw Data peak heights are 10 to 30 mAU's for calibration standards and 20-60 for regular samples. Calibration standards larger than 300 nm should be sonicated prior to use in order to break up aggregates.

23. How frequently should the CHDF capillary be calibrated?

Calibration shifts are related mainly to two factors: (i) changes in GR500 eluant composition; and (ii) changes in ambient temperature (not important for the CHDF-2000). It is recommended that the conductivity of the GR500 eluant be monitored; replace the eluant if the conductivity is outside its normal range. Calibration standards can be run prior to regular sample analysis in order to confirm calibration accuracy. These "control" standards may be added to the calibration curve if re-calibration is deemed necessary.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

24. How long does a CHDF cartridge (capillary) last?

Fractionation capillaries do not have an exact life span. Cartridges may last several years since no particle adsorption occurs inside the capillary. Capillary failures are frequently due to plugging right at the capillary inlet. Cutting-off ("snipping")a short piece at the capillary inlet frequently removes the plug. The capillary can also be back-flushed as described in the hardware manual, troubleshooting section.

25. How is the Weight Particle Size Distribution (PSD) calculated?

The particle weight percent is calculated as the Number percent multiplied by the particle diameter raised to the third power: $Wt_1\ \% = N_i\%(D_p)^3$. This assumes that the particle density is the same for all particles in the sample. The area percent is calculated as follows: $A_i\% = N_i\%(D_p)^2$

26. What particle Size Reproducibility should be expected from CHDF

About ± 5%.

27. What is the mean, 25%, median, 75%, and FWHH in the text PSD report?

The 25%, median, and 75% are the 25th, 50th, and 75th percentiles of the particle size distribution (PSD). They are calculated as the particle size evaluated at those values of the cumulative PSD. Physically, for the 25% for example, they indicate that 25% of the particles are below the reported particle size. The FWHH is the PSD Full Width at Half Height. It is the width of the differential PSD at 50% height.

28. What is the list of particle sizes at the bottom of the Text Report?

This list contains a description of all the particle size populations (also referred to as modes) automatically detected by the software in the sample. Each mode is listed by the start and end particle size. The percentages by Number, Area, and Weight are printed. The Area% is the average particle size of each mode (Weight-Weighted). The mode detection sensitivity can be changed in the current method under Optical Mode Detector. Lowering the value of Slope Sensitivity increases mode sensitivity.

29. How can the Particle Size Distribution Report be Customized or Changed?

Open your current CHDF method, and select "Report Setup". The text report, if used, should be placed at the bottom of the report.

30. What causes noticeable "tailing" in a fractogram?

The inlet to the fractionation capillary is likely dirty. Snip the fractionation (top) capillary (see hardware manual section 6.6).

31. How are odd-shape particles, e.g., discs, rods analyzed?

Particles spin in the capillary tube during CHDF fractionation. As a result, particles approach the capillary wall at different orientations. Some positions result in an exclusion layer equal to the longest particle dimension while other positions do the shortest or intermediate dimensions. The resulting elution time is equivalent to an average of all the particle dimensions. Research done at Lehigh University showed that particle doublets generated from partial latex coagulation produce a CHDF particle size equal to one particle diameter multiplied by the square root of two, or 1.41*Dp (instead of 1.5*Dp). Triplets appear to produce one diameter times the square root of 3.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

32. *Does the CHDF software extrapolate beyond the two calibration standards at the limits of the calibration curve?*
The CHDF reports particle size populations in the range specified in the CHDF2000 software under system/Configure-System. On the small particle size end, the CHDF interpolates below the smallest standard and the data point Rf=1, Dp=0, where Rf is the separation factor for a near-zero particle size (Dp) species such as the marker fluid. In fact, if you run the marker as a sample, you obtain Rf=1. Rf is calculated as the particle elution time divided by the marker elution time. Rf increases with increasing particle size as shown in the calibration curve. Elution time is peak exit time minus injection time. For larger particles, the software does extrapolate using a curve-fitting expression of the calibration-standard data points (up to the upper limit specified under System/Configure-System. This curve fit can be a sigmoidal or polynomial fit. Both options have sigmoidal shapes.

33. *What is Particle Frequency in the PSD graphs?*
"Particle Frequency" and "Volume Percentage" are different. The CHDF produces relative PSD's.

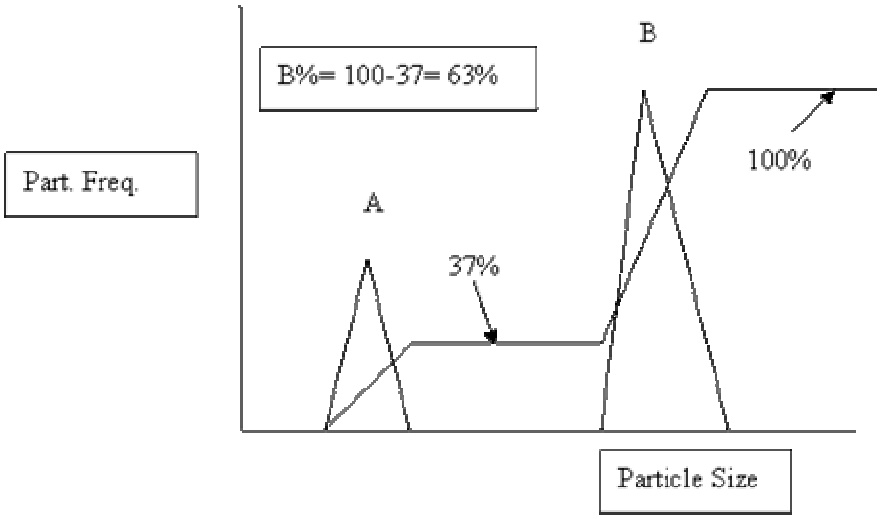
An absolute PSD shows the actual particle volume percentage. However, absolute PSD's are sometimes difficult to compare between different samples (overlay). The reason is that samples with different percent solids will show different PSD-peak heights.

Relative PSD's as currently shown by the CHDF are normalized so that the tallest peak or peaks have a height of 100 "frequency units". These are not percentage values because there can be two or more peaks with the same 100 value. Also, next to the 100 point, there are values of 98, 92, 86, and so on. If you add all these together, you would obtain much more than 100% for the sample.

The particle frequency can be used to compare two populations. For example, a 37% peak has half the percentage of a 74% peak.

If you need to know the percentage of a population, you should use the cumulative distribution (CD). You can subtract the CD values at both ends of each peak. This gives you the percentage of this peak. See example graph below.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



34. ***How can the percentage of a particle-size population be calculated?***

See “Particle Frequency” question above.

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

35.

1 Introduction to the CHDF 2000

The CHDF 2000 (Figure 1-1) uses the principle of Capillary Hydrodynamic Fractionation (CHDF) to measure particle sizes from 15 nanometers to 1.1 microns in diameter.

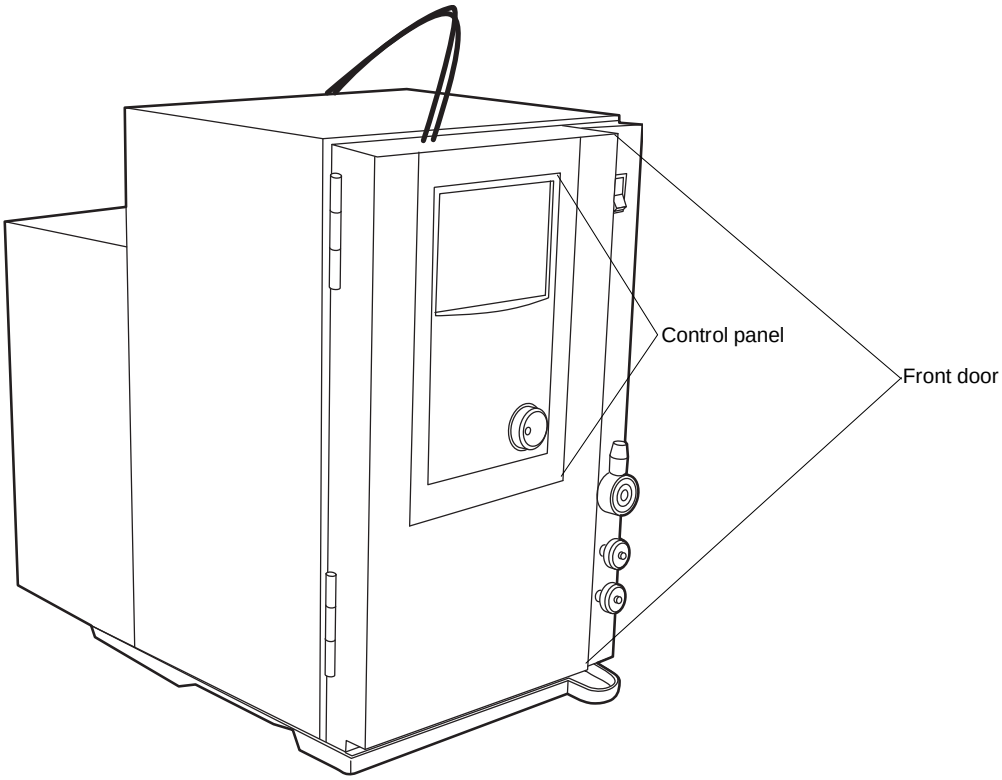


Figure 1-1 CHDF 2000

The CHDF 2000 provides the following features

- Simple front panel control of all system operations
- An eluant delivery system
- UV absorbance detection
- Reduced workbench space requirements

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.1 CHDF 2000 Components

1.1.1 System Components

The CHDF 2000 contains the following components:

- Control panel
- Manual injector valve
- Eluant Pump
- Fractionation Cartridge
- Absorbance detector
- Signal connector module

Control Panel

The CHDF 2000 control panel (Figure 1-2) provides a simple icon-based interface that allows you to directly configure, operate, and troubleshoot the CHDF 2000 . The control panel is located on the CHDF 2000 front door. Refer to Section 1.3, Using the Control Panel, for detailed instructions on using the CHDF 2000 control panel.

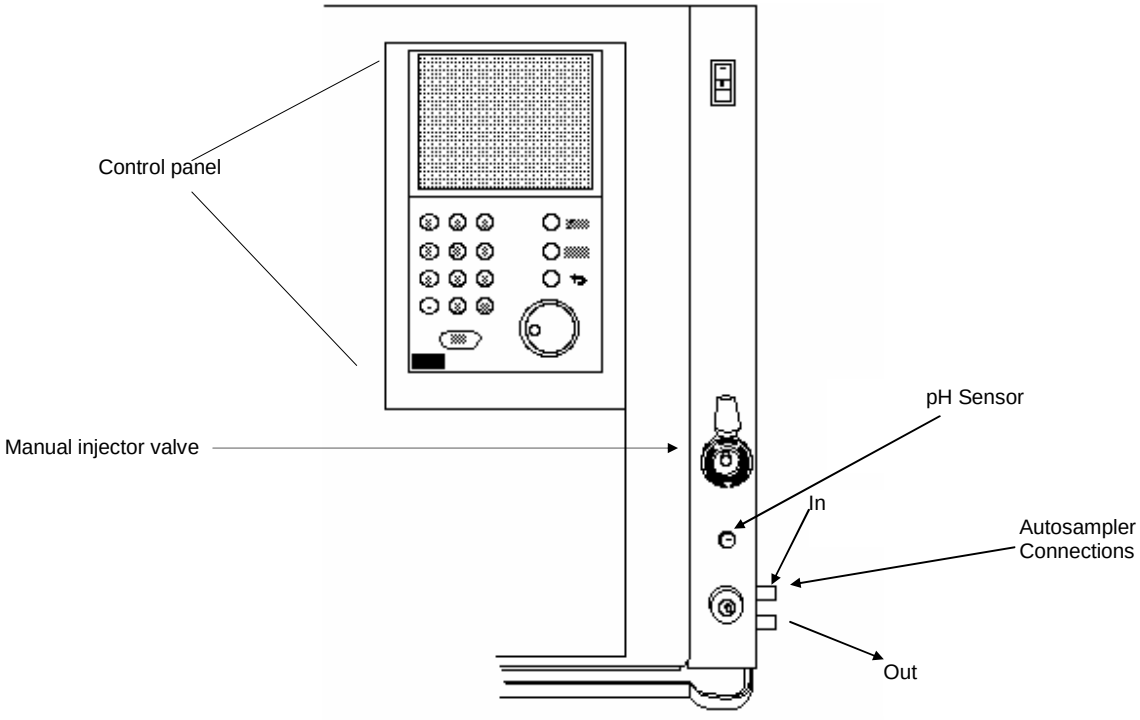


Figure 1-2 Control Panel and Manual Injector Valve

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Manual Injector Valve

The CHDF 2000 manual injector valve (see Figure 1-2) provides a port for loading a sample and a manual rotation handle to inject the sample into the eluant stream for analysis. The injector valve is mounted to the right of the front door.

An autosampler option is also available for automated sample injection.

Eluant Pump

The base system pump supplies the eluant for CHDF 2000 operation. You can access the pump behind the front door (Figure 1-3). A priming valve is connected to the pump outlet and is mounted directly below the manual injector valve.

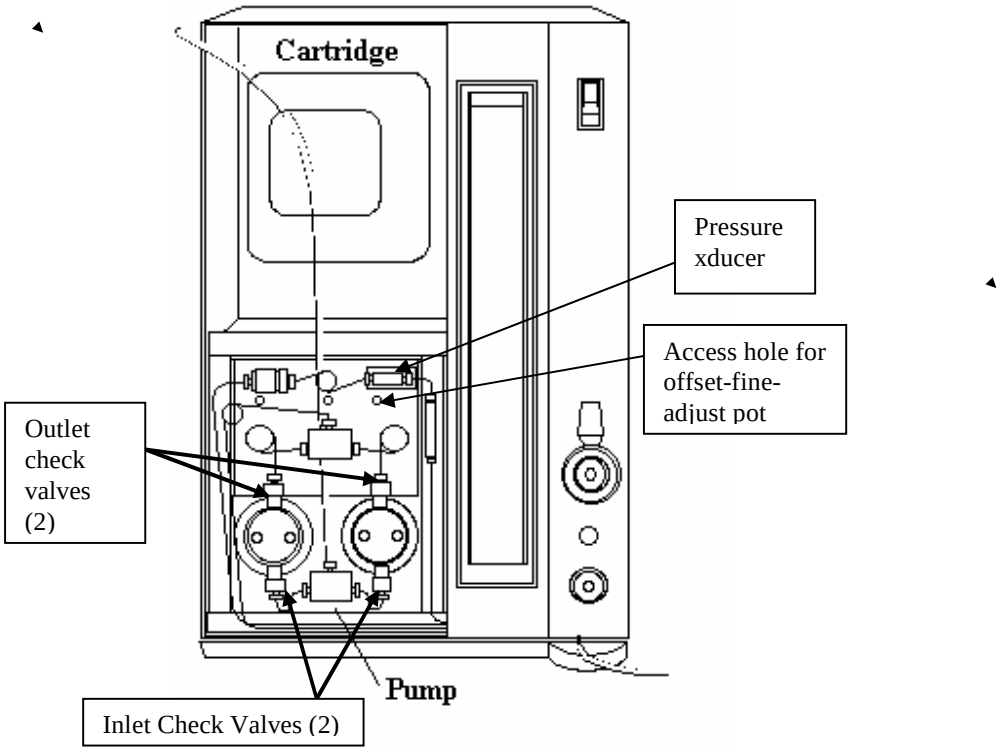


Figure 1-3 Eluant Pump and Fractionation Cartridge

Fractionation Cartridge

In the CHDF2000, a capillary column provides hydrodynamic separation of the sample

Instructions for installing the column are provided in Chapter 6.

Absorbance Detector

The CHDF 2000 includes a multi-wavelength, tunable, ultraviolet (UV) absorbance detector (Figure 1-4). The UV detector measures the Ultraviolet light extinction of particles in the flow stream from the column outlet.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

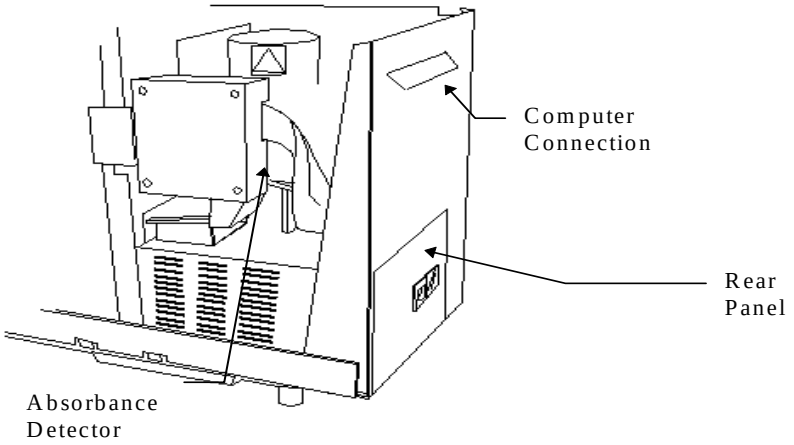
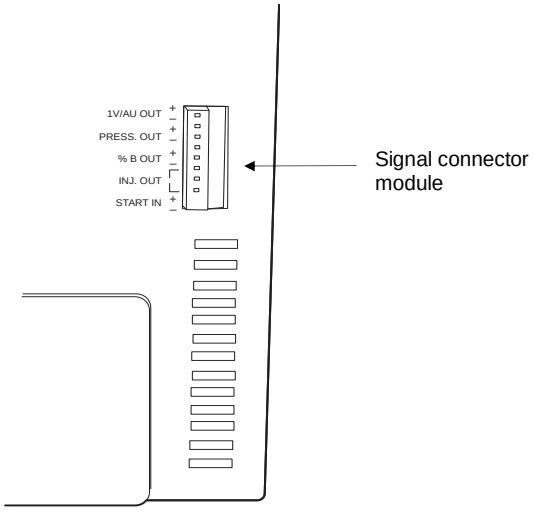


Figure 1-4 Absorbance Detector

Computer Connection

A 9 pin, RS-232 connection is used to provide data transfer between the CHDF 2000 and a personal computer. A Pentium™ processor and Windows '95™ are recommended for the personal computer.

Signal Connector Module



The signal connector module (Figure 1.5) on the CHDF 2000 rear panel provides signal connections for external devices such as a chart recorder or integrator. You can also use these signals to synchronize the operation of the CHDF 2000 with other devices.

Figure 1-5 Signal Connector Module

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.2 CHDF 2000 Operation

1.2.1 Operation

Figure 1-6 is a fluidic diagram illustrating CHDF 2000 operation.

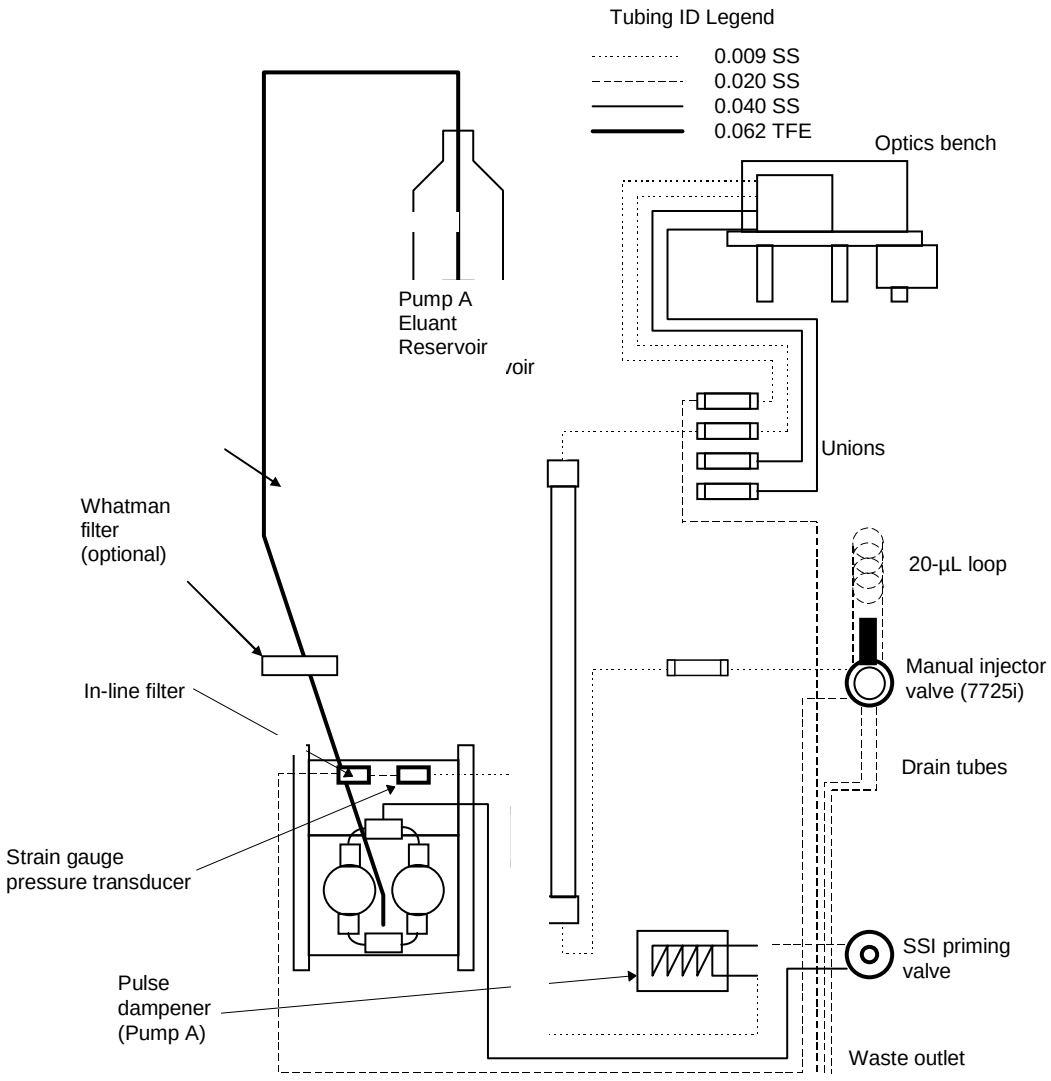


Figure 1-6 Fluidic Diagram of Operation

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.3 Using the Control Panel

The CHDF 2000 control panel provides easy control of system configuration, operation, and diagnostic functions.

The control panel components (Figure 1-7) include:

- Control panel display
- System controls

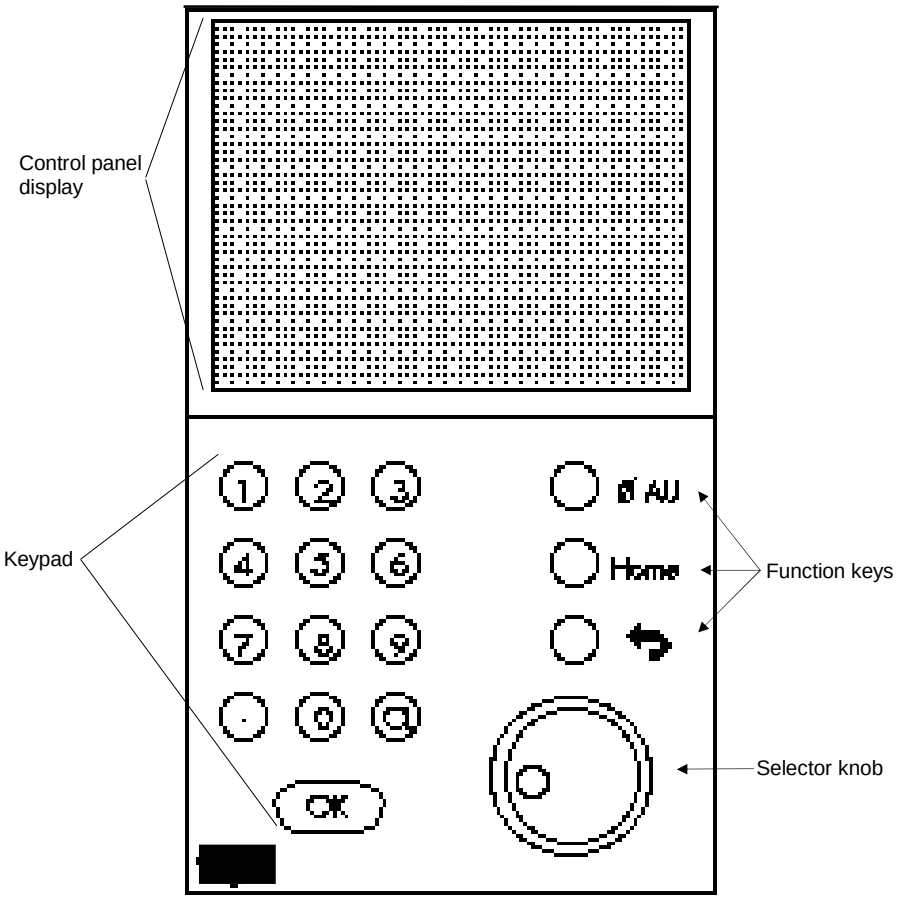


Figure 1-7 Control Panel Components.

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.3.1 Control Panel Display

The CHDF 2000 control panel display consists of four sections (Figure 1.8)

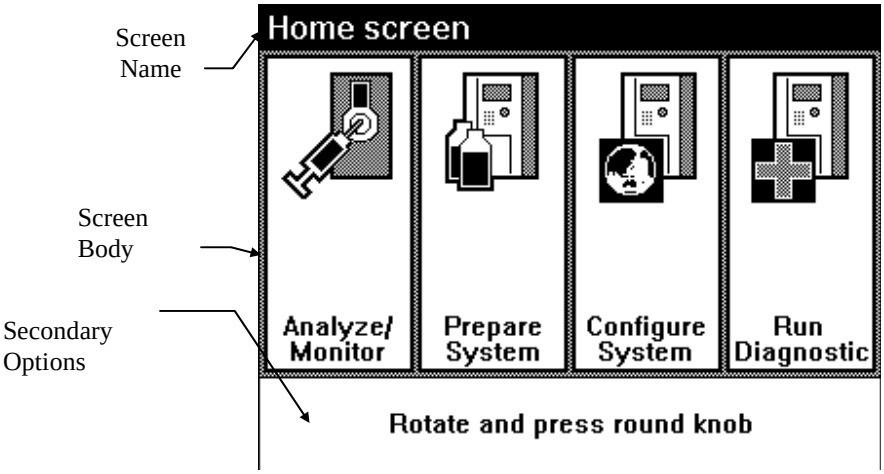


Figure 1-8 Control Panel Display Sections

Screen name – A screen name appears at the top of each screen to identify the specific program screen.

Screen body – The main body of each screen contains one or more of the following types of information:

- Control options, including system tasks and navigation options
- Status of current operation
- Instructions

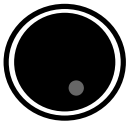
Secondary options or **instructions** – Secondary options or instructions appear at the bottom of most screens. Secondary options allow you to choose alternate navigation or control options.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.3.2 System Controls

System controls are just below the control panel display on the CHDF 2000 front door. Table 1-1 identifies the function of each CHDF 2000 control.

Table 1-1 Control Panel

Control	Function	Description
0 – 9	Number values	Used in combination to insert a value for a selected parameter.
.	Decimal point	Inserts a decimal point in a value for a parameter.
CL	Clear	Clears an entered value.
OK	Execute	Selects a highlighted option. Alternative to pressing the selector knob to select a highlighted option.
∅AU	Autozero Detector	Resets the absorbance detector output signal to the zero offset value.
Home	Go Home	Returns the control panel display to the Home screen.
⇦	Go Back	Returns the control panel display back to the preceding program screen.
	Selector knob	Rotate to change highlighted screen option or to increase or decrease a value. Press to select a highlighted option or to accept a displayed value.

1.3.3 Moving Within and Between Screens

The default option for a screen is highlighted when the screen first appears.

Moving Among Screen Options

To highlight an option on the screen, rotate the selector knob to move the cursor from screen option to screen option. As you move the cursor to a new screen option, the option appears highlighted.

For example, when you first access the Home screen, the Analyze/Monitor screen option appears highlighted (see Figure 1.8). To highlight the Configure System option on the Home screen, rotate the top of the selector knob to the right two times. The Configure System option appears highlighted.

Moving Between Screens

When you choose a screen option, the control panel display shows the screen for the option you chose. The default screen option for the new screen appears highlighted.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

1.3.4 Choosing a Screen Option or Parameter

To execute a screen option or edit a parameter, you must first choose it. Choosing a screen option or parameter requires that you:

1. Highlight the screen option or parameter.
2. Accept the screen option or parameter.

To choose a screen option or parameter:

1. Rotate the selector knob to highlight the screen option or parameter.
2. Press the selector knob to accept the screen option or parameter.

   or

   Press **OK** on the keypad.

1.3.5 Entering Parameter Values

You can enter parameter values by using either the selector knob or the keypad.

Using the Selector Knob

To enter a value using the selector knob:

1. Choose the parameter you wish to change.
2. Rotate the selector knob to change the parameter value.
3. Press the selector knob to accept the displayed value.

Using the Keypad

To enter a value using the keypad:

1. Choose the parameter you wish to change.
2. Enter the desired value using the keypad.
3. Press **OK** to accept the displayed value.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2: Theory and Sample Preparation

The CHDF 2000 determines the particle size distribution of samples suspended in aqueous solutions in the range of 15 to 1100 nm in diameter. The size distribution is determined by the measurement of the particle elution time in a series of capillary tubes.

The CHDF 2000 requires an IBM compatible computer, preferably equipped with a Pentium™ processor and Windows™ '9X or higher. The operating software gives graphical displays and a hard copy of the cumulative weight, area, and number distributions, the relative weight, area and number distribution, the weight, volume, and number average particle size, and the particle size distribution standard deviation. The measurement time, from sample injection to final calculation, is under fifteen minutes.

2.1 SCOPE

The purpose of this chapter is to provide an overall description of Capillary Hydrodynamic Fractionation. The topics covered are:

1. Scope
2. Safety
3. Intended Use
4. Description and Specifications
5. Theory
6. Data Analysis
7. Sample Preparation
8. Eluant

2.2 SAFETY PRECAUTIONS

2.2.1 Electrical

The pump and the detector in the CHDF 2000 use high voltage. Avoid contact with any high voltage area. Un-plug the entire CHDF 2000 before performing service or maintenance. Do not store or spill eluant or samples on the electrical modules. Be certain that all laboratory power receptacles are properly grounded.

The CHDF 2000 uses ultraviolet radiation. Avoid eye contact with any ultraviolet source without suitable eye shielding or protection. The UV source is fully contained within the CHDF 2000 casing.

2.2.2 Mechanical

The CHDF 2000 pumps a fluid through a set of capillaries at a relatively high pressure. The compressibility of liquids is low, therefore a system component will probably leak before excessive pressure causes other components to fracture. Always wear appropriate eye protection in the laboratory. The actual fractionation capillaries are fully contained in a removable cartridge. The potential hazard from a fracture or breakage in one of the capillaries is greatly reduced. Do not attempt to remove the capillaries from the cartridge or tamper with the internal plumbing of the cartridge.

Do not attempt to perform service while the system is pressurized. Allow the system pressure to drop below 1000 PSI before loosening any tubing fittings.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.2.3 Chemical

The GR-500 eluant for use in the CHDF 2000 consists of surface active agents in deionized water and to our knowledge poses no chemical danger. There are currently three versions of the GR500 eluant: 1X-GR500, 2X-GR500 (standard), and 2X-GR500-LA. Never use any eluants besides those supplied by Matec Applied Sciences. All samples must be suspended in water.

The marker used in the CHDF 2000 is a solution of sodium benzoate (benzoic acid) in deionized water. The marker concentration can be 0.2-1.0 wt. % depending on the kind of cartridge being used.

2.3 INTENDED USE

The CHDF 2000 is designed to determine the size distribution of particles suspended in an aqueous eluant. The particle diameter range which is covered by the standard C-202 Fractionation Cartridge and the GR-500 eluant is 15 nm to 1100 nm. The fractionation resolution is low above the upper size limit. Larger particles, up to 3 microns approximately, will not harm the fractionation capillaries. Two other fractionation cartridges are available: high resolution (C-201), and high sensitivity (C-203).

The particle size range for the high resolution cartridge is 15 nm to 700 nm. The high resolution cartridge offers resolution power two to three times higher than a standard resolution cartridge but at a lower upper particle size range (700 nm). The C-203 cartridge measures particles in the range 15 nm to 1100 nm and has about half the resolution of the C-202 cartridge. The C-203 provides particle detection sensitivity three to four times that of the C-202 cartridge. High sensitivity may be needed for the analysis of non UV-light absorbing materials such as colloidal silica and other inorganics.

The CHDF 2000 uses the Capillary Hydrodynamic Fractionation (CHDF) technique. This technique is based on the rigorously described and documented behavior of particles influenced by the velocity profile of a flowing fluid. Due to the influence of the fluid velocity profile, larger particles will have a greater average velocity than smaller ones. The particle size distribution is determined by measuring the transit time of the particles through a series of capillary tubes.

The CHDF 2000 can be used to analyze a wide variety of colloidal suspensions. The Fractionation Cartridge and the GR-500 eluant are for use with either anionic or sterically stabilized dispersions.

Typical applications include:

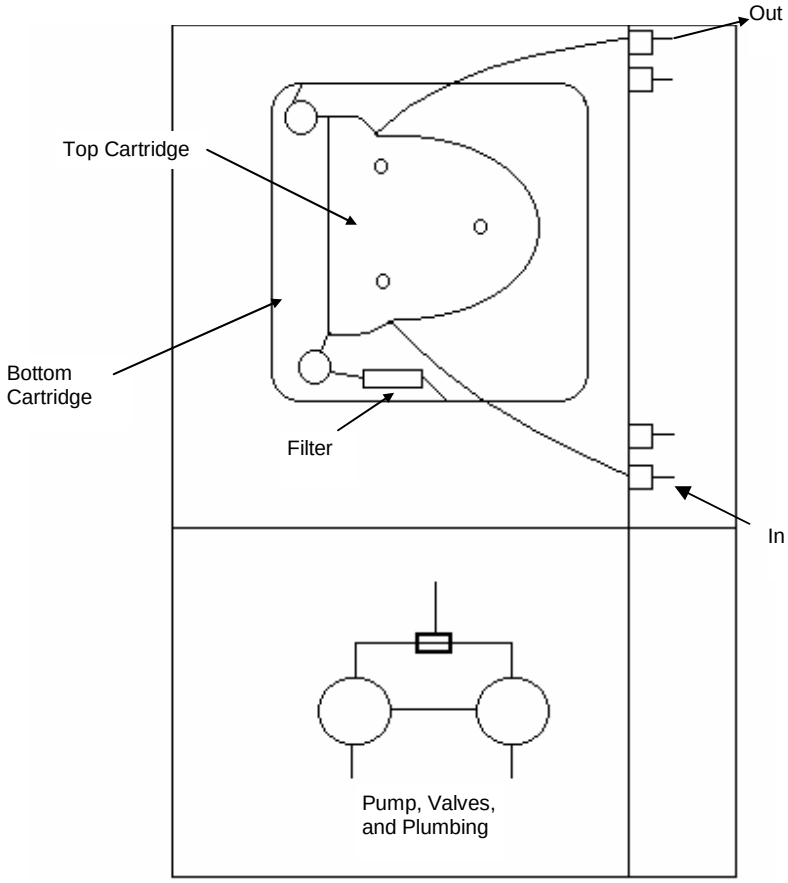
1. Latex
2. Organic pigments
3. Carbon black
4. Emulsions
5. Liposomes
6. Inorganic Particles

For more detailed information regarding different applications, please contact Matec Applied Sciences directly.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.4 DESCRIPTION AND SPECIFICATIONS

The CHDF 2000 achieves the separation of particles using a several different fractionation cartridges. These are illustrated below in Figure 2.1.



After the sample is injected, the flowing eluant carries a small portion of it into the top cartridge. The particle size separation takes place in the top cartridge. This cartridge contains a coil of glass capillary tubing as seen in Figure 2.2. This is the cartridge that needs to be replaced on occasion. The back cartridge contains two glass capillary coils which rarely need replacement. A large proportion of the eluant and sample flow through the back cartridge, and then to waste. Only a small portion of any injected sample is needed to measure a fractogram.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

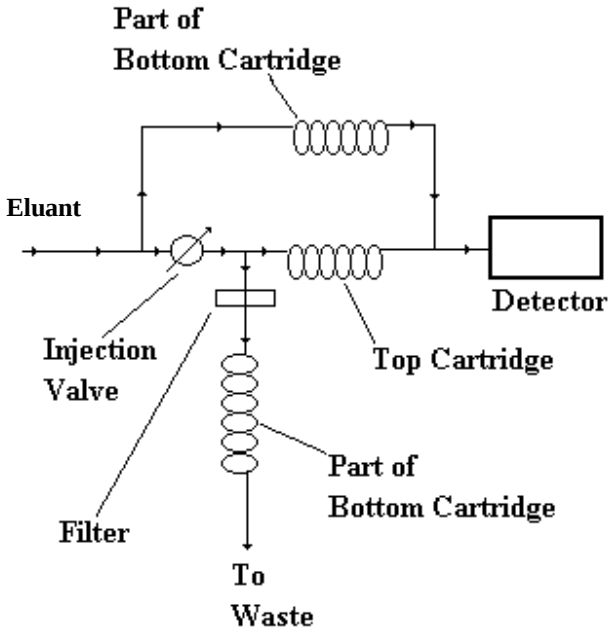


Figure 2.2 The inner workings of the top and bottom fractionation cartridges.

2.4.1 Computer Specifications

To run the CHDF Version 2.0 software you need an IBM or compatible PC equipped as follows:

1. Running Windows™ 95.
2. A Pentium™ processor.
3. 8 megabytes or more of random access memory (RAM).
4. 240 megabyte or larger hard disk drive.

If your computer does not meet these specifications do not install the software. Upgrade your computer, or find a computer that meets or exceeds the specifications given above.

2.4.2 CHDF 2000 UV Detector Specifications

1. Spectral bandwidth:	7 nm
2. Wavelength accuracy:	±2.0 nm
3. Wavelength repeatability:	±0.25 nm
4. Light source:	35 watt deuterium lamp
5. Wavelength Range	190-380 nm

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2.4.3 CHDF 2000 Pump Specifications

1.	Maximum pressure:	6000 psi
2.	Flow rate:	0.01 - 9.99 ml/min
3.	Cumulative Drift:	±1% of indicated flow rate or 10 ul/min per 10 hours with 100 psi minimum pressure
4.	Flow accuracy:	±2% @ pressures > 500 psi
5.	Flow stability:	±0.1% of indicated flow rate

2.4.4 CHDF 2000 OPERATING SPECIFICATIONS

The operating specifications of the CHDF 2000 are:

1. Particle size measurement range:

The size range of the standard C-202 Cartridge and the 2XGR-500 eluant is 15 nm to 1100 nm particle diameter. A 15 nm to 700 nm high-resolution range is available (C-201). A high-sensitivity cartridge for measurement of non-UV-absorbing materials is available (C-203).

2. Size Resolution

C-202 Standard Resolution Cartridge: Particle ratio = 1:1.2; High Resolution C-201 cartridge: Particle ratio = 1:1.1 minimum; C-203 cartridge: 1:1.3, e.g.: narrow particle size distribution particles with greater than 20% (standard resolution) or 10% (high resolution) size difference will be resolved.

3. Measurements Provided:

a) Particle size distributions by number, area, and weight
b) Cumulative particle size distribution by number, area, and weight
c) Number average particle size
d) Area average particle size
e) Weight average particle size
f) Volume average particle size
g) Absolute detector output
h) Particle size distribution standard deviation
i) Particle size distribution half height width
j) 25% percentile
k) 75% percentile

4. Measurement Time:

Twelve minutes maximum from sample injection to graphics output.

5. Data Presentation:

User selectable combination of:
a) Raw data

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

b) Tabular data of relative and cumulative size distributions
c) Graphics of relative and cumulative size distributions
d) Hard copy or terminal display only

6. Sample Size:

One milliliter at 0.1-5 volume %, pre-filtered through a 5 micron or larger pore size filter. Filters are available (See Section 6, Replaceable Parts and Consumables). The actual sample concentration to be used is a function of the detectability of the sample. All samples must be aqueous based.

7. Operating Temperature:

20 to 40 degrees Celsius. Fractionation column temperatures can be set to a maximum of 99°C. A column temperature of 35°C is recommended.

8. Sample Eluant

The CHDF GR-500 eluant consists of proprietary surface active agents in deionized water supplied by Matec Applied Sciences in concentrated form. Three types of GR-500 eluant are available: 1X-GR500, 2X-GR500, and 2X-GR500-LA. The 2X-GR500 is the standard eluant used for regular submicron particle size analysis. The 2X-GR500 normal conductivity must be in the range 22-27 µSiemens/cm. The GR500-LA conductivity must be in the range 14-18 µSiemens/cm. The 1X-GR500 conductivity should be between 6 and 10 µSiemens/cm. Contamination of the eluant, especially by electrolytes, will result in lower than expected particle sizes. The same eluant type must be used during calibration and regular operation of the cartridge. Changing the eluant type changes the calibration curve.

To prepare a batch of eluant, combine 1 part GR-500 concentrate with 9 parts water deionized water. The eluant may then be sonicated or filtered under vacuum for degassing purposes.

Lower ionic strength eluants produce stronger electrical double layer repulsion between the particles and the capillary wall. Particles traveling closer to the center of the capillary travel faster down the capillary. A lower ionic strength produces more efficient separations but a lower practical particle size range upper limit. See section 2.5, Theory of Operation.

9. Capillary Hydrodynamic Fractionator:

Integral, removable, disposable cartridge (two are provided per unit, one as spare).

10. Power Requirements:

115 VAC to 120 VAC, 60 Hz, or
220 VAC to 240 VAC, 50 Hz, 2.5A, single phase

11. Physical Dimensions:

Comfortably fits on a standard lab benchtop
a) Size:
15" (37.5 cm) Wide x 28" (71.1 cm) Deep x 15" (37.5 cm) High

b) Weight:
65 lbs. (29.6 kg)

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

12. Calibration:

The CHDF 2000 is factory pre-calibrated. Each fractionation cartridge is provided with a floppy disk. The floppy disk contains calibration curve files. The eluant flow rate and type at which the cartridge must be operated are indicated on the floppy disk label, a typical flow rate is 1.4 ml/min. Periodic field recalibration can be performed by the user directly through the CHDF software.

13. Calibration Material:

The recommended calibration materials are monodisperse polystyrene latexes. These are available from a wide variety of commercial sources or directly from Matec Applied Sciences.

14. Warranty:

Complete one year limited warranty, excluding replaceable CHDF cartridge.

ANY ATTEMPT TO REMOVE OR TAMPER WITH THE INTERNAL PLUMBING OF THE FRACTIONATION CARTRIDGE WILL VOID THE WARRANTY. THE USE OF ELUANT OTHER THAN THOSE SUPPLIED BY MATEC APPLIED SCIENCES ALSO VOIDS THE WARRANTY.

2.5 THEORY OF OPERATION

The behavior of macromolecules and particles in suspensions flowing through capillary tubes has been studied in detail because of its importance in certain biological phenomena, such as the transport of red blood cells through the smaller capillaries in the circulatory system (1), and for use in particle fractionation methodology.

The separation of particles by size due to hydrodynamic effects occurring in the flow of suspensions through capillaries was first examined by Di Marzio and Guttman in 1970 (2,3). A particle exhibiting Brownian motion suspended in a viscous fluid undergoing Poiseuille flow within a capillary tube will sample all accessible radial positions if the particle elution time is sufficiently long. The closest approach of the particle center to the inner wall of the capillary will be limited to a distance equal to the particle radius. As the particle size increases, it will become increasingly unable to sample the slowest moving eluant streamlines near the capillary wall. As a consequence of this, the particle velocity will exceed the average velocity of the eluant and the velocity of smaller particles. This effect is based only on the particle size and is not a function of the particle density or composition. In addition to this exclusion effect, the longitudinal and radial particle displacements are affected by the presence of the capillary wall, and by a fluid inertial force.

The Capillary Hydrodynamic Fractionation CHDF 2000 instrument from Matec Applied Sciences uses these phenomena to determine particle size and size distributions in the sub-micron range. The instrument moves the eluant and the particles through a set of capillaries, measures the particle concentration leaving the system as a function of the transit time, and relates this transit time to that of well characterized monodisperse size standards used in the calibration procedure. Through a mathematical interpretation of the raw data, the true particle size and size distribution is established.

The software uses two particle extinction cross section curves, one for particles that absorb UV light and one for those that do not, in order to compute the sample particle numbers. These curves are plots of particle extinction cross section against particle size. The particle size is determined from the particle elution time. The number of particles is calculated from the following expression:

$$N_i = (D.O.)/R_{ext,i}$$

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

where D.O. is the detector signal for a particle of size i, Rext is the extinction cross section, and N is the number of particles of size i. The computation of the extinction cross section by use of the Mie theory of light scattering is currently being incorporated into the CHDF 2000 software. (D.O.) is multiplied by a fractogram deconvolution parameter if the operator chooses to have the sample fractogram deconvoluted into its primary components (See Software User's Guide).

2.6 DATA ANALYSIS

The CHDF 2000 determines the concentration of particles exiting the fractionation cartridge as a function of transit time from injection. This data is compared to a calibration curve generated by measuring the transit times of monodisperse polystyrene latex size standards. The raw data can be mathematically deconvoluted into component peaks. From the deconvolution and the absolute calibration using the known size standards, the particle size and size distribution are automatically determined. The user may also choose to not have the data deconvolved.

The CHDF 2000 Operating Software automatically calculates and displays the weight average, area average, volume average, and number average particle size. In addition, the area, weight and number relative and cumulative particle size distributions along with the standard deviation, half height width, and the 25% and 75% percentiles are available on hard copy in either tabular or graphical form. The raw data, which is the absolute detector output, is available as well.

It should be noted that both the theoretical foundation for CHDF and the calibration curve are for spherical particles. If your particles are rod shaped or platelets, then the size measured by CHDF will be an approximate spherical diameter. Distributions in size will still be absolutely determined, but no specific information will be given on the aspect ratio of the particles.

2.7 SAMPLE PREPARATION

The CHDF 2000 requires 1 milliliter of sample at 0.1-5 volume %. The sample must be aqueous based. The sample can be prepared in either of two ways; dilution of a concentrate or the addition of the disperse phase to the eluant. It is recommended that the GR-500 be used for dilutions or making dispersions. The concentration of the disperse phase should be high enough to obtain good resolution and a favorable signal to noise ratio. This is particularly important when trying to identify very small sub-populations. On the other hand, it should be kept low enough to avoid particle aggregation or injection valve clogging problems. Do not run samples above 5 volume % or this may clog the injection valve or damage the fractionation cartridge.

The CHDF 2000 can determine the complete particle size distribution over the entire specified size range. Polydisperse samples do not need special pretreatment. The standard size range is 15 nm to 1100 nm particle diameter. Other size ranges will be available in the future.

The actual state of dispersion and the stability of the sample are important considerations for any particle characterization technique. The CHDF 2000 measures the effective hydrodynamic particle size. An aggregate will behave as a larger particle and will not be resolved into the smaller primary particle size. For some samples this may cause discrepancies between CHDF and transmission electron microscopy (TEM).

The standard CHDF 2000 GR-500 eluant is designed for anionic dispersions. When diluting a concentrate, use the GR-500 fluid as the dilutant. This will help ensure a stable, well dispersed system. Most systems will be anionic when dispersed in GR-500. For specific systems, this can be checked using

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

microelectrophoresis or electro-acoustic ESA analysis. Problems may occur when diluting systems stabilized with cationic surfactants. For specific information regarding specific sample systems, please contact Matec Applied Sciences directly.

Most samples will need to be sonicated before analysis. For many samples prepared by the dilution of a concentrate, placing the sample container in an ultrasonic bath will be sufficient. For dispersing powders into liquids or when dealing with materials which may have a strong tendency to form aggregates, it will be necessary to use an ultrasonic probe immersed in the sample itself. In both cases, the objective is to prepare a uniform dispersion in which the particles exist in the actual state of dispersion that you wish to characterize.

The CHDF 2000 requires that the sample be injected by syringe through an injection valve, or the sample may be injected using an autosampler. In many cases it is necessary to filter the sample before injection to remove coagulum or outsize particles. Five-micron filters which attach directly to the end of the syringe are recommended. These filters are available from Matec Applied Sciences.

2.8 ELUANT

The standard eluant supplied for use with the CHDF 2000 is designated GR-500 and is for anionic systems. Most materials will be anionic when dispersed in GR-500. The eluant consists of surface active agents in de-ionized water. GR-500 is supplied in the form of a concentrate which must be diluted 10:1 for use, e.g., 900 ml de-ionized water to 100 ml GR500 concentrate. GR-500 should be diluted with de-ionized water and the final solution should be filtered under vacuum through either a 0.1 or 0.2 µm pore size filter. The purpose of this filtration under vacuum is to remove any particulate matter and also to de-gas the GR-500. Care should always be taken when handling the GR-500 to avoid contamination. The eluant should be used within 3 days of preparation. The GR-500 concentrate should be used within one month of receipt. It should be kept refrigerated.

Matec supplies three types of GR-500 eluant: 1X-GR500, 2X-GR500, and 2X-GR500-LA. The appropriate conductivity ranges (µSiemens/cm) of final eluant solutions are 6-9 for 1X-GR500, 14-19 for 2X-GR500-LA, and 22-27 for 2X-GR500. Each of these eluants can be used with any of the three types of cartridges available. Most commonly, 2X-GR500 is used in conjunction with a C-202 cartridge.

Both 2X- eluants produce the same resolution and use the same calibration curves. The 2X-GR500 contains 10 milligrams/l sodium azide biocide; the 2X-GR500-LA eluant contains only 0.5 milligram/l sodium azide. As a result, the 2X-GR500-LA eluant degrades faster due to bacterial growth. The shelf-life for these eluants is three days for the 2X-GR500-LA, and one week for the 2X-GR500. The 2X-GR500 cannot be used at 200 nm UV-detection because of the high UV-absorption by sodium azide.

The 1X-GR500 provides higher particle resolution efficiency than the 2X- formulas. The upper particle size limit is lower with 1X-GR500. The 1X-GR500 contains 0.5 milligrams/l sodium azide. Its shelf life is three days.

NOTE:

The CHDF 2000 and the fractionation cartridge have been specifically calibrated for use with GR-500 eluant. The use of any other eluant will give erroneous results and voids the CHDF 2000 warranty.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Chapter 3

Setting Up the CHDF 2000

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

3 Setting Up the CHDF 2000

This chapter describes how to set up the CHDF 2000 for normal operation.

Before you can operate the CHDF 2000, you need to:

- Perform power-on procedures
- Review and edit operating parameters

3.1 Performing Power-On Procedures

Caution: *To prevent injury, apply power to the CHDF 2000 only after you have completed all installation tasks properly.*

When you first power-on the CHDF 2000, perform the following procedures:

- Check power-on diagnostics
- Check the Control panel display

3.1.1 Powering On the System

To apply power to the system:

1. Insert the power cord into the power outlet of an appropriate power source.
2. Move the power switch to the **1** (On) position (Figure 3-1).

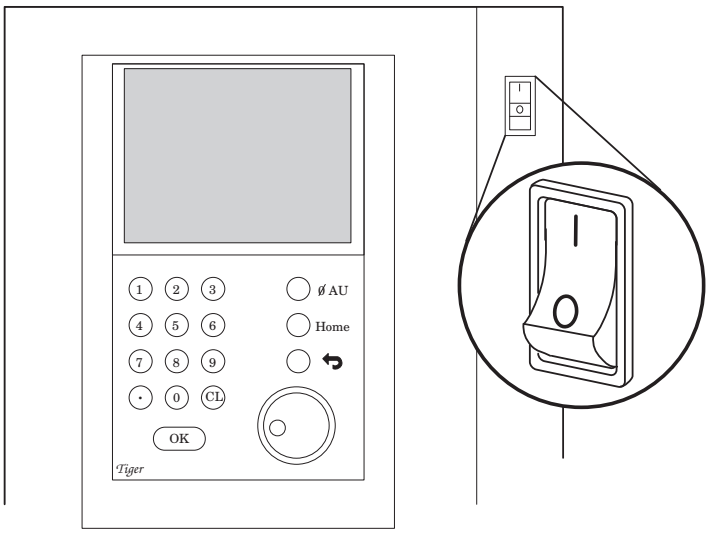


Figure 3-1 Power-On/Off Switch

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

3.1.2 Checking Power-On Diagnostics

When you power-on the CHDF 2000, the system automatically executes a set of diagnostics to verify that the system is ready for operation. The results of the power-on diagnostics appear in the Power-On Diagnostics screen (Figure 3-2).

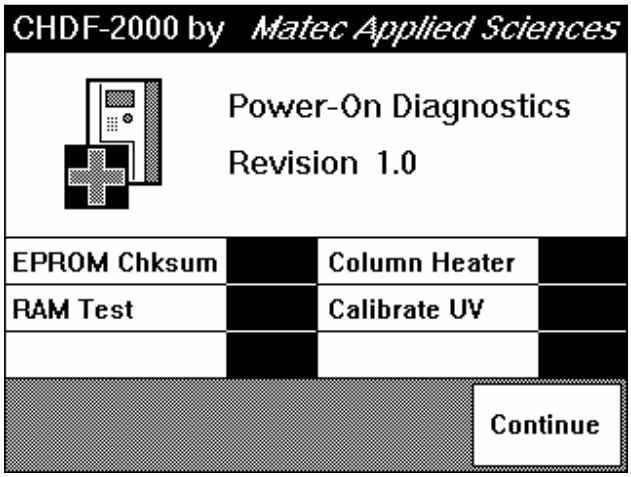


Figure 3-2 Power-On Diagnostics Screen

Table 3-1 identifies and describes the functions of the four power-on diagnostic tests.

Table 3-1 Power-On Diagnostic Tests

Name	Function
EPROM Checksum	Performs a checksum test of program memory (erasable programmable read-only memory).
RAM Test	Tests the validity of programmed method data saved in RAM (random access memory).
Cartridge Heater	Determines if an optional cartridge heater is installed.
Calibrate UV	Performs calibration and check of the absorbance detector optics. This test takes 2 to 3 minutes to complete.

If your CHDF 2000 fails any of the power-on diagnostics, refer to Chapter 5, Diagnostics, for troubleshooting information.

3.1.3 Checking the Control Panel Display

When you first power-on the CHDF 2000, check the control panel display for readability. If necessary, adjust the brightness and contrast of the control panel display, as described in Section 6.5, Adjusting the Control Panel Display.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

3.1.4 Moving to the Home Screen

You can initiate all CHDF 2000 control functions from the Home screen. From the Power-On Diagnostics screen (see Figure 3-2), press the selector knob to choose **Continue.** The Home screen appears (Figure 3-3).

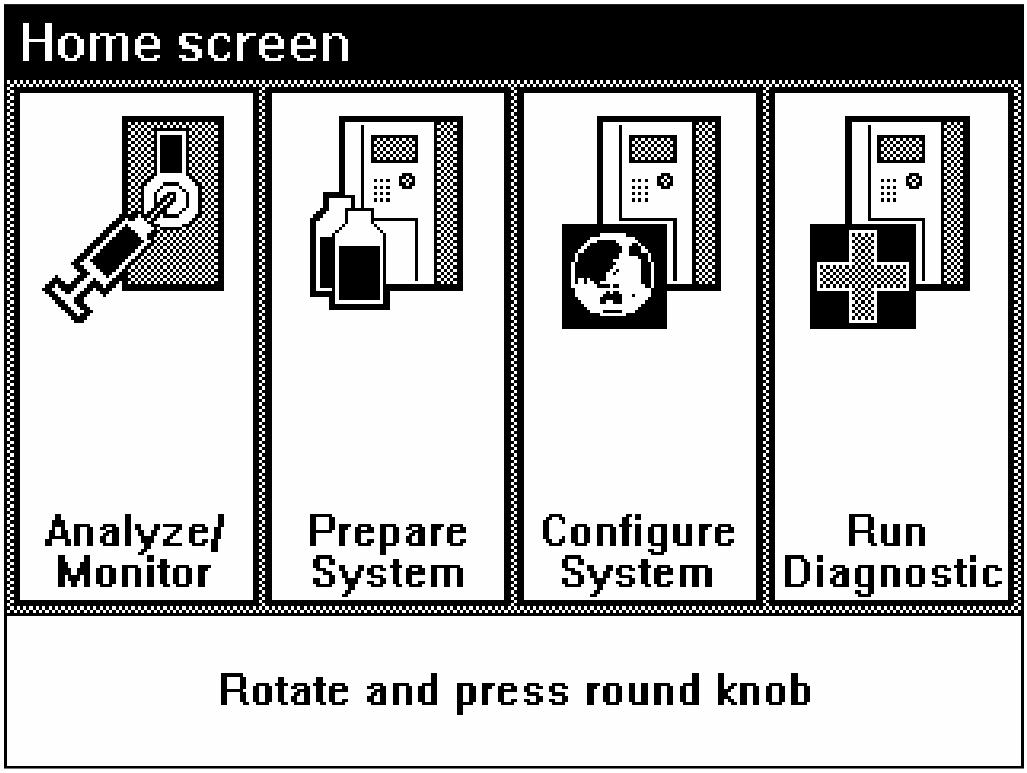


Figure 3-3 The Home Screen

You are now ready to set up the CHDF 2000 for normal operation.

Preparing the System for Use

From the Home screen (see Figure 3-3), choose **Prepare system**. The Prepare System screen appears as shown below.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

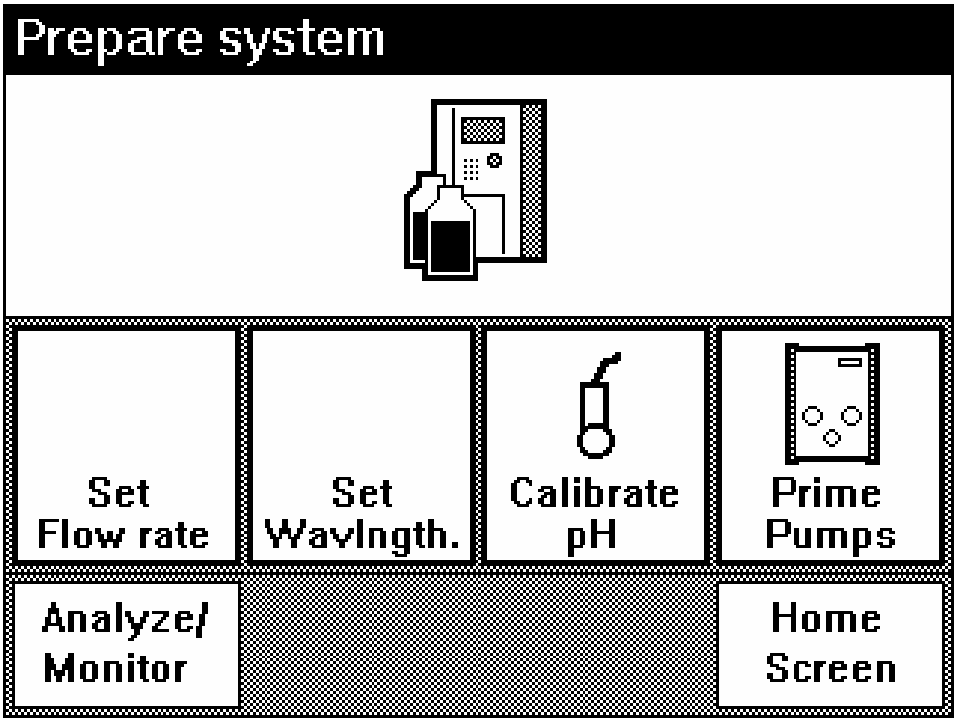


Figure 3.4 The Prepare System Screen

There are four choices in this screen used to prepare the system, **Set Flow rate**, **Set Wavlngth.**, **Calibrate pH**, **and Prime Pumps**. Rotating the selector knob on the instrument alternately highlights the different choices on the screen, and pressing the selector knob performs the highlighted action. When any of these four options are selected, a **Configure System** screen appears as shown in Figure 3.5.

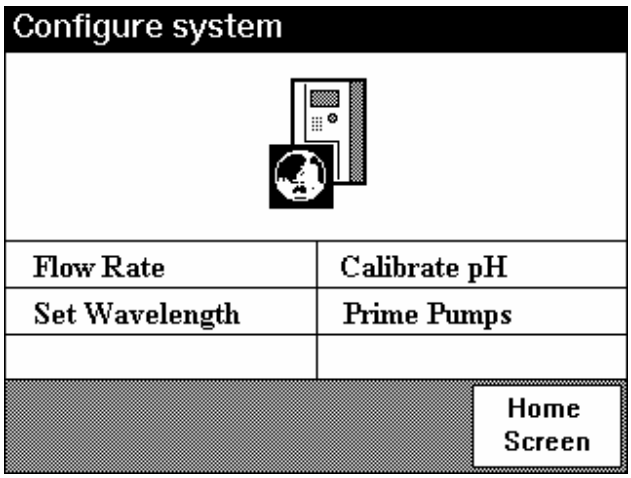


Figure 3.5 The Configure System Screen

Although there are 4 choices in this menu, only one of them can be accessed at a time. For example, if you selected **Flow Rate** from the menu in Figure 3.4, when the menu in Figure 3.5 appears, only the flow rate parameter can be changed. All other choices are disabled. Select

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Home Screen to get back to the top menu. Here is how to set the four parameters listed in the Configure System screen seen in Figure 3.5.

Flow Rate - This sets the rate at which eluant is pumped through the fractionation cartridge. To change the flow rate, simply select it, then rotate the selector knob left or right until the flow rate you desire appears on the screen. Push the selector knob to enter the value. Normally, a value of 1.4 should be selected.

Set Wavelength - This command sets the wavelength at which the UV/VIS detector will work. The wavelength is in units of nanometers. To change the wavelength, select **Set Wavelength**, rotate the selector knob left and right until the desired wavelength appears. Push the selector knob to enter the value. The recommended value is 220 nm.

Calibrate pH - Select this option when you need to calibrate the pH sensor on the CHDF 2000. You will need three buffer solutions, typically of pH 4.0, 7.0, and 10.0 to calibrate the pH sensor. The sensor is calibrated by alternately immersing the sensor in the three buffers, then entering the pH value of the buffer into the instrument. This is accomplished by first selecting **Calibrate pH** from the Configure System menu. This action brings up a new screen as shown below in Figure 3.6.

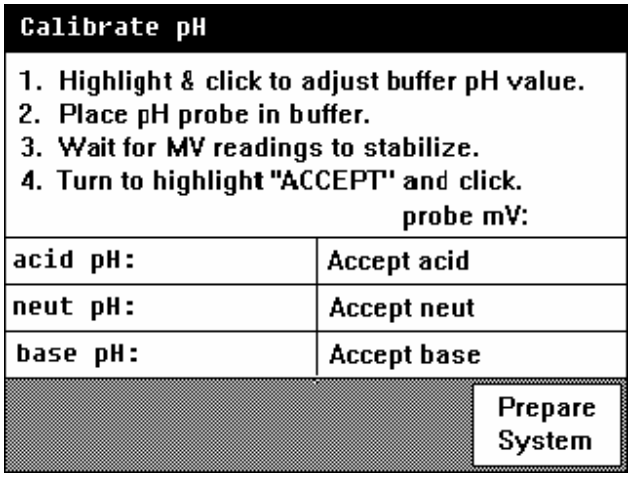


Figure 3.6 The Calibrate pH screen

As stated in the screen, first highlight the pH that is going to be set by turning the selector knob. Click the knob, then turn it to select the pH of the buffer to be used. Place the pH sensor in the buffer, wait for the Prove mV reading to stabilize, then turn the selector knob to the right to highlight either **Accept Acid**, **Accept neut**, or **Accept base**. Press the selector knob to enter the pH value. Three buffers of different pH must be used to calibrate the sensor.

Prime Pumps - The pumps are primed by drawing eluant into the pump heads to get rid of air bubbles. The process is described in a screen that can be accessed by selecting Prime Pumps. The following screen appears.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

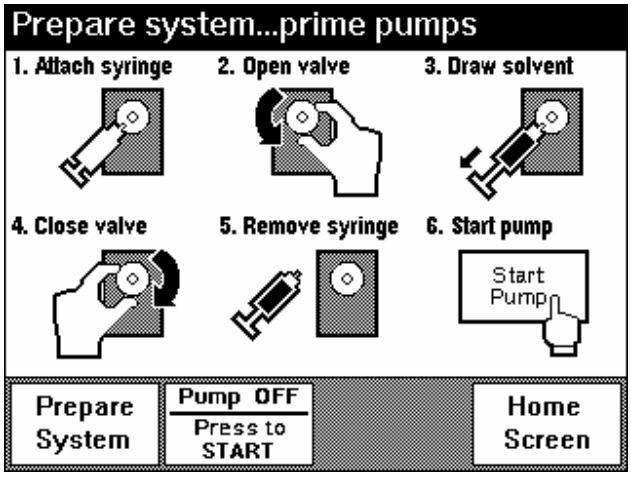


Figure 3.7 Instructions on how to prime pumps.

Note: The order of the instructions shown in this screen is not correct. The correct sequence of steps to follow are: 1, 2, 3, 6, 5, 4. The process will be explained here in words to avoid confusion. The syringe attaches to a special valve in the lower right front of the instrument called the priming valve. Attach the syringe to the priming valve, then open the valve. Draw several mls of eluant into the syringe. Start the pump by selecting **Press to START**, remove the syringe from the priming valve; wait until the pump stops, then close the valve. Please realize that this screen only gives instructions, accessing this screen by itself will not prime the pumps.

3.2 Reviewing and Editing Operating Parameters

The CHDF 2000 is configured with default values for 14 operating parameters that control specific system functions. Review all operating parameters and edit them if required to customize instrument operation for your needs.

3.2.1 Changing Operating Parameters

To review and edit the system parameter values of the CHDF 2000 choose **Configure system** from the Home Screen. The first of three Configure System screens appears (Figure 3.8).

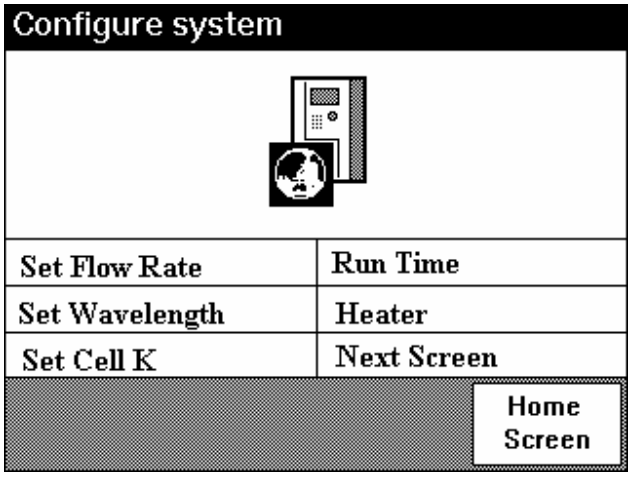


Figure 3-8 First Configure System Screen

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

When each of these commands is selected, the display changes so that a number may be entered. The parameters are described and set as follows:

Set Flow Rate - Sets the flow rate at which eluant is pumped through the fractionation cartridge in units of ml/min. When selected, the display changes to "Rate = ". Turn the selector knob to the desired value, then push the knob to enter the value. A typical flow rate is 1.4 ml/min. This is the same parameter as was set in the Configure System screen seen in Figure 3.5.

Set Wavelength - This command sets the wavelength at which the UV/VIS detector will work. The wavelength is in units of nanometers. To change the wavelength, select **Set Wavelength**. The display changes to "Run Wavelength = ". Rotate the selector knob left and right until the desired wavelength appears. Push the selector knob to enter the value. A typical wavelength is 220 nm.

Set Cell K - This parameter is used to calibrate the conductivity sensor that is onboard the CHDF 2000. When this command is selected, the display changes to "Cell K:". Rotate the selector knob to the correct value, then push the knob to enter the value. The selected value should be in the range 0.8 to 2.0.

Run Time - This number sets the length of the X axis on the display when data is viewed during a CHDF run. The default value is 15 minutes. When this command is selected the display changes to "Run Time:". Rotate the selector knob to the desired value, then push the knob to enter the value.

Heater - This parameter is used to set the temperature of the fractionation cartridge. The default is 35°C. When this parameter is selected the display changes to "Cartridge Temp: ". Rotate the selector knob to the desired value, then push the knob to enter the value.

NOTE: Changing the cartridge temperature requires the measurement of a new calibration curve.

Next Screen - Displays the second Configure System screen, seen in Figure 3.9 below.

The second Configure System screen is displayed by selecting **Next Screen** in the first Configure system screen. The second Configure System screen is shown below in Figure 3.9.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

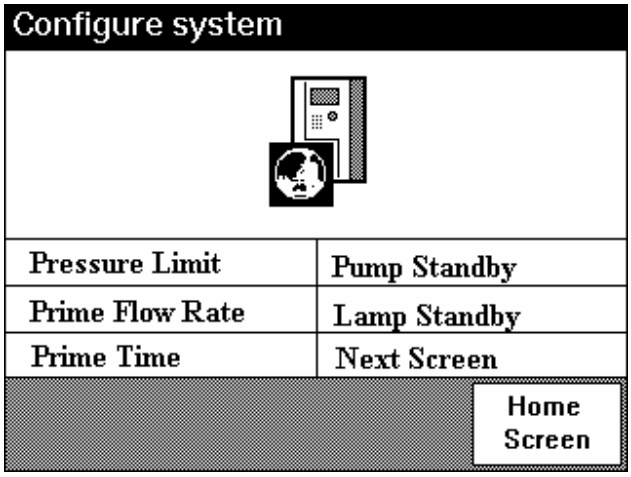


Figure 3.9 The second Configure System screen.

When each of these commands is selected, the display changes so that a number may be entered. The parameters are described and set as follows:

Pressure Limit - Used to set the pressure values above or below which the pump will shut off. Pressure is measured in units of PSI (pounds per square inch). When clicked once, the display will change to "Low Limit". Rotate the selector knob to the desired value (1000 is recommended), then push the knob to enter the value. The display will then change to "High Limit" (6000 is recommended). Rotate the selector knob to the desired value, then push the knob to enter the value.

Prime Flow Rate - Sets the flow rate to be used during priming in ml/min. When this parameter is selected, the display changes to "Rate ml/min:". Rotate the selector knob to the desired value, then push the knob to enter the value. A typical value is 8.0 ml/min.

Prime Time - Sets the amount of time the pump runs during priming (2 min. normally). Time is measured in units of minutes. When this parameter is selected the display changes to "Prime Time:". Rotate the selector knob to the desired value, then push the knob to enter the value.

Pump Standby - Sets how long the pump will run before it automatically shuts off. Time is measured in units of minutes, and a setting of "0" will cause the pump to run indefinitely. The parameter is best used in conjunction with an autosampler. For example, it can be used to shut down the pump after the instrument has concluded an overnight run. However, the Pump Standby time can expire before all samples are run, so be careful when setting this parameter. For most purposes, a value of 0 should be used and the pump can be turned off manually (or the pump can be turned off using the autosampler sequence setup options parameters in the software). When this parameter is selected, the display changes to "Standby:". Rotate the selector knob to the desired value, then push the knob to enter the value.

Lamp Standby - Is used to determine how long the lamp in the UV/VIS detector will stay on before it is automatically shut off. The purpose of shutting off the lamp is to hopefully extend its life. However, if the lamp is shut off before all your samples are run, you will not obtain any data. A value of "0" will leave the lamp on indefinitely. The lamp standby value must be equal to the pump standby value. The instrument will not allow you to set the two parameters to different values. Once this parameter is selected, the display changes to "Standby:". Rotate the selector knob to the desired value, then push the knob to enter the value.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Next Screen - Selecting Next Screen brings up the third of the three configure system screens as seen below in Figure 3.10

The third Configure System screen is displayed by selecting **Next Screen** in the second Configure system screen. The third Configure System screen is shown below in Figure 3.10.

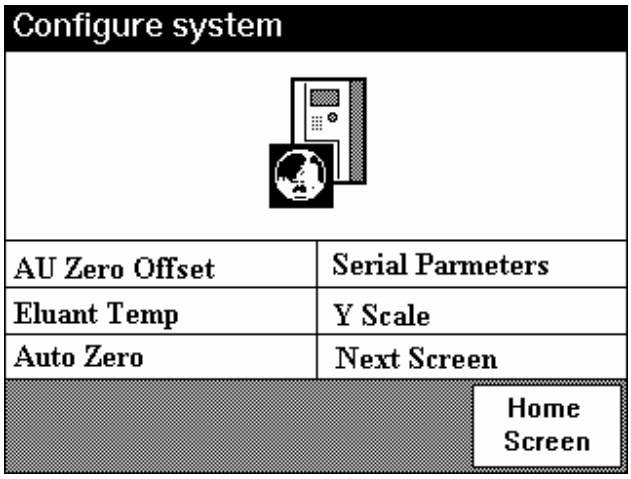


Figure 3.10 The third configure system screen.

When each of these commands is selected, the display changes so that a number may be entered. The parameters are described and set as follows:

AU Zero Offset - Tells the instrument how many absorbance units of signal being measured by the detector should be set equal to zero. Normally, this parameter should be left at zero. Once this parameter is selected, the display changes to "mAU = :". Rotate the selector knob to the desired value, then push the knob to enter the value.

Eluant Temp - This parameter is used to calibrate the temperature probe on the CHDF 2000. After selecting this parameter, rotate the selector knob until the current value of the eluant temperature appears. Then push the knob to save the changes.

Auto Zero - Turns the autozeroing ability of the instrument on or off. When on, an autozero of the detector baseline takes place each time an injection occurs. The autozero sets the detector baseline to the mAU value defined in AU Zero Offset. Once selected, this parameter is turned on or off by rotating the selector knob. Push the knob to record the change.

Serial Parameters - Sets the communication parameters that allow the CHDF 2000 to talk to a personal computer. Once selected, display changes to "Baud Rate". Rotate the selector knob the appropriate value (normally 19200), then push the knob. This value must be the same as that set in the software under system/configure system. Next, the display changes to "Echo" and is followed by an "Off" or "On" (normally off). Rotate the selector knob to the desired choice, then push the knob to save the settings.

Y Scale - Sets the maximum absorbance reading that can be displayed on the CHDF 2000 front panel raw data graph during a fractionation. When selected, the display changes to "Y Axis AU

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Max" and number appears next to this. Rotate the selector knob to the desired value, then push the knob to save the settings. A value of 5 is recommended.

Next Screen - In this instance, selecting Next screen takes you back to the first Configure System screen.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Chapter 4

Operating the CHDF 2000

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

4 Operating the CHDF 2000

This chapter describes how to perform normal CHDF 2000 operations.

4.1 Priming the Pump

Prime the pump to remove air bubbles from the eluant supply tubes or to purge eluant from the system before you use new eluant.

To prime a pump

1. From the Home screen, choose **Prepare system**. The Prepare System screen appears as shown in Figure 4.1.

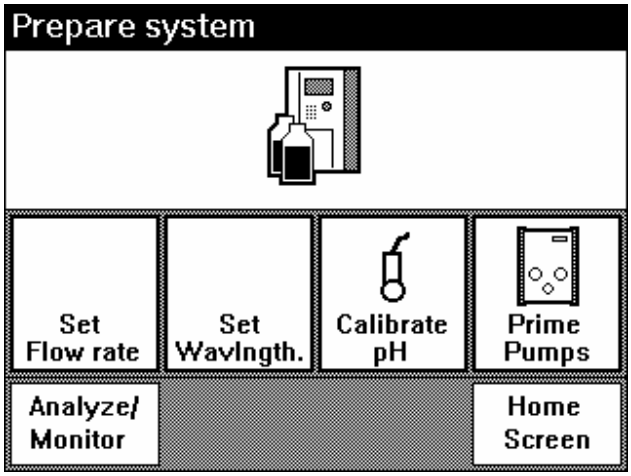


Figure 4.1 The Prepare System Screen

2. Choose **Prime pump**. The Prepare System...Prime Pump screen appears (Figure 4.2).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

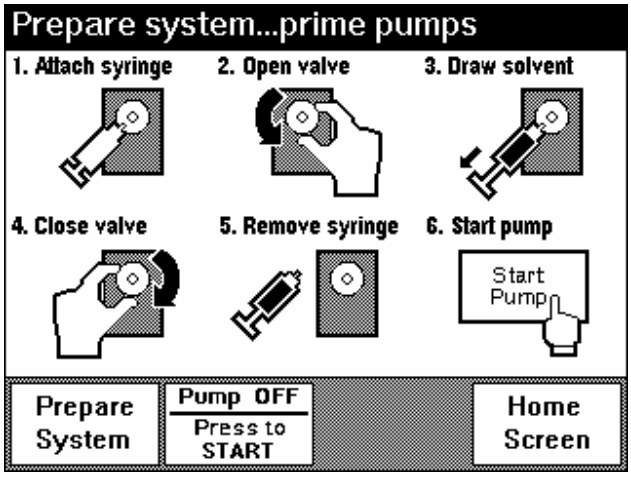


Figure 4.2 The Prepare System...Prime Pump Screen

Note: The order of the instructions on the screen shown above is incorrect. The correct sequence of steps is: 1, 2, 3, 6, 5, 4. These are described in detail below.

3. **Attach syringe** (screen step 1) – Attach the syringe to the priming valve.

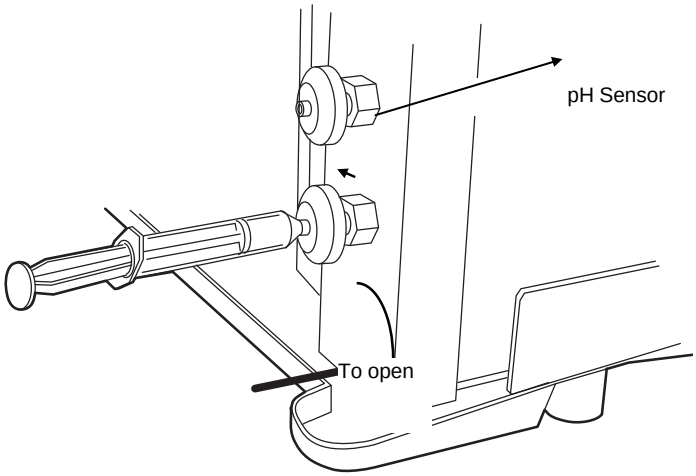


Figure 4.3 Inserting Syringe in Pump Priming Valve

4. **Open valve** (screen step 2) – With the syringe attached to the priming valve, open the valve by turning the top of the valve to the left (see Figure 4.3).
5. **Draw eluant** (screen step 3) – Carefully draw eluant into the syringe until:
   - Air bubbles disappear from the eluant inlet tubing to the pump.
   - The syringe draws steady fluid from the valve.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6. **Start pump** (screen step 6) – Choose **Press to Start**. The Pump draws eluant from the reservoir and pump it through the system.
7. **Monitor** the CHDF 2000 waste tubing until air bubbles disappear from the eluant, or the pump stops.
8. **Close valve** (screen step 4) – With the syringe still attached to the priming valve, close the valve by turning the top of the valve to the right.
9. **Remove syringe** (screen step 5) – Carefully remove the syringe from the pump priming valve.
10. Choose **Pump ON-Press to Stop** to shut off the pump. Or, the pump shuts off automatically according to the time set for the Priming Pump Time system parameter. (Refer to Chapter 3 for more information).

4.2 Analyzing Samples

This section includes procedures for the following tasks:

- Performing standard sample analysis
- Using standby mode when you are not analyzing a sample

4.2.1 Performing Standard Manual Sample Analysis

Sample can be injected manually, as described here, or using an autosampler. To use an autosampler, refer to the CHDF 2000 software manual.

To perform standard manual sample analysis:

1. From the Home screen, choose **Analyze/Monitor**, or from the Prepare System screen, choose **Analyze/Monitor**.

Note: Be sure there is an ample amount of fresh GR-500 eluant in the eluant reservoir.

The Analyze/Monitor Samples screen appears (Figure 4.4).

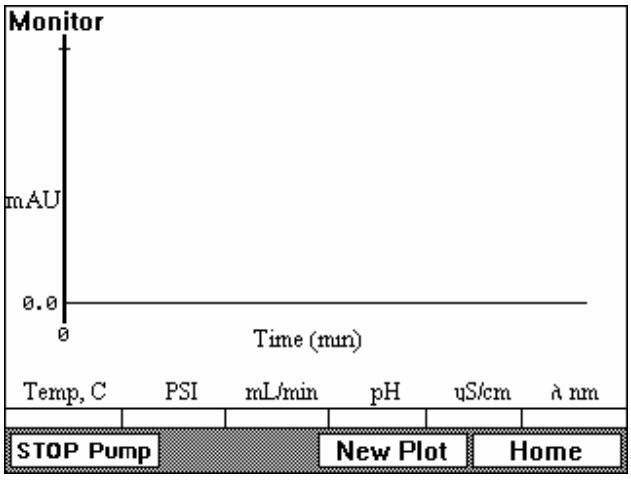


Figure 4.4 Analyze/Monitor samples Screen

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

2. Wait for the pump pressure to be stable (about 1/2 hour). Start the CHDF Software. Follow the software manual instructions for data collection and particle size distribution results.
3. With the manual injector valve in the Load position, insert the prepared sample syringe in the manual injector valve (Figure 4.5).

Note: *The loop volume of the instrument is 20 µL. Load a minimum of 80 µL of sample to ensure accurate results.*

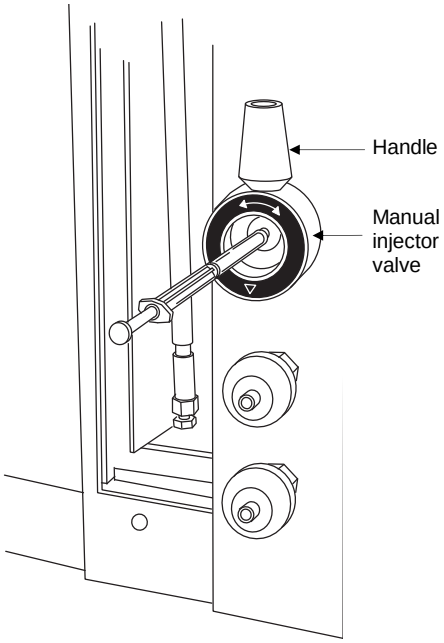


Figure 4.5 Loading Sample Into the Manual Injector Valve

Note: *The manual injector valve must be in the Load position when you load the sample.*

4. Load the sample into the manual injector valve. Do not remove the syringe until you have completed step 4.
5. Rotate the handle of the manual injector valve to the Inject position to inject the sample and start the selected method. The Analyze/Monitor Samples screen appears (Figure 4.5). Remove the sample syringe.
6. Rinse the injection valve with GR-500 eluant. Repeat the above steps to inject the marker solution. The recommended marker injection time is 60 seconds.
7. During the fractionation, the results will be displayed on the screen shown in Figure 4.4. Also, relevant measurements such as the eluant temperature, pressure, flow rate, eluant pH, eluant conductivity, and detection wavelength are displayed. Repeat the above steps for new samples. Select **Stop Pump** or **Home Screen** to end your session.

8. Review your results. If they are satisfactory, you are ready to analyze the next sample. If the results are not satisfactory, refer to Chapter 5, Troubleshooting the CHDF 2000, to locate the source of the problem.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

4.2.2 Using Standby Mode

When you finish analyzing samples, but do not want to alter the current operating conditions, use Standby mode to conserve eluant and lamp energy. The instrument will also go into standby mode if the computer and instrument have ceased talking to each other for > 60 seconds. This may happen, for example, if the computer crashes while the instrument is turned on.

Using Automatic Standby Mode

You can configure the system for two types of automatic standby mode:

- Automatic pump standby mode
- Automatic lamp standby mode

When Standby Mode is entered the following screen appears (Figure 4.6).

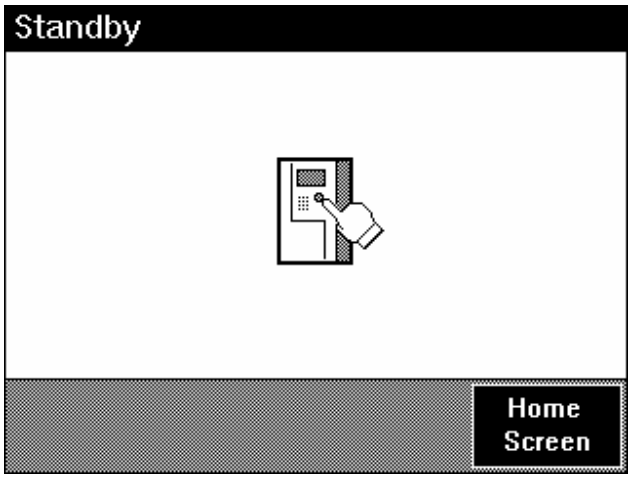


Figure 4.6 The Standby Display

Using automatic pump standby mode

In automatic pump standby mode, the pump or pump shut off after a user-specified time interval at the end of a run.

Note: *The pump standby time must be either equal to or shorter than the lamp standby time.*

You set the time interval by defining the value of the Pump Standby Delay operating parameter. Refer to Chapter 3, Reviewing and Editing System Parameters, for more information.

Using automatic lamp standby mode

In automatic lamp standby mode, the detector lamp shuts off after a user-specified time interval at the end of a method run.

Note: *The lamp standby time must be equal to or longer than the pump standby time.*

You set the time interval by defining the value of the Lamp Standby Delay operating parameter. Refer to Chapter 3, Reviewing and Editing System Parameters, for more information.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Exiting from Standby Mode

To exit from Standby mode, choose **Home screen**. The lamp turns on, the detector recalibrates, and the Home screen reappears.

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Chapter 5

Troubleshooting the CHDF 2000

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

5 Troubleshooting the CHDF 2000

This chapter provides procedures that explain how to:

- Respond to CHDF 2000 error messages
- Use troubleshooting checklists to quickly identify potential sources of system problems
- Run CHDF 2000 diagnostics

5.1 Responding to Error Messages

During operation of the CHDF 2000, conditions may occur that cause error messages to appear on the control panel display. The error messages are:

- High pressure limit exceeded
- Low pressure limit exceeded
- Power lost
- Detector calibration failure

5.1.1 High Pressure Limit Exceeded

The High Pressure Limit error message (Figure 5-1) appears when the system pressure exceeds the high-pressure limit for at least two seconds. You configure the high-pressure limit using the Pressure Limit Set operating parameter (refer to Chapter 3, Reviewing and Editing Operating Parameters).



Figure 5-1 High Pressure Limit Exceeded Error Message

Recommended response

High pressure sometimes occurs if you are priming the pump with a cartridge installed. To prevent high pressure during pump priming, follow the procedures specified in Section 4.1, Priming the Pump.

This message usually results from a blockage in the eluant flow path. Check the eluant flow path and pump. Clear any blockages, if necessary. The most common cause of a blockage is a plug or sample somewhere in the instruments tubing. There are several ways of clearing such a blockage. They will be discussed below in order of increasing difficulty.

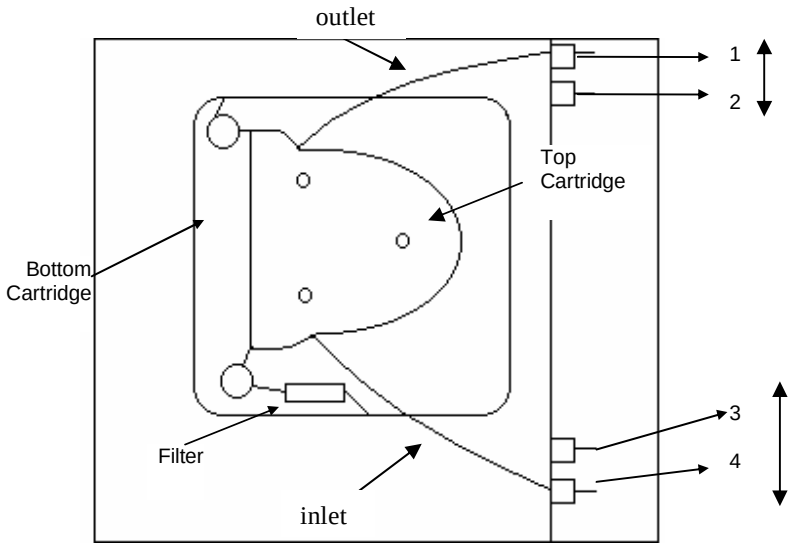
Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Clearing System Blockages

1. If you are analyzing latex or other polymeric based samples, a blockage may be removed by pumping acetone or tetrahydrofuran (THF) through the system for 15 minutes. This will hopefully dissolve the plug.

2. Change (or clean) the in-line particle filter or its element (See section 6.6).

3. You can also try backflushing the fractionation cartridge to remove a plug of material. The idea is to pump eluant through the cartridge in a direction opposite to that which is normal. To backflush the cartridge, open up the front door of the CHDF 2000. There is a panel on the right hand side held in place with two screws. Remove these screws (they should come out by hand), and you should see the following.



The idea is to swap the tubes at fittings 1, and 2, and separately 3 and 4 as follows: Disconnect the stainless steel tube from Fitting 1. Connect the tube on Fitting 2 to Fitting 1; then connect the tube from Fitting 1 to Fitting 2. Disconnect Fitting 3. Connect the tube on Fitting 4 to Fitting 3 and Fitting 3 to Fitting 4. Run the pump for 15 minutes. Reconnect all fittings as they are normally, and try running the pump again. If the high pressure message does not re-appear, your backflushing was successful.

4. Snip off end of the glass capillary tube in the bottom cartridge (see Section 6.6).

5. Replace the entire bottom fractionation cartridge (see Section 6.6).

Do not restart sample analysis until you correct the cause of high system pressure.

Press the selector knob to clear the High Pressure Limit Exceeded error message.

5.1.2 Low Pressure Limit Exceeded

The Low Pressure Limit error message (Figure 5-2) appears when the system pressure falls below the low-pressure limit for more than 110% of the value defined for the Pump Priming Time parameter. You configure the low-pressure limit using the Pressure Limit Set operating parameter (refer to Chapter 3, Reviewing and Editing Operating parameters).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



Figure 5-2 Low Pressure Limit Exceeded Error Message

Recommended response

This message often occurs when there is a leak in the injector, pump, or eluant flow path. Check eluant flow path, injector, and pump and repair any leaks or damage.

Reprime the pump and eluant tubing. Do not restart sample analysis until you correct the cause of low system pressure.

Press the selector knob to clear the Low Pressure Limit Exceeded error message.

5.1.3 Power Lost

The Power Lost error message (Figure 5-3) appears if the CHDF 2000 temporarily loses power during sample analysis.



Figure 5-3 Power Lost Error Message

Recommended response

This message occurs when the CHDF 2000 temporarily loses power due to a power outage or inadvertent powering-off of the system during sample analysis. Before you resume sample analysis:

1. Verify that your operating parameters are correct.
2. Prepare the system by priming the pump, if necessary.

Press the selector knob to clear the Power Lost error message.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

5.1.4 Detector Calibration Failure

The Detector Calibration error message (Figure 5-4) appears if the detector fails to calibrate during system power-on tests.

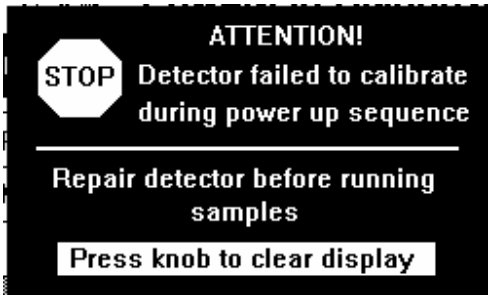


Figure 5-4 Detector Calibration Failure Error Message

Recommended response

Although the system allows you to continue operation when this message appears, the results of the analysis are not reliable. Correct the source of the detector problem before continuing operations. Refer to Section 5.2.5, Absorbance Detector Troubleshooting, for more information.

Press the selector knob to clear the Detector Calibration Failure error message.

5.2 Quick Troubleshooting Checklists

Use the tables in this section to quickly identify corrective actions for common CHDF problems.

This section covers the following topics:

- General troubleshooting
- Fractionation troubleshooting

5.2.1 General Troubleshooting

Use Table 5-1 to quickly identify corrective actions for general system problems.

Table 5-1 General Problems – Quick Troubleshooting Checklist

Symptom	Possible Cause	Corrective Action	Refer to
Unit will not power-on	Power cord not connected	Connect power cord or try another cord.	—
	No power at outlet	Reset circuit breaker or restore line voltage.	Power company
	Power supply fuse blown	Replace fuse.	

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Fan not operating	Fan wiring or motor problem	Repair wiring or replace fan.	Your local service representative
Screen is blank	Control panel display not adjusted correctly	Adjust control panel display.	Section 6.6
	CPU requires reset	Turn instrument power off. Wait five seconds. Turn power on.	—
	Power supply fuse blown	Replace fuse.	
	Control panel ribbon cable disconnected	Reconnect ribbon cable.	
No response from keypad	CPU requires reset	Turn instrument power off. Wait five seconds. Turn power on.	—
	Keypad is defective	Check keypad using Test Keypad diagnostic.	Section 5.3

5.2.2 Fractionation Troubleshooting

Use Table 5-2 to quickly identify corrective actions for fractionation system problems.

Table 5-2 Fractionation Problems – Quick Troubleshooting Checklist

Symptom	**Possible Cause**	**Corrective Action**	**Refer to**
Erratic retention times	Air bubble in pump head	Degas all eluants, re-prime pump.	Section 4.1
	Malfunctioning pump check valves	Clean or replace pump check valves.	Section 6.1.3
	Leak at tube fitting	Check that all tube fittings are tight.	
	Leaking pump seals	Replace pump seals.	Section 6.1.1
Increased sample and marker retention times	Incorrect flow rate	Adjust flow rate.	Section 3.2
	Incorrect eluant composition	Adjust eluant composition.	
	Cartridge partially blocked	Change eluant, backflush cartridge, snip off end of capillary, or replace top cartridge.	Sections 5.1 and 6.6.
	Column heater not connected	Connect column heater.	Instructions supplied with

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Symptom	Possible Cause	Corrective Action	Refer to
			column heater
Doubled retention times	Air bubble in pump head	Prime pump to remove air bubble.	Section 4.1
	Malfunctioning pump check valves	Clean or replace the pump check valves.	Section 6.1.3
	Broken pump plunger	Replace the plunger.	Section 6.1.2
	Defective top fractionation cartridge.	Snip or replace cartridge	Sections 5.1 and 6.6
Reduced retention times	Incorrect flow rate	Adjust flow rate.	Section 3.2
	Incorrect eluant composition	Adjust eluant composition.	
	Low column temperature	Increase column temperature.	
Increased sample retention times, marker retention times correct.	Eluant conductivity too high.	Prepare new eluant, check conductivity.	Section 2.8.
No peaks detected	Plugged capillary	Change eluant, backflush cartridge, snip off end of capillary, or replace top cartridge.	Sections 5.1 and 6.6.
	Pump shutdown	Start pump	Chapter 4
Reduced retention times (cont.)	Incorrect mobile phase	Use correct mobile phase.	—
	Incorrect Cartridge	Replace with correct cartridge.	
Reproduci-bility errors	Eluant not properly degassed	Degas eluant.	—
Baseline drift, rapid			
	Detector not allowed to warm up	Allow detector to warm up until baseline stabilizes. Time varies based on wavelength and sensitivity.	

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

	Eluant contaminated	Use fresh eluant.	—
	Eluant not properly degassed	Degas eluant.	—
	Flow fluctuations	Correct pump malfunctions. Replace pump seals, check valves.	Section 6.1
	Lamp Standby Delay too short	Increase value of Lamp Standby Delay parameter.	Section 3.2
	Incorrect wavelength for eluant	Change eluant or wavelength.	UV absorbance specifications of eluant supplier
Baseline drift, slow	Eluant contaminated	Use fresh eluant.	—
	Decreased UV lamp energy	Check lamp energy.	Section 5.3
	Ambient temperature fluctuations	Prepare the system to proper equilibration.	
	UV detector flow cell leaking	Check flow cell, tighten connections.	Section 6.4

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

5.3 Running CHDF 2000 Diagnostics

This section describes how to run the user-initiated CHDF 2000 diagnostics.

This section describes how to:

- Initiate CHDF 2000 diagnostics
- Interpret CHDF 2000 diagnostics

5.3.1 Accessing CHDF 2000 Diagnostics

To access CHDF 2000 diagnostics from the Home screen, choose **Run Diagnostic**. The Run Diagnostic screen appears (Figure 5-5).

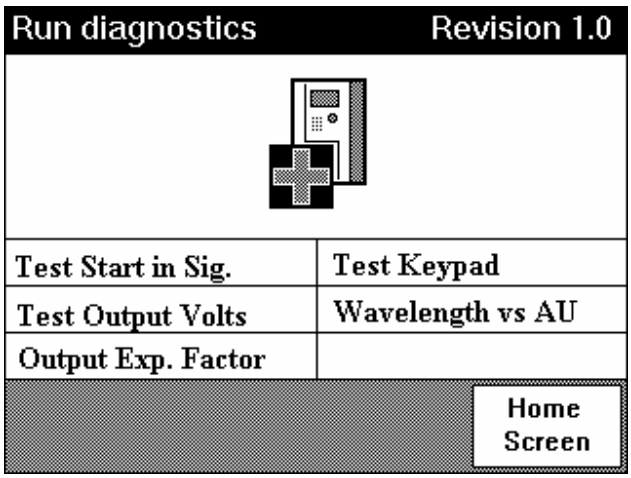


Figure 5-5 Run Diagnostics Screen

When each of these commands is selected, the display changes so that a number may be entered or observed. The parameters are described and set as follows:

Test Start in Sig. - This displays the voltage signal sent by the CHDF 2000 to a computer so the computer knows when samples have been injected and loaded. When selected, the displays changes to “Inject:” and the inject signal is displayed in volts. When the selector knob is pressed a second time the display changes to “Load:” and the load signal is displayed in volts.

Test Output Volts - This voltage is used to run a strip chart recorder or other recording device. It is not important when a computer is used to run the CHDF 2000. When selected, the display changes to “DAC Value =“ and a voltage is displayed.

Output Exp. Factor - Exp. Factor stands for Exponential Factor. This is another parameter used when using a strip chart recorder or other recording device. This parameter is not used when a computer is attached to the CHDF 2000. When selected, the display changes to “Exp Factor =“. Rotate the selector knob to a value between 1 and 100, then push the knob to save the entry.

Test Keypad - This function is used to test whether all of the keys on the CHDF 2000 keypad are functioning properly. Once selected, the display changes to show the series of characters that can

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

be typed in from the keypad. The character most recently pressed is highlighted on the display. If a key is pressed and it does not light up on the display, the key may be malfunctioning.

Wavelength vs AU - This stands for Wavelength vs. Absorbance Units, and is used to observe raw detector signal. When selected the display changes to:
Ref = Smp = L = AU =
Ref is reference, Smp is sample, L is wavelength, and AU is absorbance units. The reference and sample numbers are the raw detector signals measured by the sample and reference channels on the detector. L refers to the wavelength being measured, and AU gives the current absorbance being measured by the detector. These parameters should be monitored over time to see if they change. Changes may indicate that the detector cell windows are becoming dirty, or that the lamp strength is deteriorating.

5.3.2 Initiating and Interpreting Diagnostics

The CHDF 2000 diagnostics are explained in more detail in the following sections

Test START IN Signal Diagnostic

The Test START IN Signal diagnostic provides two functions:

- Tests the START IN signal on the signal connector module
- Tests the manual injector valve

Testing the START IN signal

To test the START IN signal on the signal connector module:

1. Place the manual injector valve in the Load position.
2. Choose **Test START IN Sig**. LOAD should appear in the display.
3. Connect an insulated wire between pins 9 and 10 on the signal connector module. Check that INJECT appears in the display.
4. Remove the wire connection between pins 9 and 10. LOAD should reappear in the display.

Testing the Manual Injector Valve

To test the manual injector valve:

1. Ensure that there is no electrical connection between pins 9 and 10.
2. Choose **Test START IN Sig**. LOAD should appear on the display.
3. Place the manual injector valve in the Inject position.
4. INJECT should appear in the display.

Press the selector knob to return to the Run Diagnostics screen.

Test Volt. Out Diagnostic

Use the Test Volt. OUT diagnostic to test the AU OUT, PRESS OUT, and %B OUT signals on the signal connector module.

Table 5-3 identifies the expected voltages ranges of each signal.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Table 5-3 Signal Voltage Ranges

Signal	Signal Connector Module Terminals	Minimum Voltage	Maximum Voltage
AU OUT	1 (+) and 2 (–)	–100 mV	+2000 mV
PRESS OUT	3 (+) and 4 (–)	0 mV	10 mV
%B OUT	5 (+) and 6 (–)	0 mV	10 mV

To test the signals:

1. Choose **Test Volt. out**. To display the voltage (in mV) being applied to the AU OUT, PRESS OUT, and %B OUT terminals on the signal connector module.
2. Connect a voltmeter to the AU OUT terminals on the signal connector module.
3. Rotate the selector knob to vary the voltage being applied to the AU OUT terminals on the signal connector module.
4. Compare the displayed voltage to the voltage indicated on the voltmeter to gauge the accuracy of the signal on the signal connector module. The voltages should be within ± 1.0 mV of each other.
5. Repeat steps 2 through 4 for the PRESS OUT and %B OUT signals.
6. Press the selector knob to return to the Run Diagnostics screen.

Test Keypad Diagnostic

Use the Test Keypad diagnostic to test the keypad control keys.

To test the keypad control keys:

1. Choose **Test Keypad**. The display shows a line of characters, each of which corresponds to one of the controls keys on the control panel keypad.
2. Test the control keys by pressing each key and verifying that the corresponding screen character highlights.
3. Press the selector knob to return to the Run Diagnostics screen.

Wavelength vs AU Diagnostic

Use the Show Energy, AU, λ diagnostic to verify correct absorbance detector calibration.

To verify correct absorbance detector calibration:

1. Choose **Wavelength vs. AU**. The absorbance detector parameters appear (Figure 5-6).

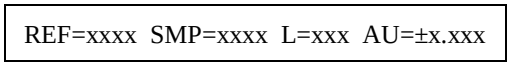


Figure 5-6 Show Energy, AU, λ Diagnostic Display

Each Show Energy, AU, λ diagnostic parameter is defined below.

- **REF** – The level of lamp energy measured by the reference photodiode.
- **SMP** – The level of lamp energy measured by the sample photodiode.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

- **L** – The wavelength of the light energy (in nm) produced by the diffraction grating in the absorbance detector.
- **AU** – The calculated level of absorbance based on the following equation:

$$\text{AU (Absorbance)} = \text{Log K } \frac{\text{REF}}{\text{SMP}}$$

Where K is configured as a constant of the instrument optics at the time of the Autozero function. K is held constant throughout a given separation while the values of REF and SMP are continuously monitored to compute the value of AU.

2. Rotate the selector knob to vary L (in 1-nm increments) while you monitor the changes in REF, SMP, and AU values.
3. Press the selector knob to return to the Run Diagnostics screen.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Chapter 6

Maintaining the CHDF 2000

Matec Applied Sciences
56 Hudson St., Northborough, MA 01532 Ph: (508) 393-0155 Fax: (508) 393-5476
www.matec.com e-mail: colloid@matec.com

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6 Maintaining the CHDF 2000

This chapter provides procedures for maintaining the CHDF 2000.

6.1 Maintaining the Pump

Figure 6-1 identifies the major components of a CHDF 2000 eluant pump.

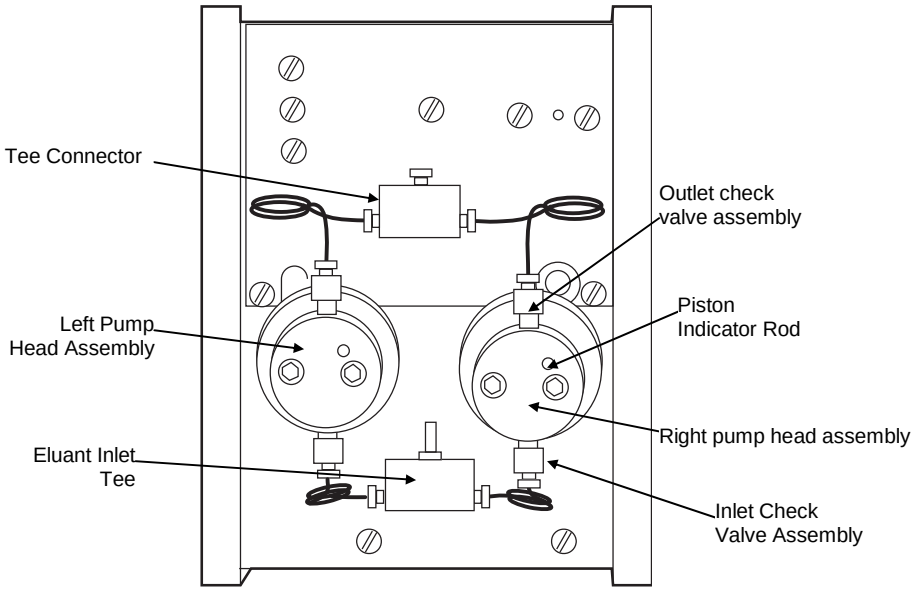


Figure 6-1 Pump Components

This section includes the following pump maintenance procedures:

- Replacing the plunger seals
- Cleaning and replacing the plunger
- Cleaning and replacing the check valves
- Replacing the in-line filter
- Handling plugged cartridges

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.1.1 Replacing the Plunger Seals

The two plungers in each pump are ultra-smooth, chemically inert sapphire rods. Salt crystals can form on a plunger and cause wear on the plunger seals and on the plunger itself.
Small pieces of a worn plunger seal may get caught in the outlet check valve of the pump head. If the plunger seals are worn, you may have to inspect and replace the outlet check valve. To replace an outlet check valve, see Section 6.1.3, Cleaning and Replacing the Check Valves.

Required Materials

- 5 mL syringe
- 5/16-inch open-end wrench
- 5/32-inch Allen wrench
- Brass machine screw
- Replacement seals and washers

To Replace a Plunger Seal:

1. Insert a syringe into the priming valve for the pump (see Figure 4-6).
2. Open the priming valve.
3. Place the eluant reservoir below the level of the eluant inlet tube. Remove the inlet line from the inlet tee of the pump and allow the eluant to drain back into the reservoir.
4. Set the Priming Flow Rate parameter to **0.5** mL/min (refer to Section 3.3, Reviewing and Editing Operating Parameters).
5. Use the Prime Pump option to turn on the pump (refer to Section 4.2, Priming the Pumps).
6. Use the syringe to extract as much eluant as possible from the pump.
7. Use the Prime Pump option to stop the pump when the indicator rod is retracted into the pump head (this protects the plunger from breaking under the weight of the pump head).
8. Using the 5/16-inch open-end wrench, remove the outlet tube from the outlet check valve of the pump head (see Figure 6-1).
9. Remove the inlet tube from the inlet check valve (see Figure 6-1).
10. Using a 5/32-inch Allen wrench, remove the two pump-head-assembly mounting bolts (Figure 6-2).

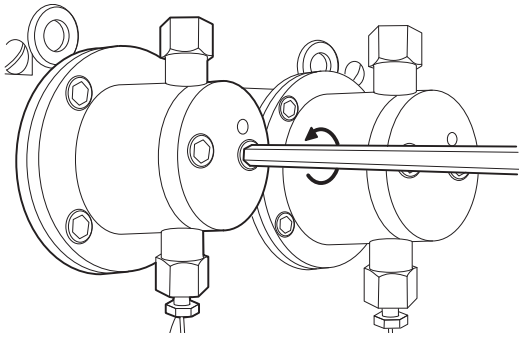


Figure 6-2 Unscrewing the Pump Head

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

7. Carefully slide the pump head assembly off the sapphire plunger by pulling and gently twisting the head straight off the pump assembly.

8. Screw the brass machine screw into the center opening in the plunger seal (Figure 6-3). Pull out the plunger seal and washer.



Figure 6-3 Removing the Plunger Seal

9. Place the new washer on the new plunger seal and insert the plunger seal into the pump head assembly.

10. Wet the seal and plunger with eluant.

***Note**: If you remove the pump head, pull out and gently release the piston indicator rod to have it rest on the piston inside the pump. This prevents damage to the rod during maintenance.*

11. Slide the pump head assembly into position over the plunger and replace the two pump head mounting bolts. Tighten the bolts alternately.

    If you are replacing plunger seals in both pump heads, repeat steps 4 through 11 for the other pump head.

12. Use the Prime Pump function to turn on the pump (refer to Section 4.2, Priming the Pumps). Check that the indicator rod moves freely.

13. Use the Prime Pump function to turn off the pump (refer to Section 4.2, Priming the Pumps).

14. Reconnect the eluant lines.

15. Return the eluant reservoir to its normal position.

16. Prime the pump, as described in Section 4.2, Priming the Pumps.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.1.2 Cleaning and Replacing the Plungers

The plungers require careful handling. Although cleaning the plungers is not difficult, it is important to follow these instructions carefully to avoid damaging the plungers. Assemble all materials and read the procedure thoroughly before you begin.

This section provides the following plunger maintenance procedures.

- Accessing the plunger
- Cleaning the plunger
- Inspecting the plunger
- Replacing the plunger

Required materials

- 5/16-inch open-end wrench
- 5/32-inch Allen wrench
- 9/64-inch Allen wrench
- Snap-ring pliers
- Plunger insertion tool
- Fine abrasive, such as toothpaste

Accessing the Plunger

To remove the plunger from the pump head:

1. Remove the pump head as described in Section 6.1.1, Replacing the Plunger Seals.
2. Remove the four plunger support screws with the 9/64-inch Allen wrench. Carefully slide the head support assembly and the indicator rod off the pump and set the head support assembly on the benchtop (Figure 6-4).

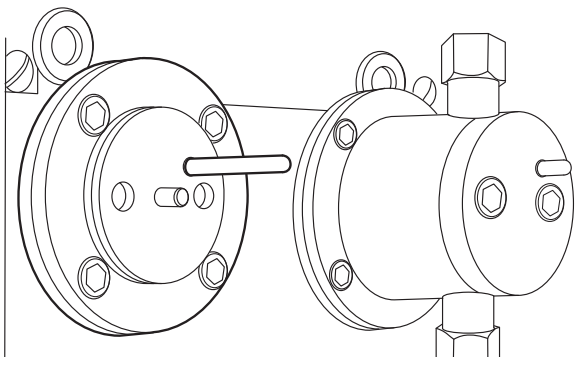


Figure 6-4 Left Head Support Assembly Exposed

3. Set the Priming Flow Rate parameter to **0.5** mL/min (refer to Section 3.3, Reviewing and Editing Operating Parameters).
4. Use the Prime Pump function to turn on the pump (refer to Section 4.2, Priming the Pumps).
5. Run the pump until the plunger is fully extended, then turn off the pump.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Caution: *To prevent injury, be sure to secure the plunger at all times during this procedure to prevent it from ejecting from the pump head.*

6. Use the plunger insertion tool to compress the plunger spring with one hand. With the other hand, use the snap-ring pliers to remove the snap-ring that holds the plunger in place.
7. Remove the plunger assembly (Figure 6-5) and set it on a clean flat surface.

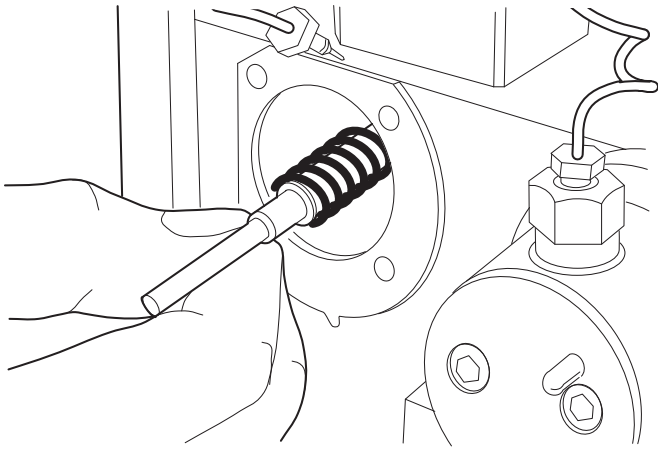


Figure 6-5 Removing the Plunger Assembly

Cleaning the Plunger

Buffers and impurities in eluants can coat the plunger and reduce the life of the seal. Cleaning the pump plunger periodically can prolong the life of the seal. Clean the plunger with a fine pumice or clear abrasive toothpaste. Rinse the plunger thoroughly with water or methanol to remove all traces of the abrasive.

The parts of the plunger assembly are shown in Figure 6-6.

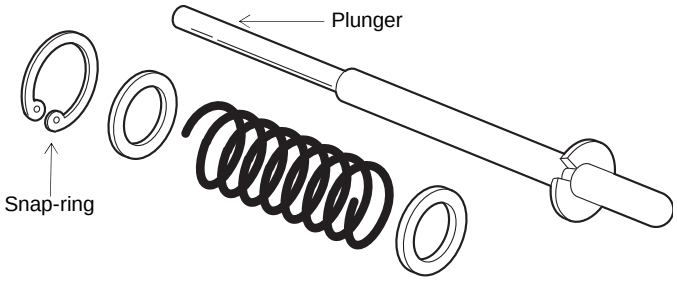


Figure 6-6 Disassembled Plunger

Inspecting the Plunger

After you clean the plunger, inspect it for damage by holding it under a bright white light and looking down its length for nicks and scratches. It is easier to see scratches under a bright light than to feel them with your fingers.

If the plunger is not scratched or otherwise damaged, reassemble the plunger. If the pump was leaking, install new pump seals. If the plunger is damaged, replace the plunger and the seals.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Replacing the Plunger

Whether you are reinstalling a plunger that you have cleaned and inspected, or replacing a damaged plunger with a new one, use the following procedure:

Caution: *Be very careful when handling the plunger. Wear safety glasses and use the plunger insertion tool. If you break the plunger, sharp pieces may be ejected from the instrument.*

To reinstall the plunger:

1. Assemble the plunger and use the plunger insertion tool (Figure 6-7) to reinsert the plunger into the pump.

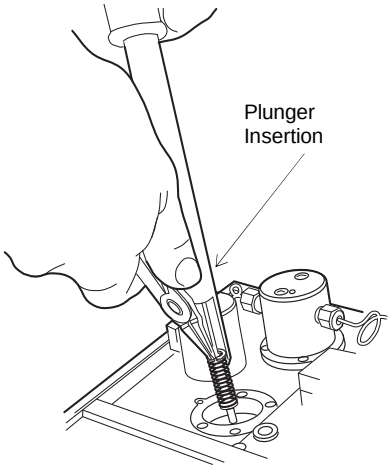


Figure 6-7 Inserting the Plunger

2. Use the Prime Pump function to turn on the pump (refer to Section 4.2, Priming the Pumps).
3. Wait for the plunger to retract, then turn off the pump.
4. Reinstall the head support assembly with the indicator rod in the upper right corner. Alternately tighten the four screws.
5. Wet the seal and plunger with eluant.
6. Gently slide the pump head onto the plunger and alternately tighten the two mounting bolts. Check the head alignment visually by observing the gap between the pump head and the pump head support. Make sure the indicator rod moves freely. If the head is misaligned, repeat step 4.
7. Set the Priming Flow Rate system parameter to **0.3** mL/min (refer to Section 3.3, Reviewing and Editing System Parameters).
8. Use the Prime Pump function to turn on the pump (refer to Section 4.2, Priming the Pumps).
9. Pull out and release the indicator rod. If the rod does not snap back easily, the head is misaligned. Loosen the pump head and repeat steps 6 and 9.
10. Reconnect the eluant lines, reposition the eluant reservoir, and prime the pump (see Section 4.2, Priming the Pumps).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.1.3 Cleaning and Replacing the Check Valves

This section provides the following check valve procedures:

- Cleaning the check valves
- Replacing an inlet check valve
- Replacing an outlet check valve

Cleaning the Check Valves

Clean the check valves (see Figure 6-1) every six months, or more frequently if your application warrants. It is a good idea to keep a spare set of clean check valves on hand. If you need to remove the check valves for a full cleaning, you can install the spare check valves immediately and clean the dirty check valves at a convenient time.

Required materials

- If you use organic eluants – 1 L of a compatible cleaning eluant, such as a 1:1 mixture of methanol and water with liquid detergent
- If you use aqueous buffers – 1 L of a mixture of water and liquid detergent

On-line cleaning

To clean the check valves while they are installed in the system:

1. Set the following operating parameter values (refer to Section 3.3, Reviewing and Editing Operating Parameters:
   - Priming Flow Rate to **10.0** mL/min
   - Priming Pump Time to **15** min.
2. Fill the pump eluant reservoir with at least 400 mL of cleaning eluant.
3. Open the priming valve of the pump to be cleaned.
4. Position a large beaker under the priming valve.
5. Use the Prime Pumps function to pump the cleaning eluant through the pump and into the beaker below the priming valve.
6. Repeat steps 1 to 5 if additional cleaning is required.
7. When you finish, close the priming valve.
8. Restore the Priming Flow Rate and Priming Pump Time operating parameters to their original values.

Full cleaning

If the on-line cleaning procedure is insufficient, take the check valves off the pump (as described in the next two sections) and immerse the valves in an upright position for 10 minutes in methanol. If a check valve does not work properly after cleaning, replace the valve.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Replacing an Inlet Check Valve

Required materials

- 5/16-inch open-end wrench
- 1/2-inch open-end wrench

To replace the inlet check valves:

1. Place the eluant reservoir below the level of the eluant inlet line. Remove the inlet tube from the inlet tee and allow the eluant to drain back into the reservoir.
2. Use the 5/16-inch open-end wrench to remove the tubing from the check valve.
3. Use the 1/2-inch open-end wrench to loosen the inlet check valve. Then remove the check valve by hand.
4. Remove the old gasket.
5. Install a new check valve and gasket. Hand-tighten the check valve, then tighten it another half-turn with the wrench.
6. Reconnect the eluant lines, reposition the eluant reservoir, and prime the pump (see Section 4.2, Priming the Pumps). Check for leaks.

Replacing an Outlet Check Valve Seal and Gasket

Required materials

- 5/16-inch open-end wrench
- 1/2-inch open-end wrench
- Dentist's pick
- Thick plastic pen cap or other blunt tool (such as the plunger insertion tool)

To replace the outlet check valve seal and gasket:

1. Use the 5/16-inch wrench to disconnect the outlet tubing.
2. Use the 1/2-inch open-end wrench to loosen the check valve a half-turn. Do not remove it.
3. Remove the pump head (refer to Section 6.1.1, Replacing the Plunger Seals, steps 1 to 7).
4. Holding the pump head upside-down in one hand with the outlet check valve facing the floor, unscrew the outlet check valve (**Error! Reference source not found.**) and set the pump head down. Set aside the outlet check valve in a safe place, ensuring that it stays upside down (to prevent the internal parts from falling out).

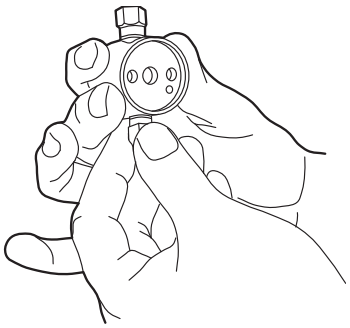


Figure 6-8 Removing the Outlet Check Valve

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

5. Being careful not to scratch the inside of the pump head, use the dentist's pick to pry the old gasket and outlet check valve seal out of the pump head. Note the orientation of the gasket.
6. Orient the new gasket in the same direction as the old gasket. Press the new seal and gasket into place with the end of the blunt tool.
7. Screw the check valve into the upside-down pump head until finger-tight. Use the wrench to tighten the check valve another 1/8-turn.
8. Install the pump head as in Section 6.1.1, Replacing the Plunger Seals.
9. Reconnect the eluant line.

6.1.4 Replacing the In-Line Filter

Replace the in-line filter when the system experiences high back pressure.

Caution: *To avoid chemical and electrical hazards, always wear safety glasses, turn off the instrument power, and disconnect the power cord before you perform this procedure.*

Required materials

- Two 5/8-inch open-end wrenches
- One 5/16-inch wrench
- New filter insert (from start-up kit)

To replace the in-line filter:

1. Reduce the pump flow rate to 0 mL/min.
2. Hold the in-line filter on the front of Pump A with a 5/8-inch wrench while you disconnect the two compression screws with a 5/16-inch wrench.
3. Disassemble the in-line filter using the two 5/8-inch wrenches.
4. Wash the in-line filter housing with methanol.
5. Assemble the in-line filter with a new filter insert (Figure 6-9).

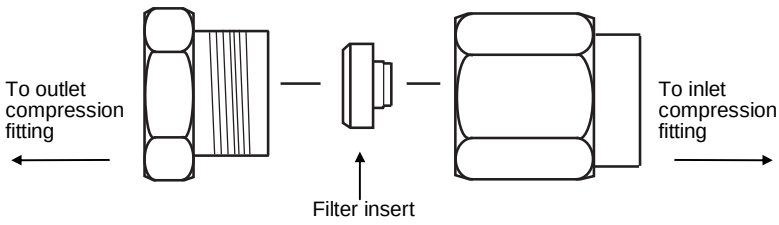


Figure 6-9 Assembling the In-Line Filter

6. Re-attach the assembled in-line filter at the front of the pump.
7. Reconnect the power cord, then turn on instrument power.
8. Inspect the in-line filter for leaks. Tighten 1/4 turn (if necessary).
9. Precondition the in-line filter by running methanol through it at 1.0 mL/min for approximately 15 minutes.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.2 Maintaining the Manual Injector Valve

Figure 6-10 identifies the major components of the CHDF 2000 manual injector valve.

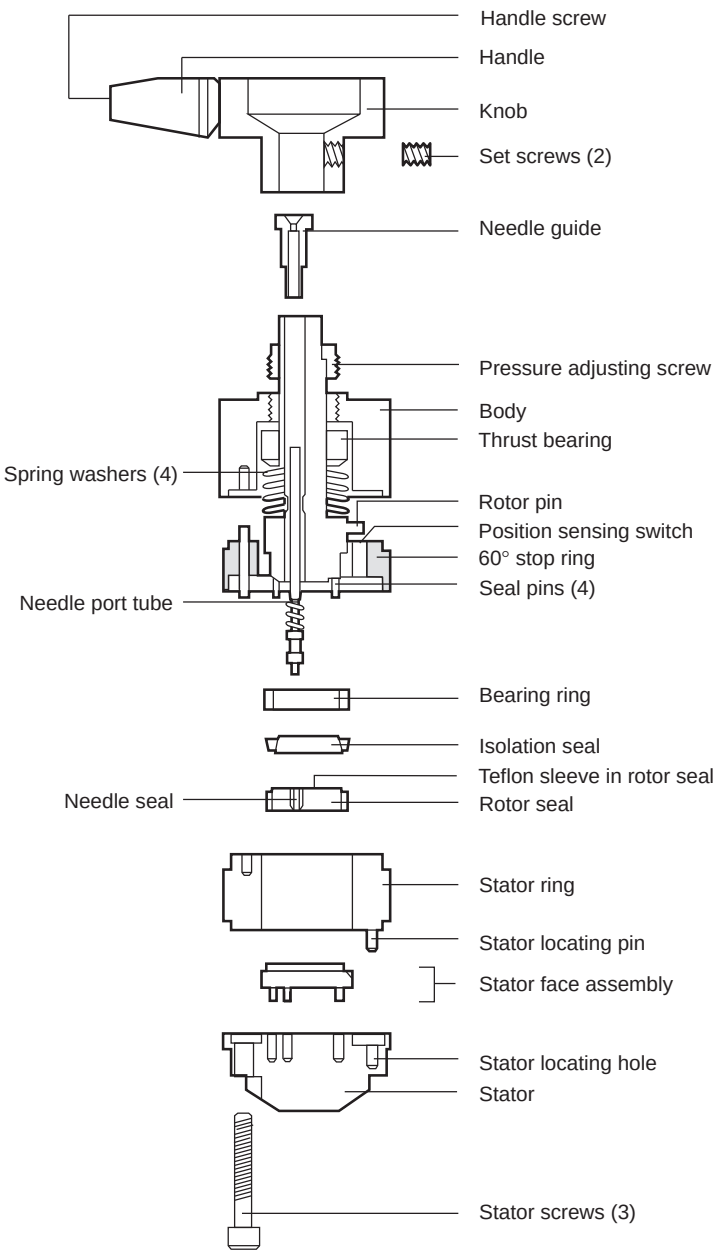


Figure 6-10 Exploded View of Manual Injector Valve

This section includes the following injector maintenance procedures:

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

- Tightening the pressure adjusting screw
- Replacing the rotor seal
- Tightening the needle seal

6.2.1 Tightening the Pressure-Adjusting Screw

Tighten the pressure-adjusting screw to stop minor leaks in the manual injector valve.



Caution: *To avoid chemical and electrical hazards, always wear safety glasses, turn off the instrument power, and disconnect the power cord before you perform this procedure.*

Required materials

- Phillips-head screwdriver
- 5/64-inch Allen wrench.

To tighten the pressure-adjusting screw:

1. Turn off instrument power and disconnect the power cord.
2. Locate the pressure-adjusting screw on the shaft of the injector (see Figure 6-10).
3. Loosen the two injector handle set screws.
4. Let the handle slide down the shaft so that its two tabs fit into the slots on the adjusting screw.
5. Use the knob as a wrench to tighten the pressure-adjusting screw approximately 1/20th of a turn. Use the 20 dial markings on the body and the painted spot on the screw to gauge how far to tighten the screw.
6. If the new setting fails to accomplish leak-free operation, repeat step 5 only once. Avoid excessive tightening, which increases rotor seal wear.
7. Finish by retightening the knob set screws onto the flats of the shaft.
8. If this procedure fails to stop the leak, replace the rotor seal as described in Section 6.2.2, Replacing the Rotor Seal.

6.2.2 Replacing the Rotor Seal

Replace the rotor seal if the manual injector valve leaks (after first tightening the pressure-adjusting screw).



Caution: *To avoid chemical and electrical hazards, always wear safety glasses, turn off the instrument power, and disconnect the power cord before you perform this procedure.*

Required materials

- Phillips-head screwdriver
- Flat-blade screwdriver
- 9/64-inch Allen wrench
- 5/16-inch open-end wrench

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Removing the Rotor Seal

To remove the rotor seal:

1. Turn off instrument power and disconnect the power cord.
2. Remove the CHDF 2000 top front cover.
3. Leave the injector attached to the front panel of the system, and leave the knob on.
4. Disconnect the tubing from the back of the injector valve.
5. Remove the three stator screws.
6. Pull axially to remove the following items:
   - Stator and stator face assembly (remove together). If the stator face assembly is damaged, replace it.
   - Stator ring.
7. Use a flat-blade screwdriver to pry the rotor seal off the four seal pins. Leave the isolation seal and bearing ring in place.

Reassembling the Injector

To reassemble the injector with a new rotor seal:

1. Loosen the pressure-adjusting screw one half-turn.
2. Note the original position of the two red dots.
3. Orient the rotor seal as shown in Figure 6-11, with the rotor seal slots facing the stator.

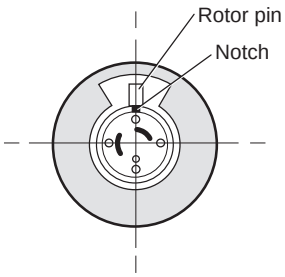


Figure 6-11 Rotor Seal Orientation (Viewed from Stator)

4. Replace the stator ring so that the pin in the 60° stop ring enters the mating hole in the stator ring.
5. Install the stator face assembly on the stator. The three pins on the stator face assembly fit only one way into the mating holes in the stator.
6. Install the stator and stator face assembly on the injector so that the pin in the stator ring enters the mating hole in the stator.
7. Tighten each of the three stator screws a little at a time to keep the stator surface parallel to the stator ring surface until all parts are held firmly in place.
8. Retighten the pressure-adjusting screw until the red dots are aligned as you noted them in step 2.
9. Replace the knob and tighten the two set screws against the two flat areas on the shaft.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.2.3 Tightening the Needle Seal

The needle seal, a Teflon sleeve in the rotor seal, may not seal correctly around a thin needle. A poor seal reduces accuracy in sample loading.

Reshaping the Teflon Sleeve

To reshape the Teflon sleeve to make a good seal:

1. Remove the needle from the needle port.
2. Push gently on the plastic needle guide with the eraser end of a pencil. Do not squash the Teflon sleeve.
3. Repeat if necessary.

Note: *To avoid damaging the rotor seal, always use a needle of the correct type and gauge to load the injector.*

Checking the Needle Seal

To check for a proper seal around the needle:

1. Turn off the pump using the Prime Pumps option.
2. Fill the syringe with water.
3. Place the injector in the Load position and slowly discharge the water in the syringe into the injector.
4. Note the low resistance to syringe discharge.
5. Fill the syringe with water and repeat step 2 with the injector handle halfway between the Load and Inject positions.
6. Verify that the resistance to discharging the syringe is now much greater than in step 3.

Note: *The needle seal holds only a few bars of pressure, and does not completely prevent syringe discharge when the injector handle is in the halfway position.*

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.3 Maintaining the Priming Valves

Replace the priming valve seal if you suspect a leak.

To replace the priming valve seal:

1. Obtain a seal replacement kit from your authorized service agent. The kit contains two sets of seals and a hex tool.
2. Turn off the pump (use the Prime Pumps option) and remove the eluant tubing from the reservoir.
3. Remove the valve stem assembly from the valve by unscrewing the hex nut (Figure 6-12) to prevent any siphon flow. Catch any eluant drainage in a suitable container.

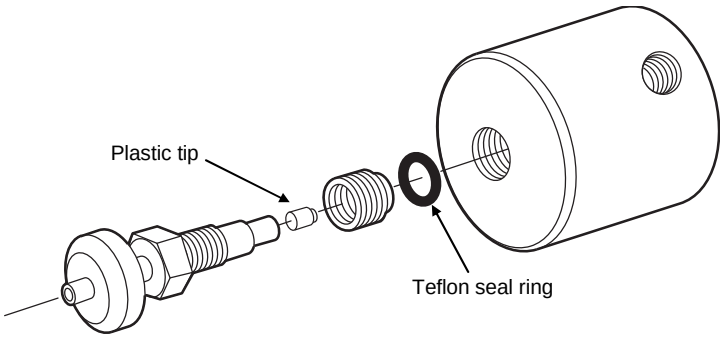


Figure 6-12 Priming Valve Assembly

Note: *Be careful not to scratch or otherwise damage the internal parts of the valve while removing the seal retainer.*

4. Use the hex tool to turn the hex socket seal retainer and remove it.
5. Using a wire with a small right angle bend formed in the end, carefully remove the Teflon seal ring from the valve body. Do not scratch the sealing surface around the seal.
6. Pull the plastic tip from the stem using tweezers or small pliers.
7. By hand, insert a new plastic tip into the stem assembly, blunt end first. Seat the tip firmly with light pressure against a clean hard surface.
8. Insert a new Teflon seal ring into the valve body fully until it rests flat against the body beyond the threads.
9. Replace the seal retainer and tighten firmly with the hex tool.
10. Replace the stem assembly into the priming valve body and turn one or two turns past the point where you feel initial resistance.
11. Reconnect the eluant tubing.
12. Use the Prime Pumps option to run the pump for a few minutes to purge the valve.
13. Close the valve firmly to seat the tip seal.
14. Run the pump and check for leaks around the priming valve.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.4 Maintaining the Detector

This section provides procedures for replacing the deuterium lamp in the absorbance detector.

The procedure include:

- Removing the old lamp
- Installing the new lamp
- Adjusting the lamp position

Lamp Characteristics

The intensity of the deuterium source lamp varies by wavelength (Figure 6-13).

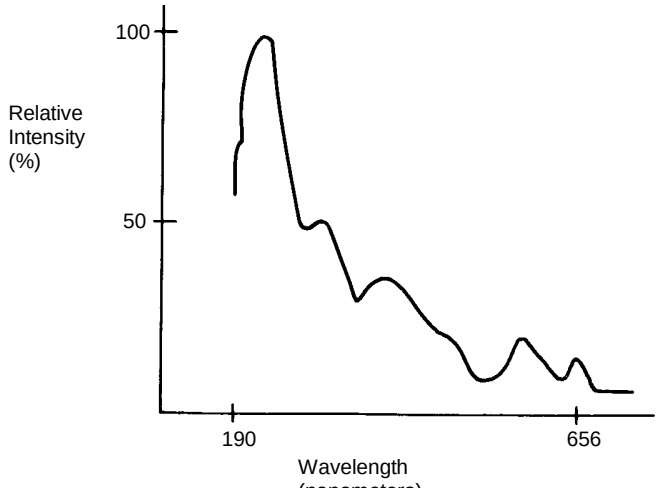


Figure 6-13 Deuterium Lamp Intensity Profile

When to Replace the Lamp

Performance requirements and permitted tolerances vary from application to application. If the lamp no longer provides an adequate signal-to-noise ratio for your application, replace it.

It is not unusual for lamps to show a 30 to 50 percent decrease in intensity before the noise increases by a factor of two.

Lamp Timer

The detector lamp has a life of approximately 1000 hours. One of the leads attached to the lamp includes a 1000-hour timer to indicate lamp usage.

Before you replace the lamp, check the lamp timer (see Figure 6-14).

The lamp timer is a mercury column with a scale of 0 to 10 where 10 represents 1000 hours. As the lamp ages, the bubble in the mercury column moves toward the 10.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Required materials

- Allen wrench
- Open-end wrench (3/8-inch)
- Flat-head screwdriver (small)
- Phillips-head screwdriver

Save all parts (screws, nuts, washers) after removal unless otherwise indicated. The parts are needed to install the new lamp.

6.4.2 Removing the Lamp



Caution: *To prevent eye damage from ultraviolet radiation exposure, turn off the unit and strictly adhere to the following procedures while changing the lamp.*

When you remove the lamp housing protective cover, the UV lamp automatically turns off.

Before removing the lamp, set the wavelength to 230 nm. If the lamp is bad or fails to light and this wavelength setting is not possible, remove the lamp and replace it as follows.



Caution: *The lamp housing becomes extremely hot during operation. To prevent burns to the skin, allow the lamp to cool for one hour before removing.*

1. Turn off the unit power and allow the lamp to cool for one hour.
2. Remove the front and rear system covers.
3. Loosen, but do not remove, the two screws that secure the lamp housing protective cover.
4. Remove the lamp housing by sliding it up and away from the unit.
5. Disconnect the lamp connector from the lamp interlock cable (Figure 6-14).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

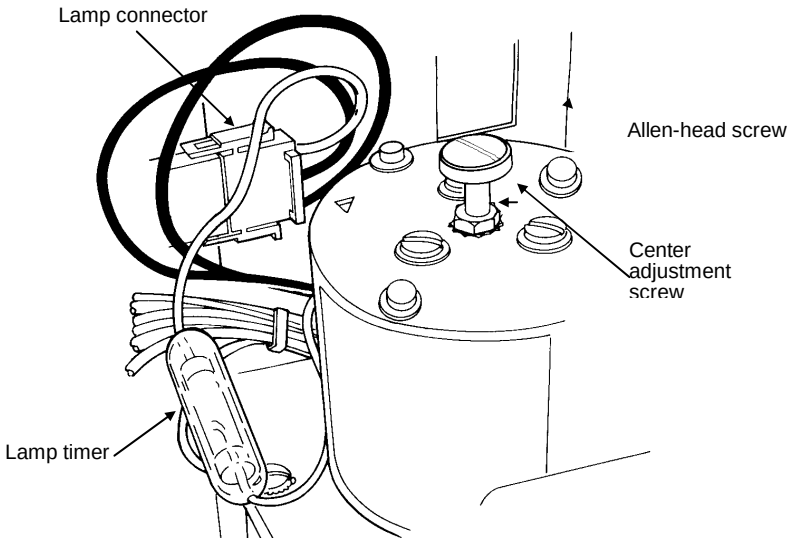


Figure 6-14 Lamp Assembly and Connections

4. From the top of the lamp assembly, remove the three Allen-head screws and flat washers (see Figure 6-14).
5. Pull the lamp assembly out of the lamp housing (Figure 6-15).

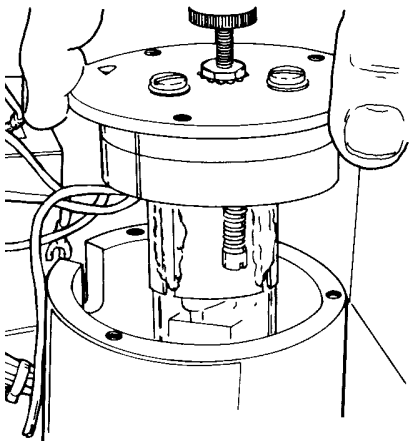


Figure 6-15 Removing the Lamp

6. From the top of the lamp assembly, remove the center adjustment screw along with the star washer and the hex nut. Place the lamp assembly on a flat surface with the bulb positioned upward (Figure 6-16).

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

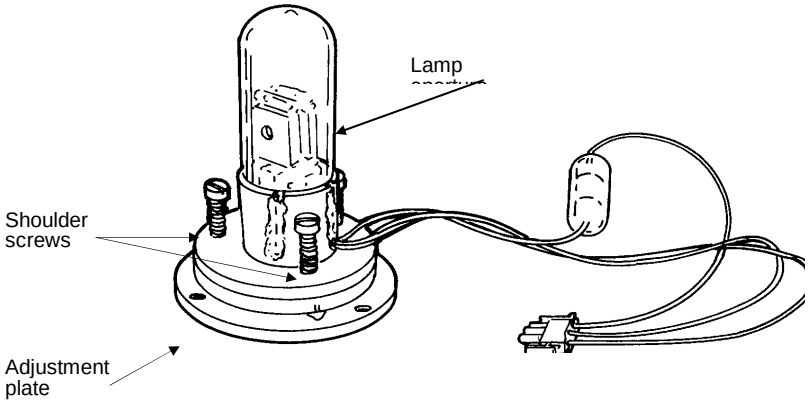


Figure 6-16 Lamp Assembly and Adjustment Plate

7. Remove the three shoulder screws and their springs from the lamp. The springs are loosely coiled around the screws and do not spring off the screw when removed. The lamp adjustment plate can now be separated from the deuterium lamp.

Caution: *Lamp gas is under slight pressure. Use care when disposing of the lamp to prevent shattering the glass.*

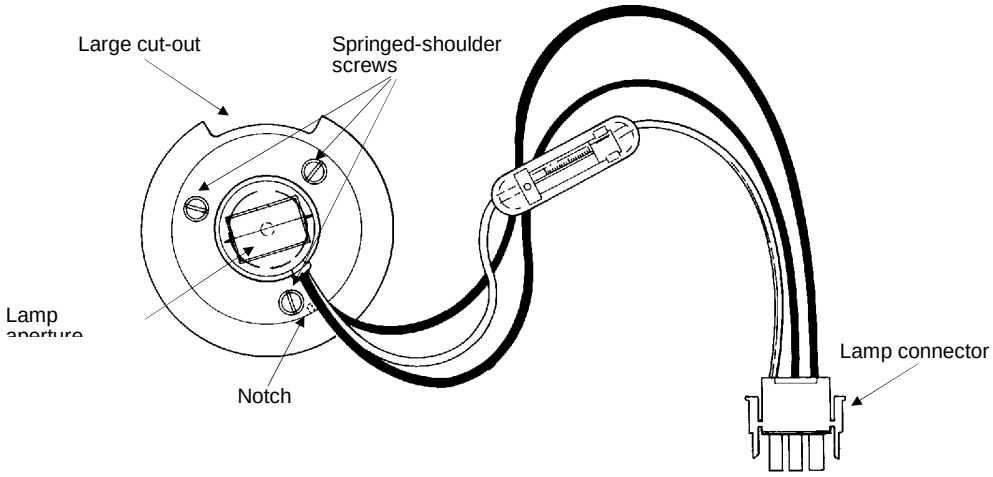
6.4.3 Installing the New Lamp

Unpack the lamp from its packing material. The new lamp may vary slightly from the lamp illustrated in Figure 6-16.

Note*: Do not touch the glass bulb on the new lamp. Dirt or fingerprints on the bulb affect detector operation. If the lamp needs cleaning, gently clean the bulb with ethanol and lens tissue. Do not use any abrasive tissue or excessive pressure.*

To install the new lamp:

1. Position the lamp housing on a flat surface so the large cut-out on the adjustment plate faces away from you (Figure 6-17).



Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Figure 6-17 Aligning the Lamp (Top View)

2. With the lamp aperture facing away from you, place the new lamp on top of the adjustment plate and secure it with the shoulder screws. The lamp leads should line up with the notch on the lamp adjustment plate.
3. Without touching the bulb, turn the assembly so that the bulb is positioned downward.
4. Place the center adjustment screw with the hex nut and star washer in the center opening of the adjustment plate. In preparation for maximizing the lamp energy, turn the screw until the lamp assembly moves about 3-4 mm away from the adjustment plate.
5. Place the lamp assembly onto the lamp housing. Make sure the lamp leads slide freely into the housing slot.
6. Secure the lamp assembly to the housing and tighten the three Allen-head screws.
7. Plug the lamp connector into the lamp power cable assembly (see Figure 6-14).
8. Replace the protective shield to prevent direct access to the lamp when instrument power is on.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.4.4 Adjusting the Lamp Alignment

After you install the lamp, position the lamp for maximum lamp energy. The flowchart in Figure 6-18 illustrates the procedure for positioning the lamp adjustment plate (Figure 6-16) for maximum lamp energy.

Note: *The detector does not have to be stabilized to perform this procedure.*

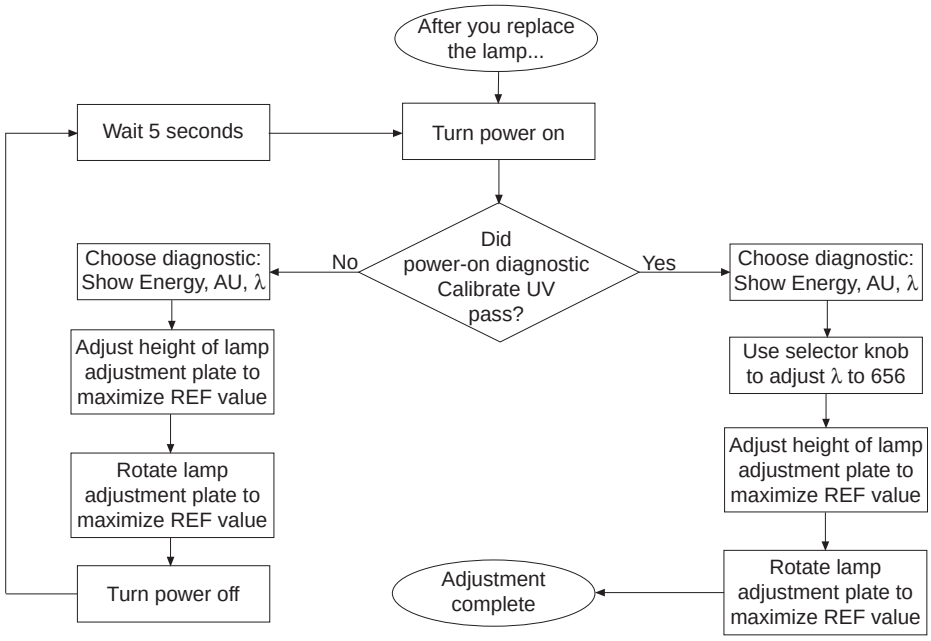


Figure 6-18 Adjusting for Maximum Lamp Energy

To maximize lamp energy (see Figure 6-18):

1. Turn on power for the CHDF 2000. The Power-On Diagnostics screen appears (see Figure 3-2).

Caution: *To prevent the possibility of exposing your eyes to ultraviolet radiation, wear eye protection that filters ultraviolet light and keep the lamp in the lamp housing during operation.*

2. Make a note indicating the result (pass/fail) of the Calibrate UV power-on diagnostic.
3. Choose **Run diagnostics**. The Run Diagnostics screen appears (see Figure 5-5).
4. Choose **Show Energy, AU, λ**. The current wavelength (λ) setting, the detected absorbance (AU), and the current lamp sample (SMP) and reference energy (REF) values appear (see Figure 5-6).

   If the result of the Calibrate UV power-on diagnostic was Pass, go to step 10.

   If the result of the Calibrate UV power-on diagnostic was Fail, continue with step 5.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com



Caution: *To prevent burn injury, do not touch the lamp assembly directly.*

5. Observe the displayed REF value while you turn the center adjustment screw (see Figure 6-14) to maximize the REF value. Be sure the displayed value is stable before continuing.
6. If the screws on top of the lamp assembly (see Figure 6-14) are tightened, use an Allen-head wrench to loosen them.
7. Observe the displayed REF value while you rotate the lamp adjustment plate to maximize the REF value.
8. Turn off power to the CHDF 2000.
9. Wait five seconds, then restart this procedure from step 1.
10. Rotate the selector knob to set the wavelength (AU) to **656** (nm).



Caution: *To prevent burn injury, do not touch the lamp assembly directly.*

11. Observe the displayed REF value while you turn the center adjustment screw (see Figure 6-14) to maximize the REF value. Be sure the displayed value is stable before continuing.
12. If the screws on top of the lamp assembly (see Figure 6-14) are tightened, use an Allen-head wrench to loosen them.
13. Observe the displayed REF value while you rotate the lamp adjustment plate to maximize the REF value.
14. Repeat steps 11 through 13 to further maximize lamp energy.
15. Turn off the instrument and check that all detector screws and connectors are secure.
16. Replace the instrument covers and fasten the cover screws.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.5 Adjusting the Control Panel Display

This section provides procedures for adjusting the brightness and contrast of the control panel display.

To adjust the control panel display brightness and contrast:

1. Loosen, but do not remove, the access cover retaining screws on the inside of the front door (see Figure 2-9).
2. Remove the access cover.

Caution: *To avoid damage to the control panel electronics, use a non-metal screwdriver to adjust the Brightness and Contrast controls.*

3. Observe the control panel display while you use a non-metal, flat-blade screwdriver to adjust the Brightness adjustment screw on the back of the front door (**Error! Reference source not found.**).

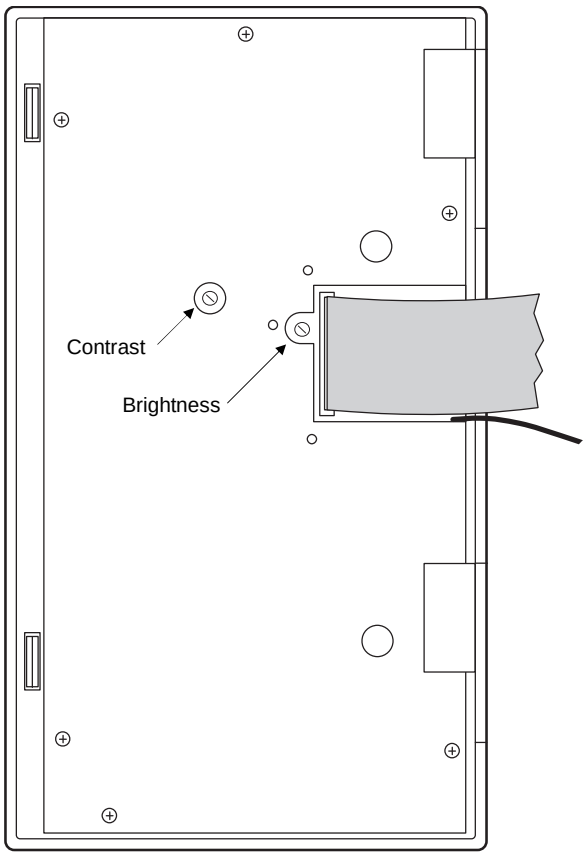


Figure 6-19 Brightness and Contrast Adjusting Screws

5. Observe the control panel display while you use a non-metal, flat-blade screwdriver to adjust the Contrast adjustment screw (see **Error! Reference source not found.**).
6. Reinstall the access cover and tighten the retaining screws. Be careful not to damage the control panel ribbon cable or ground wire.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.6 Dealing with Plugged Cartridges

The CHDF capillaries are contained in two cartridges as follows (also see section 2.4):

1. The top, or Fractionation cartridge (white).

2. The bottom cartridge (black).

The top cartridge contains the capillary responsible for particle fractionation. The bottom cartridge contains the "make-up", as well as the "waste" capillaries. The CHDF cartridges can be responsible for the following two problems:

1. No peaks are detected upon injection of samples and /or marker.
This condition is frequently caused by a faulty top cartridge.

2. High pump pressure. This is usually caused by a faulty bottom cartridge, or a plugged in-line filter.

Normally, the top cartridge is not responsible for high pressure, even if it is totally plugged. This is because only a very minor portion of the eluant flows into the fractionation capillary. High pressure can also be caused by a blocked cartridge in-line filter as described in section 6.6.3.
There are 3 options for fixing the top cartridge fault which is responsible for lack of sample detection. These are snipping off the end of the capillary, backflushing the capillary (see section 5), and replacing the fractionation capillary.
Chapter 5 discusses how to flush the cartridge with solvent, or backflush it to remove a plug. If either of these techniques are not successful, there are two techniques discussed here that will ultimately take care of the problem. The first, snipping off the end of the capillary, assumes the plug is in the very end of the cartridge, and that this section will be removed when snipped off. The second method described here, changing the fractionation cartridge, guarantees that the plugging problem will be resolved.

A third section below shows how to clean the cartridge in-line filter in order to eliminate high pump backpressure.

6.6.1 Snipping off the End of the Capillary

The idea is to dismount the white fractionation cartridge from the CHDF2000, with the two 5-inch long, 1/16" stainless steel tubes still attached to the cartridge; once on the bench, remove the inlet SS tube, leaving the capillary exposed, then snip the capillary. See Fig. 6.20. Details are shown below.

1. Stop the pump on the CHDF 2000, wait for the pump pressure to drop below 1,000 psi.

2. Open the front door of the instrument. The cartridge is located in the center top position, and is shown in Figure 6.20.

3. Disconnect the cartridge fitting from the instrument at locations 1, 2, 3, and 4 shown in Figure 6.20. CAUTION: DO NOT LOOSEN FITTINGS NUMBER 5 and 6 YET. The fused-silica capillary stretches into the two stainless steel tubes, 7 and 8. Detaching tubes 7 and 8 at this point will result in fractionation capillary breakage.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

4. Unscrew the three screws holding the cartridge in place. Gently remove the cartridge from the instrument.

5. Using your thumb, straighten SS tube 7 somewhat.

6. Disconnect the fitting from the cartridge at location 6 (bottom cartridge inlet) shown in figure 6.20.

7. Pull the SS tube 7 away from the cartridge to expose the small, brown, glass capillary tube.

8. Score the capillary with a ceramic disc, then break off a 1/8" piece of capillary.

9. Replace the tubing in reverse order that it was disconnected.

Replace the cartridge using the screws provided. Re-connect the four fittings. Do not over-tighten the fittings. Tighten ¼ turn past finger-tight.

10. Run known samples or standards to see if the cartridge is working properly.

6.6.2 Replacing the Fractionation Cartridge

If the snipping procedure described in the above section does not solve your problem, the fractionation cartridge on the CHDF 2000 will need to be replaced. Once a new cartridge is purchased from Matec, it is a relatively simple procedure to install it. The cartridge is located behind the front door of the instrument, in the center, top position. Changing the cartridge is performed in the following steps.

1. Stop the pump on the CHDF 2000.

2. Open the front door of the instrument. The cartridge is located in the center top position, and it should look like this (Figure 6.20):

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

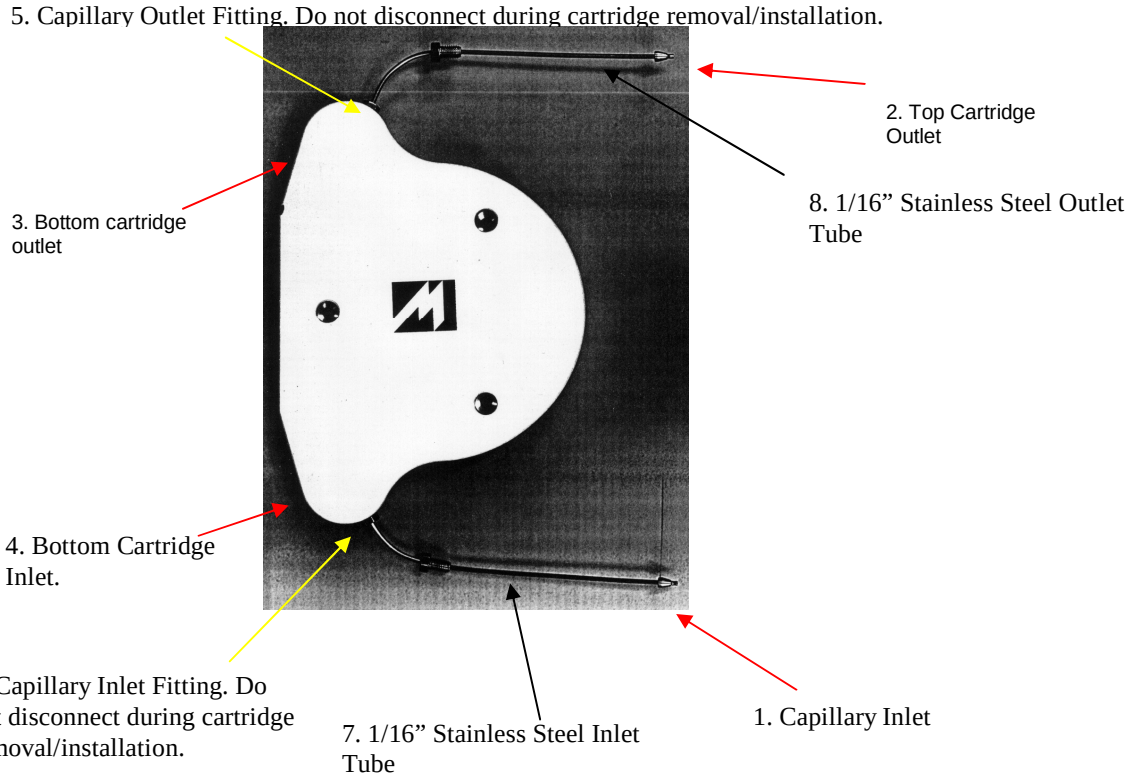


Figure 6.20 The Fractionation Cartridge. IMPORTANT NOTE: This is how your cartridge should look upon uninstalling it from the CHDF unit. Do not remove the Inlet and Outlet tubes until after this whole assembly has been removed from the CHDF unit. Otherwise, you will break the capillary inside these two stainless steel tubes.

3. Disconnect the cartridge fitting from the instrument at locations 1, 2, 3, and 4 shown in Figure 6.20. CAUTION: DO NOT LOOSEN FITTINGS NUMBER 5 and 6 YET. The fused-capillary stretches into the two stainless steel tubes, 7 and 8. Detaching tubes 7 and 8 at this point will result in fractionation capillary breakage.

Unscrew the three screws holding the cartridge in place.

4. Gently remove the cartridge from the instrument.

5. Remove the plastic caps from the tube ends of the new cartridge if they are present. Take the new cartridge and place it gently into the instrument.

6. Replace the cartridge using the 3 screws provided.

7. Reconnect the cartridge tubing to the instrument. Do not over-tighten the fittings. Tighten ¼ turn past finger-tight.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

6.6.3 Cleaning the In-line Filter (High Pump Pressure)

This filter keeps large particles from plugging the outlet to waste from the fractionation cartridge. It may plug on occasion causing high backpressure, and may need to be cleaned. It is cleaned as follows:

1. Open the door of the instrument and locate the filter next to the fractionation cartridge as seen in the drawing below.

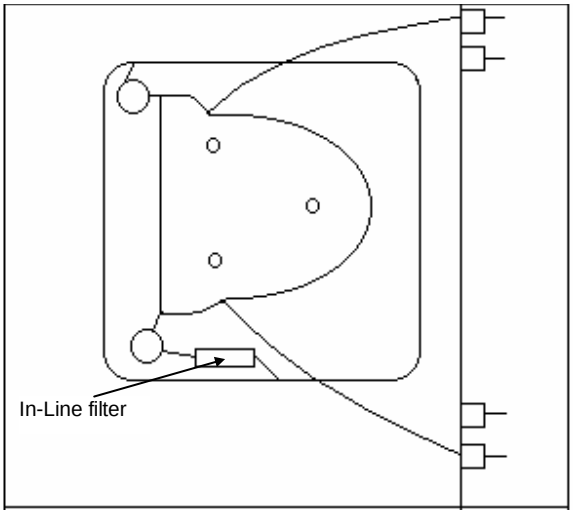


Fig. 6.21. Cartridge In-Line filter.

2. Using a wrench, loosen the connections on both ends of the filter, and remove the filter from the instrument.

3. The filter consists of a small metal piece screwed into a larger metal piece. The smaller metal piece contains the filter element. Unscrew the smaller piece and remove it from the larger piece.

4. Remove the fritted stainless steel filter element from the piece you just unscrewed.

5. If you analyze polymer samples, unplug the filter element by sonicating the element in acetone or tetrahydrofuran for at least 15 minutes. If you analyze silica or other inorganic substances, sonicate the element in a 50/50 (by volume) mixture of methanol and 1M NaOH for at least 15 minutes.

6. Re-install the filter in the reverse order of the steps above.

7. Cleaning the filter may also be performed by backflushing, as described in section 5.1.1.

8. If cleaning the filter element fails, you can replace the filter element with a new unit.

Artisan Technology Group - Quality Instrumentation ... Guaranteed | (888) 88-SOURCE | www.artisantg.com

Artisan Technology Group is an independent supplier of quality pre-owned equipment

Gold-standard solutions

Extend the life of your critical industrial, commercial, and military systems with our superior service and support.

We buy equipment

Planning to upgrade your current equipment? Have surplus equipment taking up shelf space? We'll give it a new home.

Learn more!

Visit us at **artisantg.com** for more info on price quotes, drivers, technical specifications, manuals, and documentation.

Artisan Scientific Corporation dba Artisan Technology Group is not an affiliate, representative, or authorized distributor for any manufacturer listed herein.

We're here to make your life easier. How can we help you today?

(217) 352-9330 | sales@artisantg.com | artisantg.com

ARTISAN® TECHNOLOGY GROUP