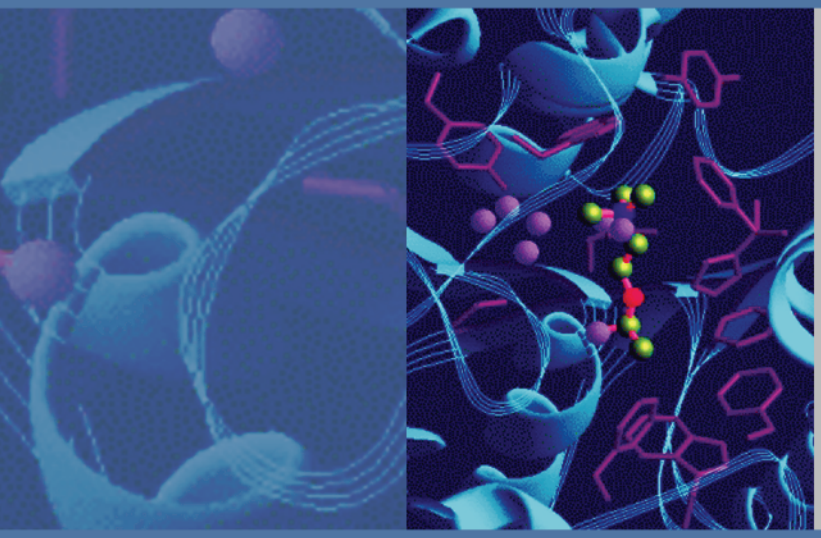

SpectraSYSTEM

P4000 Gradient Pump

User Guide

A0099-97004 Revision A August 2008

Thermo SCIENTIFIC

Release history: Revision A released August 2008

For Research Use Only. Not regulated for medical or veterinary diagnostic use by U.S. Federal Drug Administration or other competent authorities.

Regulatory Compliance

Thermo Fisher Scientific performs complete testing and evaluation of its products to ensure full compliance with applicable domestic and international regulations. When the system is delivered to you, it meets all pertinent electromagnetic compatibility (EMC) and safety standards as described below.

EMC Directive 2004/108/EC

EMC compliance has been evaluated by TUV Rheinland of North America.

CISPR 11: 1998	EN 61000-4-4: 2004
EN 55011: 1998, A1:1999, A2, 2002	EN 61000-4-5: 2001
EN 61000-3-2: 2000	EN 61000-4-6: 2003
EN 61000-3-3: 1995, A1: 2001	EN 61000-4-11: 2001
EN 61000-4-2: 2001	EN 61326-1: 1997, A1: 1998, A2: 2001, A3: 2003
EN 61000-4-3: 2002	CFR 47: 2007

Low Voltage Safety Compliance

Low voltage safety compliance has been evaluated by TUV Rheinland of North America.

This device complies with Low Voltage Directive 2006/95/EC, harmonized standard EN 61010-1: 2001, IEC 61010-1: 2002, UL 61010A-1: 2004, and CAN/CSA 22.2 61010-1: 2004.

Changes that you make to your system may void compliance with one or more of these EMC and safety standards. Changes to your system include replacing a part or adding components, options, or peripherals not specifically authorized and qualified by Thermo Fisher Scientific. To ensure continued compliance with EMC and safety standards, replacement parts and additional components, options, and peripherals must be ordered from Thermo Fisher Scientific or one of its authorized representatives.

FCC Compliance Statement

THIS DEVICE COMPLIES WITH PART 15 OF THE FCC RULES. OPERATION IS SUBJECT TO THE FOLLOWING TWO CONDITIONS: (1) THIS DEVICE MAY NOT CAUSE HARMFUL INTERFERENCE, AND (2) THIS DEVICE MUST ACCEPT ANY INTERFERENCE RECEIVED, INCLUDING INTERFERENCE THAT MAY CAUSE UNDESIRED OPERATION.

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CAUTION Read and understand the various precautionary notes, signs, and symbols contained inside this manual pertaining to the safe use and operation of this product before using the device.

Notice on Lifting and Handling of Thermo Scientific Instruments

For your safety, and in compliance with international regulations, the physical handling of this Thermo Fisher Scientific instrument ***requires a team effort*** to lift and/or move the instrument. This instrument weighs 18 kg (40 lbs) and is too heavy for one person alone to handle safely.

Notice on the Proper Use of Thermo Scientific Instruments

In compliance with international regulations: Use of this instrument in a manner not specified by Thermo Fisher Scientific could impair any protection provided by the instrument.

Notice on the Susceptibility to Electromagnetic Transmissions

Your instrument is designed to work in a controlled electromagnetic environment. Do not use radio frequency transmitters, such as mobile phones, in close proximity to the instrument.

For manufacturing location, see the label on the instrument.

WEEE Compliance

This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:

Thermo Fisher Scientific has contracted with one or more recycling or disposal companies in each European Union (EU) Member State, and these companies should dispose of or recycle this product. See www.thermo.com/WEEERoHS for further information on Thermo Fisher Scientific's compliance with these Directives and the recyclers in your country.

WEEE Konformität

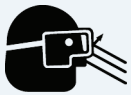
Dieses Produkt muss die EU Waste Electrical & Electronic Equipment (WEEE) Richtlinie 2002/96/EC erfüllen. Das Produkt ist durch folgendes Symbol gekennzeichnet:

Thermo Fisher Scientific hat Vereinbarungen mit Verwertungs-/Entsorgungsfirmen in allen EU-Mitgliedsstaaten getroffen, damit dieses Produkt durch diese Firmen wiederverwertet oder entsorgt werden kann. Mehr Information über die Einhaltung dieser Anweisungen durch Thermo Fisher Scientific, über die Verwerter, und weitere Hinweise, die nützlich sind, um die Produkte zu identifizieren, die unter diese RoHS Anweisung fallen, finden sie unter www.thermo.com/WEEERoHS.

Conformité DEEE

Ce produit doit être conforme à la directive européenne (2002/96/EC) des Déchets d'Equipements Electriques et Electroniques (DEEE). Il est marqué par le symbole suivant:

Thermo Fisher Scientific s'est associé avec une ou plusieurs compagnies de recyclage dans chaque état membre de l'union européenne et ce produit devrait être collecté ou recyclé par celles-ci. Davantage d'informations sur la conformité de Thermo Fisher Scientific à ces directives, les recycleurs dans votre pays et les informations sur les produits Thermo Fisher Scientific qui peuvent aider la détection des substances sujettes à la directive RoHS sont disponibles sur www.thermo.com/WEEERoHS.

CAUTION Symbol	CAUTION	VORSICHT	ATTENTION	PRECAUCION	AVVERTENZA
	**Electric Shock:** This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	**Elektroschock:** In diesem Gerät werden Hochspannungen verwendet, die Verletzungen verursachen können. Vor Wartungsarbeiten muß das Gerät abgeschaltet und vom Netz getrennt werden. Betreiben Sie Wartungsarbeiten nicht mit abgenommenem Deckel. Nehmen Sie die Schutzabdeckung von Leiterplatten nicht ab.	**Choc électrique:** L'instrument utilise des tensions capables d'infliger des blessures corprelles. L'instrument doit être arrêté et débranché de la source de courant avant tout intervention. Ne pas utiliser l'instrument sans son couvercle. Ne pas elensver les étuis protecteurs des cartes de circuits imprimés.	**Descarga eléctrica:** Este instrumento utiliza altas tensiones, capaces de producir lesiones personales. Antes de dar servicio de mantenimiento al instrumento, éste debera apagarse y desconectarse de la línea de alimentacion eléctrica. No opere el instrumento sin sus cubiertas exteriores quitadas. No remueva las cubiertas protectoras de las tarjetas de circuito impreso.	**Shock da folgorazione.** L'apparecchio è alimentato da corrente ad alta tensione che puo provocare lesioni fisiche. Prima di effettuare qualsiasi intervento di manutenzione occorre spegnere ed isolare l'apparecchio dalla linea elettrica. Non attivare lo strumento senza lo schermo superiore. Non togliere i coperchi a protezione dalle schede di circuito stampato (PCB).
	**Chemical:** This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	**Chemikalien:** Dieses Gerät kann gefährliche Chemikalien enthalten. Tragen Sie Schutzhandschuhe beim Umgang mit toxischen, karzinogenen, mutagenen oder ätzenden/reizenden Chemikalien. Entsorgen Sie verbrauchtes Öl entsprechend den Vorschriften in den vorgeschriebenen Behältern.	**Chimique:** Des produits chemiques dangereux peuven se trouver dans l'instrument. Proted dos gants pour manipuler tous produits chemiques toxiques, cancérigènes, mutagènes, ou corrosifs/irritants. Utiliser des récipients et des procédures homologuées pour se débarrasser des déchets d'huile.	**Química:** El instrumento puede contener productos quimicos peligrosos. Utilice guantes al manejar productos quimicos tóxicos, carcinogenos, mutagenos o corrosivos/irritantes. Utilice recipientes y procedimientos aprobados para deshacerse del aceite usado.	**Prodotti chimici.** Possibile presenza di sostanze chimiche pericolose nell'apparecchio. Indossare dei guanti per maneggiare prodotti chimici tossici, cancerogeni, mutageni, o corrosivi/irritanti. Utilizzare contenitori aprovo e seguire la procedura indicata per lo smaltimento dei residui di olio.
	**Heat:** Before servicing the instrument, allow any heated components to cool.	**Hitze:** Warten Sie erhitzte Komponenten erst nachdem diese sich abgekühlt haben.	**Haute Temperature:** Permettre aux composants chauffés de refroidir avant tout intervention.	**Altas temperaturas:** Permita que lop componentes se enfríen, ante de efectuar servicio de mantenimiento.	**Calore.** Attendere che i componenti riscaldati si raffreddino prima di effetturare l'intervento di manutenzione.
	**Fire:** Use care when operating the system in the presence of flammable gases.	**Feuer:** Beachten Sie die einschlägigen VorsichtsmaBnahmen, wenn Sie das System in Gegenwart von entzündbaren Gasen betreiben.	**Incendie:** Agir avec précaution lors de l'utilisation du système en présence de gaz inflammables.	**Fuego:** Tenga cuidado al operar el sistema en presencia de gases inflamables.	**Incendio.** Adottare le dovute precauzioni quando si usa il sistema in presenza di gas infiammabili.
	**Eye Hazard:** Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	**Verletzungsgefahr der Augen:** Verspritzte Chemikalien oder kleine Partikel können Augenverletzungen verursachen. Tragen Sie beim Umgang mit Chemikalien oder bei der Wartung des Gerätes eine Schutzbrille.	**Danger pour les yeux:** Dex projections chimiques, liquides, ou solides peuvent être dangereuses pour les yeux. Porter des lunettes de protection lors de toute manipulationde produit chimique ou pour toute intervention sur l'instrument.	**Peligro par los ojos:** Las salicaduras de productos químicos o particulas que salten bruscamente pueden causar lesiones en los ojos. Utilice anteojos protectores al mnipular productos químicos o al darle servicio de mantenimiento al instrumento.	**Pericolo per la vista.** Gli schizzi di prodotti chimici o delle particelle presenti nell'aria potrebbero causare danni alla vista. Indossare occhiali protettivi quando si maneggiano prodotti chimici o si effettuano interventi di manutenzione sull'apparecchio.
	**General Hazard:** A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.	**Allgemeine Gefahr:** Es besteht eine weitere Gefahr, die nicht in den vorstehenden Kategorien beschrieben ist. Dieses Symbol wird im Handbuch auBerdem dazu verwendet, um den Benutzer auf Anweisungen hinzuweisen.	**Danger général:** Indique la présence d;un risque n'appartenant pas aux catégories citées plus haut. Ce symbole figure également sur l'instrument pour renvoyer l'utilisateur aux instructions du présent manuel.	**Peligro general:** Significa que existe un peligro no incluido en las categorias anteriores. Este simbolo también se utiliza en el instrumento par referir al usuario a las instrucciones contenidas en este manual.	**Pericolo generico.** Pericolo non compreso tra le precedenti categorie. Questo simbolo è utilizzato inoltre sull'apparecchio per segnalare all'utente di consultare le istruzioni descritte nel presente manuale.
	When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.	Wenn Sie sich über die Sicherheit eines Verfahrens im unklaren sind, setzen Sie sich, bevor Sie fortfahren, mit Ihrer lokalen technischen Unterstützungsorganisation für Thermo Fisher Scientific San Jose Produkte in Verbindung.	Si la sûreté d'un procédure est incertaine, avant de continuer, contacter le plus proche Service Clientèle pour les produits de Thermo Fisher Scientific San Jose.	Cuando la certidumbre acerca de un procedimiento sea dudosa, antes de proseguir, pongase en contacto con la Oficina de Asistencia Tecnica local para los productos de Thermo Fisher Scientific San Jose.	Quando e in dubbio la misura di sicurezza per una procedura, prima di continuare, si prega di mettersi in contatto con il Servizio di Assistenza Tecnica locale per i prodotti di Thermo Fisher Scientific San Jose.

CAUTION Symbol	CAUTION	危険警告	危險警告
	**Electric Shock:** This instrument uses high voltages that can cause personal injury. Before servicing, shut down the instrument and disconnect the instrument from line power. Keep the top cover on while operating the instrument. Do not remove protective covers from PCBs.	**電撃：**この計測器は高電圧を使用し、人体に危害を与える可能性があります。保守・修理は、必ず操業を停止し、電源を切ってから実施して下さい。上部カバーを外したままで 計測器を使用しないで下さい。プリント配線板の保護カバーは外さないで下さい。	電擊：儀器設備使用會造成人身傷害的高伏電壓。在維修之前，必須先關儀器設備並切除電源。務必要在頂蓋蓋上的情況下操作儀器。請勿拆除PCB保護蓋。
	**Chemical:** This instrument might contain hazardous chemicals. Wear gloves when handling toxic, carcinogenic, mutagenic, or corrosive or irritant chemicals. Use approved containers and proper procedures to dispose waste oil.	**化学物質：**危険な化学物質が計測器中に存在している可能性があります。毒性、発がん性、突然変異性、腐食・刺激性などのある薬品を取り扱う際は、手袋を着用して下さい。廃油の処分には、規定の容器と手順を使用して下さい。	化學品：儀器設備中可能存在有危險性的化學物品。接觸毒性致癌、誘變或腐蝕／刺激性化學品時，請配帶手套。處置廢油時，請使用經過許可的容器和程序。
	**Heat:** Before servicing the instrument, allow any heated components to cool.	**熱：**熱くなった部品は冷えるのを待ってから保守・修理を行って下さい。	高溫：請先等高溫零件冷卻之後再進行維修。
	**Fire:** Use care when operating the system in the presence of flammable gases.	**火災：**可燃性のガスが存在する場所でシステムを操作する場合は、充分な注意を払って下さい。	火災：在有易燃氣體的場地操作該系統時，請務必小心謹慎。
	**Eye Hazard:** Eye damage could occur from splattered chemicals or flying particles. Wear safety glasses when handling chemicals or servicing the instrument.	**眼に対する危険：**化学物質や微粒子が飛散して眼を傷つける危険性があります。化学物質の取り扱い、あるいは計測器の保守・修理に際しては防護眼鏡を着用して下さい。	眼睛傷害危險：飛濺的化學品或顆粒可能造成眼睛傷害。處理化學品或維儀器設備時請佩戴安全眼鏡。
	**General Hazard:** A hazard is present that is not included in the above categories. Also, this symbol appears on the instrument to refer the user to instructions in this manual.	**一般的な危険：**この標識は上記以外のタイプの危険が存在することを示します。また、計測器にこの標識がついている場合は、本マニュアル中の指示を参照して下さい。	一般性危險：說明未包括在上述類別中的其他危險。此外，儀器設備上使用這個標誌，以指示用戶本使用手冊中的說明。
	When the safety of a procedure is questionable, contact your local Technical Support organization for Thermo Fisher Scientific San Jose Products.	**安全を確保する手順がよくわからない時は、作業を一時中止し、お近くのサーモエレクトロンサンローゼプロダクトのテクニカールサポートセンターごご連絡ください。**	如对安全程序有疑问，请在操作之前与当地的菲尼根技术服务中心联系。

Contents

Preface

About This Guide

This guide describes how to install and maintain the SpectraSYSTEM P4000 gradient pump, as well as how to control the gradient pump from the front panel keypad.

Safety and Special Notices

Caution!

A caution alerts you to situations that could result in personal injury. It also tells you how to avoid them.

High Voltage!

A high voltage caution alerts you to the presence of high voltage and to the potential injury that could occur from electrical shock were you to come in contact with a specific instrument area or component. It also tells you how to avoid contact with the high-voltage areas in your instrument.

Hot Surface!

A hot surface caution alerts you to potential injury that could occur from coming in contact with a heated surface or area on or in an instrument. It also tells you how to avoid contact with the heated surfaces in your instrument.

Note

Notes alert you to the correct operating or maintenance procedures needed to prevent equipment or data damage. They also alert you to important exceptions, side effects, or unexpected occurrences that may result from certain action(s).

Hint

Hints call out general rules or shortcuts. They specify ways to obtain the best performance and results from your instrument.

Manual Conventions

This manual uses several conventions. Among them are menu displays, text conventions (brackets, slashes, and so on), and standard words.

Displays

We will depict the two-line display as shown below. Note that in menu illustrations, the triangular cursor location is indicated by a caret (>).

```
>FILES              QUEUE                    TESTS
          COMMANDS              OPTIONS
```

A two-line menu display

Frequently the two lines shown on the display are only part of a longer menu which you would see by pressing the down-arrow key. In this manual, menus having more than two lines are represented as follows:

```
Edit File                        1
File Name
------------------------------------------------------------
>Solvent Program
Options
Timed Events
```

A menu longer than two lines

Text Conventions

Three typographic conventions are used to differentiate between keys, menus, and fields.

Brackets

Brackets, [ ], indicate instrument keys, For example: Press the [MENU] key.

Slashes

Slashes, / /, are text conventions used around menu choices. For example: From the Main Menu, select /FILES/, /Edit/.

Capitalization

Capitalization is used to make field and menu names appear just as they do on the display. Generally the first letters of field names are capitalized. For example: In /Solvent Program/ go to the Flow field.

Standard Words

We have also standardized the meanings of two words: “select” and “enter.”

Select

The word “select” is used when you need to choose from among available options. For example, to select a particular menu choice, you would move the cursor to the appropriate choice and press [ENTER]. To “select” a field entry, move the cursor to the appropriate field and use the [+] and [–] keys to scroll to the desired choice.

Enter

The word “enter” is used when you need to specify individual alphanumeric digits. To “enter” a particular value, move the cursor to the field and use the [+] and [–] keys to increment or decrement each digit in the field until the desired value or letter appears.

Good Laboratory Practices

To obtain optimal performance from your LC system and to prevent personal injury or injury to the environment, do the following:

- Keep good records
- Read the manufacturers’ Material Safety Data Sheets for the chemicals being used in your laboratory
- Remove particulate matter from your samples before you inject them into the liquid chromatograph
- Use HPLC grade solvents
- Connect the drainage tubes from the pump, autosampler, and detector to an appropriate waste receptacle. Dispose of solvents as specified by local regulations

Keep Good Records

To help identify and isolate problems with either your equipment or your methodology, keep good records of all system conditions (for example,% RSDs on retention times and peak areas, peak shape and resolution). At a minimum, keep a chromatogram of a typical sample and standard mixture, well documented with system conditions, for future reference. Careful comparison of retention times, peak shapes, peak sensitivity, and baseline noise can provide valuable clues to identifying and solving future problems.

Chemical Toxicity

Although the large volume of toxic and flammable solvents used and stored in laboratories can be quite dangerous, do not ignore the potential hazards posed by your samples. Take special care to read and follow all precautions that ensure proper ventilation, storage, handling, and disposal of both solvents and samples. Become familiar with the toxicity data and potential hazards associated with all chemicals by referring to the manufacturers' Material Safety Data Sheets (MSDS).

Sample Preparation

Always consider the solubility of your sample in the solvent/mobile phase. Sample precipitation can plug the column, tubing or flowcell causing flow restriction. This obstruction can result in irreparable damage to the system. To avoid damage caused by particulate matter, filter samples through 0.45 or 0.2 micron (or less) filters.

Solvent Requirements

Many chemical manufacturers provide a line of high-purity or HPLC-grade reagents that are free of chemical impurities. Routine filtration of all solvents or eluents through a 0.45 or 0.2 micron (or less) fluorocarbon filter before placing them in the solvent reservoir significantly prolongs the life and effectiveness of the inlet filters, check valves and seals, injector, and column. Typically, HPLC-grade solvents do not require filtration.

Choose a mobile phase that is compatible with the sample and column you have selected for your separation. Remember that some solvents are corrosive to stainless steel.

Solvent Disposal

Make sure you have a solvent waste container or other kind of drain system available at or below the benchtop level. Most solvents have special disposal requirements and should not be disposed of directly down a drain. Follow all governmental regulations when disposing of any chemical.

High-pressure Systems and Leaks

LC systems operate at high pressures. Because liquids are not highly compressible they do not store much energy. Accordingly, there is little immediate danger from the high pressures in an LC system. However, if a leak occurs, correct it as soon as possible. Always wear eye and skin protection when operating or maintaining an LC system. Always shut down the system and return it to atmospheric pressure before attempting any maintenance.

Contacting Us

There are several ways to contact Thermo Fisher Scientific for the information you need.

❖ To contact Technical Support

Phone	800-532-4752
Fax	561-688-8736
E-mail	us.techsupport.analyze@thermofisher.com
Knowledge base	www.thermokb.com

Find software updates and utilities to download at mssupport.thermo.com.

❖ To contact Customer Service for ordering information

Phone	800-532-4752
Fax	561-688-8731
E-mail	us.customer-support.analyze@thermofisher.com
Web site	www.thermo.com/ms

❖ To copy manuals from the Internet

Go to mssupport.thermo.com and click Customer Manuals in the left margin of the window.

❖ To suggest changes to documentation or to Help

- Fill out a reader survey online at www.thermo.com/lcms-techpubs.
- Send an e-mail message to the Technical Publications Editor at techpubs-lcms@thermofisher.com.

1

Installation and Specifications

Introduction

This chapter contains information necessary to install your Thermo Scientific, SpectraSYSTEM P4000 pump. The step-by-step instructions describe how to set the voltage, how to connect tubing, and how to prime and purge the pump. Use the checklist on the next page to complete pump installation. Also, be sure you read the Safety Information at the front of this manual before proceeding with any installation. If you have any questions or need further assistance, refer to the Preface for the product support or technical assistance numbers.

Start-up Checklist

This list is a brief summary of tasks that should be completed to install your pump. Complete installation information is contained in this chapter.

- ❒ Inspect your instrument
- ❒ Check for parts shortages
- ❒ Set the voltage
- ❒ Place the pump
- ❒ Connect the power cord
- ❒ Check initial response to power-on
- ❒ Hardwire to eight-pin port, using external function connector, making electrical connection to other SpectraSYSTEM instruments
- ❒ Install kits or accessories
- ❒ Prepare and connect solvents
- ❒ Connect inlet lines
- ❒ Prime with solvent
- ❒ Purge solvent lines
- ❒ Connect to system

This pump was installed by:

_____ _____

(Name) *(Date)*

Unpacking

INSPECT YOUR INSTRUMENT

Your pump was shipped in a special container designed to provide excellent protection from routine wear and tear encountered in transit.

After unpacking, inspect your pump and its accessories for missing parts and/or physical damage. If damage is found, notify both the carrier and your sales representative. DO NOT return any goods without prior authorization from Thermo Fisher Scientific.

The contents of your ship kit is as follows:

1	Pump
1	Accessory Kit (A4070-010)
1	Tubing Kit (See Appendix C)
1	Declaration of Conformity
1	SpectraSYSTEM documentation CD

OPTIONS AVAILABLE

A variety of options, kits, and accessories is available for your pump. Refer to Appendix C for a description and parts list for each. If you purchased an inert/biocompatible pump, the correct tubing and liquid ends were installed at the factory before shipment. For a list of all available accessories, upgrades, and kits, contact your Thermo Fisher Scientific sales representative. Note that all upgrades require installation by Thermo Fisher Scientific.

NOTE: The pump features a bypass valve pre-installed as standard equipment.

Figure 1.1 The SpectraSYSTEM P4000 pump

Installation

LIFTING AND CARRYING THE PUMP

The correct way to carry the pump is to use the two hand holds, one located underneath the front of the pump, and the other at the top of the back, near the power switch. Grasp the pump well underneath the front when lifting and carrying.

SETTING THE VOLTAGE

All pumps are configured at shipment for 230 VAC (50/60 Hz) operation. Depending upon the country of use, you might need to change the voltage setting.

NOTE: Check the position of the voltage select barrel located on the rear of the instrument. If the indicated voltage setting is not consistent with your area, DO NOT CONNECT THE POWER CORD!

Figure 1.2 shows the pump rear panel.

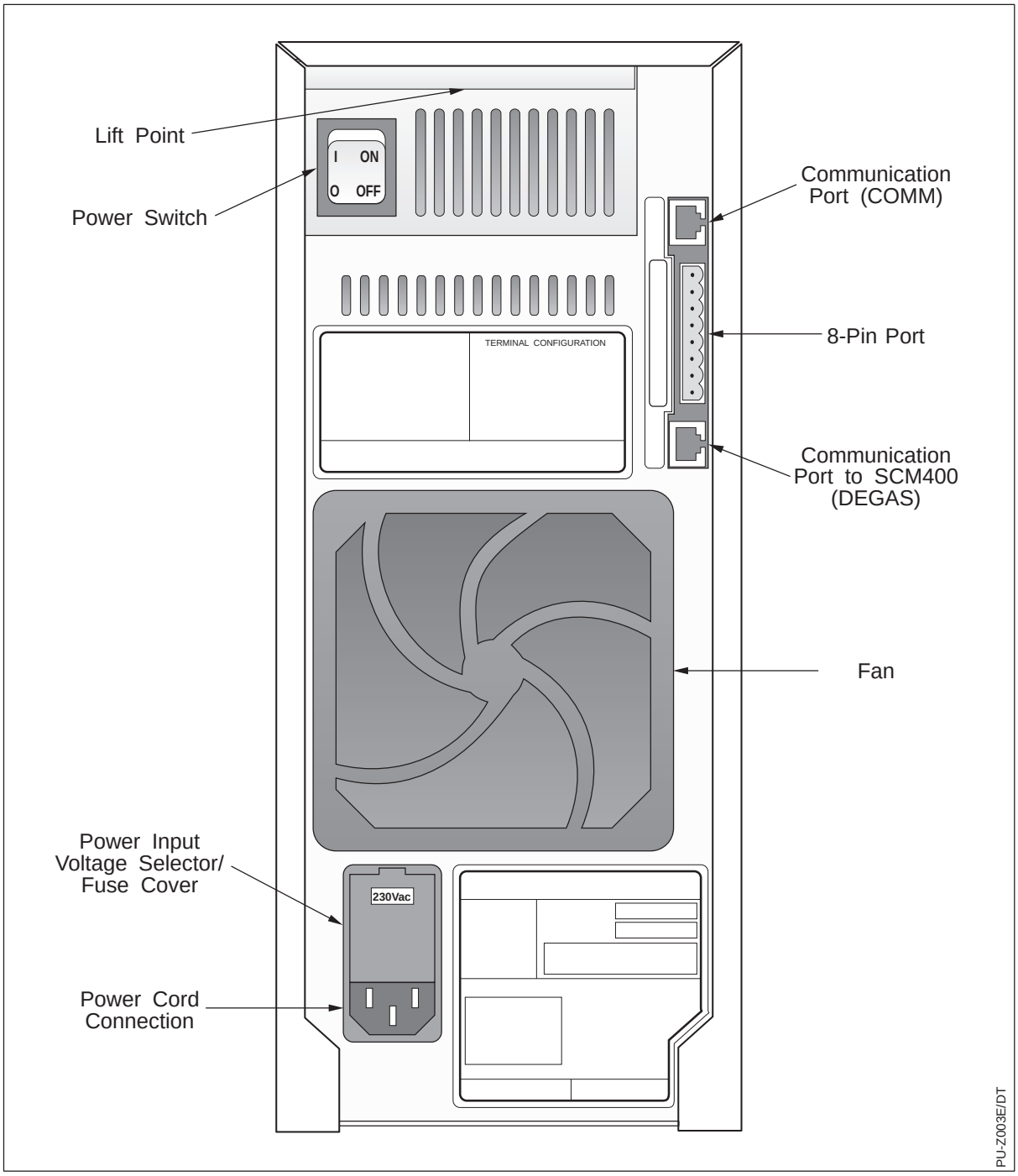


Figure 1.2 Rear panel

The voltage setting can be easily modified as follows:

1. Remove the tape label covering the power entry receptacle.
2. Ensure that the power cord is <u>not</u> connected to the pump.
3. Use a small, flat blade screwdriver to pry open the power selector/fuse cover to expose the voltage selector barrel. You will probably hear the top edge of the cover snap as it is pried open (Figure 1.3).

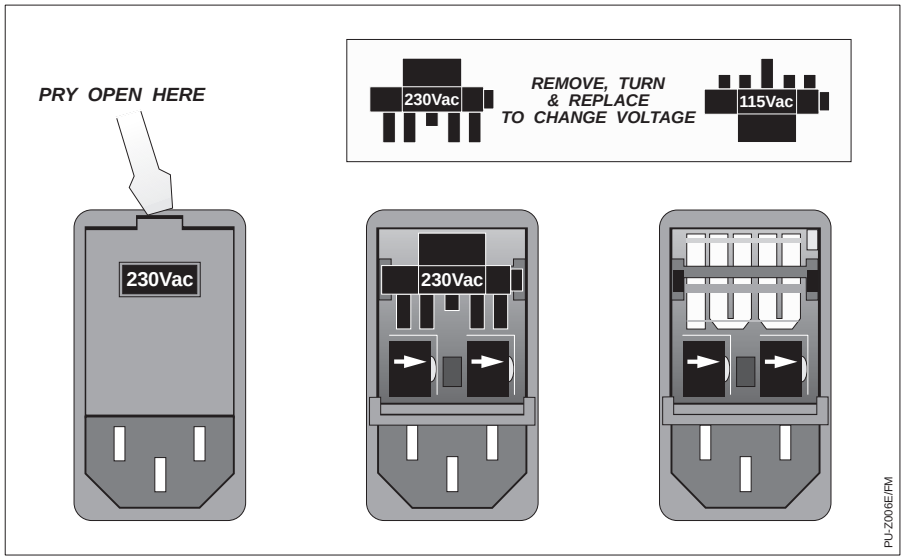


Figure 1.3 Power selector/fuse cover, closed, with "voltage window." The opened power selector/fuse cover, with barrel selector removed.

4. Remove the plastic selector barrel by pulling it straight out.
5. Rotate the barrel until you see the desired voltage (either 115 VAC or 230 VAC) and insert the barrel back into the housing with the desired voltage visible.
6. Firmly snap the housing cover back in place. Be sure that the selected voltage is visible in the voltage window.

HINT: Use two thumbs to push up on the top half of the cover as you push in. The voltage selected will be visible through the window.

PLACING THE PUMP

The pump weighs approximately 38 pounds (18 kg) and requires at least 6 inches (16 cm) of bench width and at least 19 inches (48 cm) of bench depth. If used with a manual injector bracket, the pump requires 9 inches (23 cm) of bench width. The pump needs a space at least 15 inches (38 cm) high.

Place the pump on a level surface. Leave 2 inches (6 - 7 cm) behind the instrument for good air flow and access to electrical connections. Keep the pump away from heating and cooling ducts, and avoid exposing the pump to direct sunlight. The pump should be placed to the far left of your LC system if it is used with a SpectraSYSTEM autosampler or detector.

CONNECTING THE POWER CORD

Attach the AC power cord (Figure 1.3). Plug the power connector into an appropriately grounded power outlet.

NOTE: For safe operation and optimum performance, the pump must be connected to a properly grounded power receptacle.

CHECKING INITIAL RESPONSE TO POWER ON

Turn the power on by pressing the power switch (Figure 1.2). With the pump's front panel facing you, the power switch is located in the back, on the upper right-hand side. The fan starts and the display shown in Figure 1.4 appears for one second.

```
Version  (X.YY.ZZ)
```

Figure 1.4 Brief power-up message

If this message does not appear, double-check the electrical connections and try turning on the pump once more, watching the screen closely. If the message still does not appear, contact your Thermo Fisher Scientific representative.

Next, the display shows the Status Screen, similar to Figure 1.5.

```
Status          Flow          PSI          MaxP
  STOP          1.00          154          3000♦
```

Figure 1.5 4000 Status Screen

HARDWIRING EXTERNAL EVENTS

Pin 6 on the eight-pin port on the back of the pump (Table 1.1) allows you to control another device, such as a column switching valve or fraction collector. If you plan to control such a device or instrument using the pump's Timed Events feature, insert the external function connector into the eight-pin port.

The pins are labeled both on the port and on the external function connector. Ensure that the pin numbers match whenever plugging into the connector to the port.

Hardwire your device using the 4-connection cable. Loosen pin 6's small screw, insert the wire, then tighten the screw.

You must also insert the external function connector if you use a SpectraSYSTEM autosampler. More information about making hardwire connections to an autosampler is found on page 17, and in the *SpectraSYSTEM Autosampler User Guide*.

Pin assignments for the eight-pin port are shown in Table 1.1:

Table 1.1 Eight-pin port assignments

Pin #	Description
1	READY (Output)
2	+5 VDC 100 mA MAX
3	GROUND
4	PRESSURE 0.1 V/1000 PSI
5	STOP (Input)
6	TIMED EVENT (Output)
7	RUN (Input)
8	INJ HOLD (Output)

All outputs (pin 1, pin 6, and pin 8) are open-collector type, capable of sinking up to 30 mA at a maximum of 30 VDC.

INSTALLING KITS OR ACCESSORIES

Refer to Appendix C for complete instructions for installing the Manual Injection Valve Bracket (column holder).

PREPARING AND CONNECTING SOLVENTS

If you did not purchase a solvent degassing apparatus or solvent bottles from Thermo Fisher Scientific, skip this section. Continue with *Connecting Inlet Lines* on the next page.

Solvent Bottles

Prepare your solvent bottles by following the steps below.

1. Rinse the bottles with LC-grade solvent to remove any dust.
2. Fill the bottles with appropriate LC-grade solvents.
3. The bottle caps are pre-assembled to include an inlet line and filter. Ensure that the filters are tightly assembled to their fittings, and the filter fittings are firmly attached to the inlet lines. Place the solvent filter/inlet line into each bottle, making sure that the inlet filter rests on the bottom of the bottle. Cap the bottle.
4. Attach the appropriate A, B, C, or D label to each solvent bottle cap to identify it.
5. Run vent lines from each bottle to an appropriate exhaust apparatus.

Degassing

There are two recommended methods for degassing solvents for use with your pump: vacuum degassing and helium degassing.

NOTE: Solvent degassing is required when proportioning (blending) solvent using your SpectraSYSTEM gradient pump. Degassing is not required for isocratic, premixed pump operation, but is recommended because of improved detector performance.

If you purchased a Thermo Scientific degasser, set up your degas apparatus as described in the degasser kit and continue the pump installation when you have a supply of degassed solvent available.

CONNECTING INLET LINES

Refer to Figure 1.6 to make the plumbing connections.

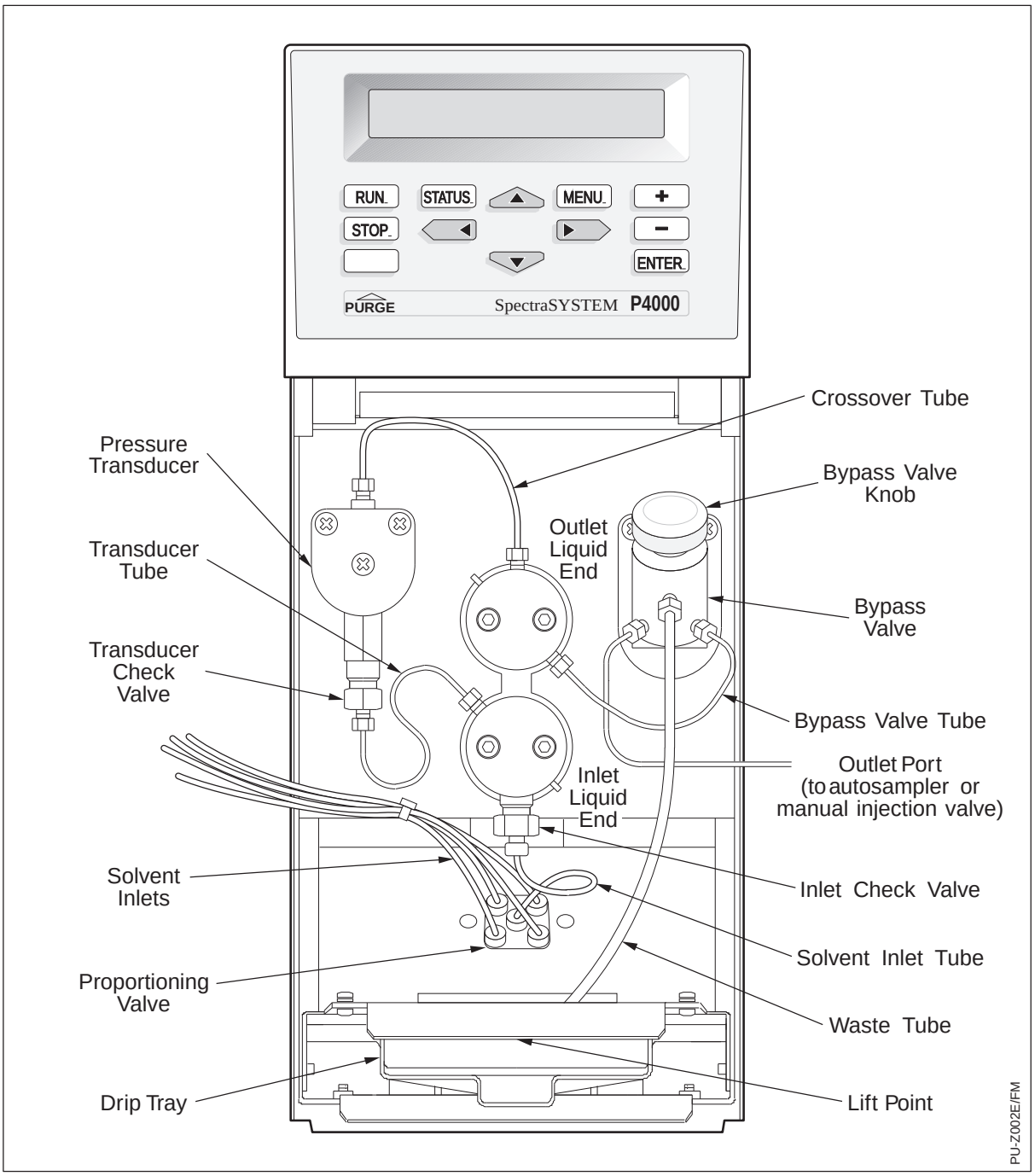


Figure 1.6 Front panel, cover removed

BYPASS VALVE

The bypass valve is shown in Figure 1.7. Do not attach a line to the waste/prime port (the middle port) now, but have the solvent waste tube (provided in the accessory kit) ready. You will attach the tube to the waste/prime port after priming the pump.

PRIMING AND PURGING

Each pump is shipped with methanol in the pump heads and connecting tubing. If the first solvent you'll use is not miscible with methanol, the pump must first be primed with an intermediary solvent. Once primed, you should purge the pump to remove any air bubbles.

HINT: It is best to prime the pump initially with methanol to fully wet all internal surfaces. Priming with 100% water can often result in trapped air due to the high surface tension of the water. Trapped air affects flow stability.

Priming the Pump

To prime the pump with your solvent and simultaneously flush the methanol out, you will need the 20 mL priming syringe and Luer adapter found in the accessory kit. You will also need a solvent waste container.

1. Remove the waste line from the waste/prime port of the bypass valve, if connected.
2. Install the Luer adapter to the waste/prime port (Figure 1.7). Tighten to finger-tight, then use a wrench to tighten approximately 1/4-turn. (This allows the priming syringe to be attached and detached conveniently.)

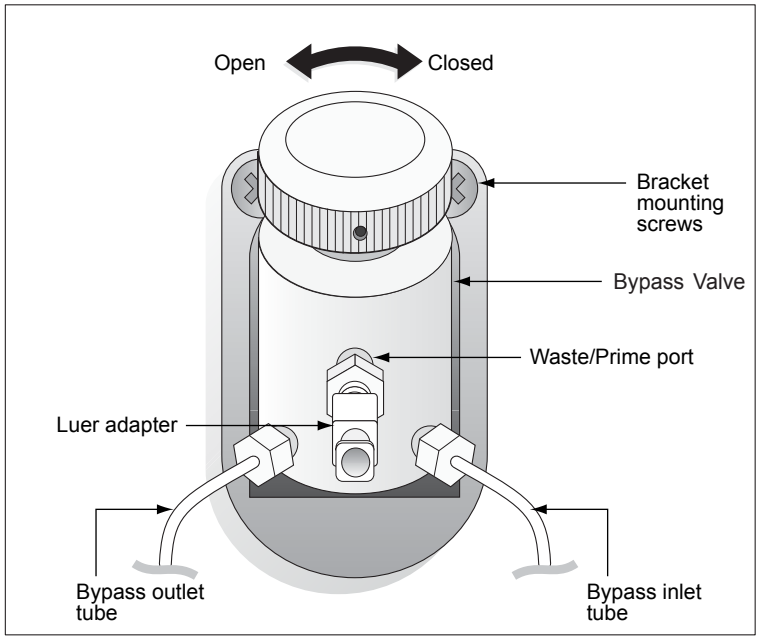


Figure 1.7 Bypass valve with Luer adapter connected

3. Make sure that the 20 mL Luer-tip priming syringe is fully depressed. Connect the syringe to the adapter in the waste/prime port (Figure 1.8) twisting the syringe slightly to make a leak-free connection.
4. Position a solvent waste container nearby to collect the syringe discharge, since two or three syringe volumes may be needed to prime the pump. Make sure that all tube connections are air-tight.

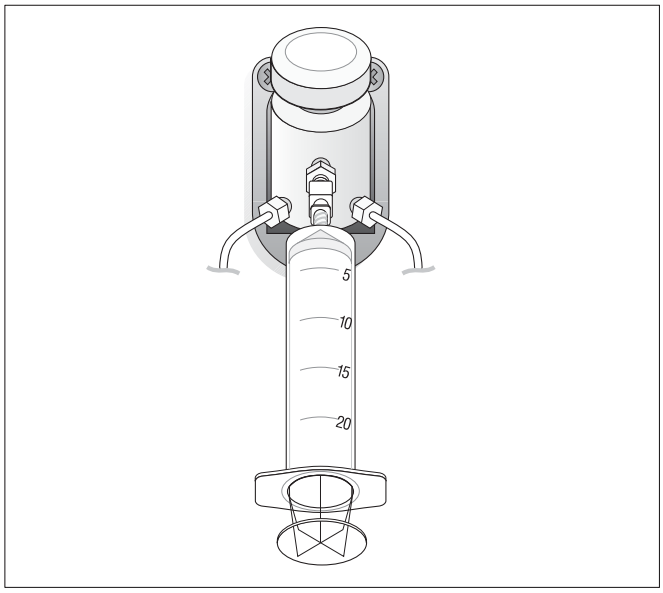


Figure 1.8 Bypass valve with syringe attached

5. Open the bypass valve by turning the knob fully counterclockwise.

NOTE: Solvents flow through the pump when the pump is purging or when a file is initialized (loaded as a run file).

6. Turn on power to the pump (if it not already on) and press [PURGE]. The cursor should appear under the word Purge on the display. Press [+] until the word ALL appears on the P4000.

NOTE: Do not purge ALL or BOTH unless all solvent lines are connected, and contain solvent. If this is not the case, select the appropriate solvent (A, B, C, D), instead.

NOTE: Purging starts whenever the cursor is moved out of the Time field using the [ENTER] key.

Purge	Flow	Time
ALL	1.00	0.0

Figure 1.9 Purge Menu (P4000)

NOTE: The Flow field may be labeled Pres instead of Flow, depending on the purge mode that is set. The purge mode can be changed from the [MENU], /OPTIONS/, /More/, Purge Mode field.

7. *For analytical applications:* Move the cursor to the Flow field. Set the flow rate to 10 mL/min. If the purge mode is pressure, set the pressure to 1000 psi (69 bars, 7 MPa).

   For narrow-bore applications: Move the cursor to the Flow field. If the purge mode is pressure, set the pressure to 1000 psi (69 bars, 7 MPa).

8. Move the cursor to the Time field, then press [ENTER]. You will hear the pump's motor start.

9. Slowly pull the syringe plunger back, thus creating a small vacuum in the solvent lines and drawing the solvent from the solvent bottles (or the vacuum degasser), into the pump heads. *Do not draw back so far as to remove the plunger from the syringe barrel!*

   If more than one draw is necessary to prime the pump, (*i.e.*, the syringe fills with air before solvent enters the pump heads), press [STOP], close the bypass valve (turn clockwise), remove the syringe, and depress the plunger. Reconnect the syringe, open the bypass valve, press [PURGE], enter a flow rate or time and press [ENTER] and finish drawing the solvents into the pump as described at the beginning of this step.

   If you notice a leak in one of the fittings, or need to stop the solvent flow, press [STOP]. This will immediately stop flow through the pump.

10. When solvent steadily appears in the syringe and no air bubbles are present, press [STOP], then close the bypass valve.
11. Gently remove the syringe and empty it into the solvent waste container.
12. Remove the Luer adapter from the valve port and store it, along with the priming syringe, for later use.
13. Connect the solvent waste line to the waste/prime port of the valve and tighten to finger-tight. Route the waste tubing to an appropriate solvent waste container.

Purging the Pump

With the pump primed, you now must purge the lines containing your chosen solvents.

1. Ensure that the solvent inlet filters inside each solvent bottle are in a vertical position so that air within the filter will not be trapped.
2. Fully open the bypass valve.
3. We suggest that you initially purge the lines with a volume of 10 mL.

   Follow the steps below to begin the purge operation:

   a) Press [PURGE] to reach the Purge Menu. If desired, change the purge mode by pressing [MENU] and selecting /OPTIONS/, /More/, Purge Mode. (The words in the top line of the display will change, depending on your purge mode preference.)

   b) Select "ALL" or the solvent of choice in the Purge field, then press [ENTER]. (See Figure 1.10).

   c) If the purge mode is Flow, do the following:

      For analytical applications: If the purge mode is Flow, enter a flow of 10.00 mL/min. [If pressure, set to 1000 psi (69 bars, 7 MPa).]

      For narrow-bore applications: If the purge mode is Flow, enter a flow of 5.00 mL/min. [If pressure, set to 1000 psi (69 bars, 7 MPa).]

   d) Press [ENTER] and enter a time of 1.00 minute. Ten milliliters should be enough volume to remove any trapped air and ensure that the pump and tubing are cleansed of any contaminants.

   e) Press [ENTER]. The pump's motor will start. Purging will automatically stop after one minute and will initialize the pump. After the pump has stopped, be sure to close the purge valve.

Purge	Flow	Time
ALL	10.00	1.00

Figure 1.10 Purge Menu with flow and time values entered

If you choose to purge without setting a time in the Time field, you can stop the purge by pressing [STOP]. This will immediately stop flow through the pump.

4. After you completed the purge, and the pump has stopped, close the bypass valve.

LC System Connections

Once the pump is purged, you can plumb it to the rest of your chromatographic system. Figure 1.7 and Figure 1.8 illustrate the bypass valve, showing the pump's outlet. Using a pre-cut piece of stainless steel tubing, connect the outlet to your autosampler or manual injection apparatus.

If you are using a manual injector valve from Thermo Fisher Scientific, Appendix C for complete information for installing the injector valve bracket.

If you are using a SpectraSYSTEM autosampler, the pump can send a ready signal to the autosampler through pin 1, and can receive a stop signal from the autosampler through pin 5. In addition, the ground contact (pin 3) must also be connected to the autosampler's ground contact. For complete information on how to make these connections, refer to the Chapter 1 of the *SpectraSYSTEM Autosamplers User Guide*. The table below summarizes the hardwire connections necessary between a SpectraSYSTEM autosampler and pump.

Table 1.2 Pump connections to SpectraSYSTEM autosampler

Pump		Autosampler
READY (Output)	Pin 1	Pin 5 *PUMP READY*
GROUND	Pin 3	Pin 1 *GROUND*
STOP (Input)	Pin 5	Pin 3 *PUMP STOP*
RUN (Input)	Pin 7	Pin 4 *GRAD START*
INJ HOLD (Output)	Pin 8	Pin 7 *INJ HOLD*

Performance Verification

After installing your SpectraSYSTEM pump, for best results, run a performance test to verify that the instrument is working properly. Common tests for pumps include flow accuracy and precision, gradient linearity, and compositional accuracy. Procedures for these performance tests are described below.

FLOW ACCURACY

There are many ways to test pump flow accuracy: graduated cylinder vs. time, calibrated flowmeter, or gravimetric vs. time. The procedure below describes how to measure the flow accuracy using a gravimetric procedure. In general you will set the pump to a flow rate, collect eluent for a specified time in a weighed flask, and determine the flow rate and accuracy. The actual flow rate can also be used to calibrate your pump. This test will take approximately 20 minutes.

Test Setup

Column or flow restrictor: To deliver 1000 psi backpressure

Flow rate: Any flow rate to be tested for accuracy

Mobile phase: MeOH or other appropriate mobile phase

Experimental

Using the following steps to calculate the flow rate accuracy:

1. Prepare the desired mobile phase for the accuracy test.
2. Filter and degas the mobile phase.
3. Purge the pump and the column or flow restrictor being used.
4. Set the pump at the desired flow rate and start.
5. Weigh a clean flask capable of holding approximately 10 minutes of volume at the set flow rate. Include top and any other items attached to the flask.

HINT: A volumetric flask is ideal because it helps to minimize evaporation. Also, wrap tube and top of flask with aluminum foil to help eliminate evaporation.

6. Record this weight.
7. Place the flask under the flow stream from the pump and start timing.

HINT: Use a stopwatch for the timing portion of the test. The more accurate the stopwatch the better the results.

8. Collect the pump's eluent for 20 minutes.
9. Reweigh the flask and record.
10. Calculate the actual flow rate:

$$[(Weight_{full} - Weight_{empty}) / Density] / Time = Flow\ Rate$$

11. Set a flow rate of 0.5 mL/min using MeOH as the mobile phase.

Flow Rate = [(22.8577 g - 14.8858 g) / 0.7894 g/mL] / 19.9687 min

Flow Rate = [(7.9719 g) / 0.7894 g/mL] / 19.9687 min

Flow Rate = 10.0987 mL / 19.9687 min

Flow Rate = 0.506 mL / min

12. Calculate the flow accuracy of the pump:

$$\text{Flow Accuracy} = 100 \times \left| (FR_{set} - FR_{actual}) / FR_{set} \right|$$

$$\text{Flow Accuracy} = 100 \times 0.006\ \text{mL / min}$$

Example

Using the data from step 10 gives the following results:

$$\text{Flow Accuracy} = 100 \times \left| (0.5\ \text{mL/min} - 0.506\ \text{mL/min}) / 0.5\ \text{mL/min} \right|$$

$$\text{Flow Accuracy} = 100 \times (0.006\ \text{mL} / 0.5\ \text{mL/min})$$

Flow Accuracy = 1.2%

HINT: For flow rates above 0.5 mL/min, it is best to install an in-line 100 µL mixer replacing the 3 µL mixer.

Specification

Typical flow accuracy results are less than 1.5% (absolute) using this gravimetric procedure in the 100 µL/min to 2 mL/min flow rate range.

The factory specification is less than 1% (absolute) using a calibrated flowmeter and methanol as the mobile phase.

FLOW PRECISION

Flow precision checks the reproducibility of the flow rate. This procedure is simply running the accuracy test 7 or more times and calculating a percent relative standard deviation. This procedure will require at least a couple of hours.

Test Setup

Set up the flow precision test above to perform 7 or more replicate runs.

Column or flow restrictor: To deliver 1000 psi back pressure

Flow rate: Any flow rate that accuracy is to be tested

Mobile phase: MeOH or other appropriate mobile phase

HINT: For flow rates above 0.5 mL/min, it is best to install a 100 µL mixer in-line replacing the 3 µL mixer.

Experimental

1. Repeat the flow accuracy test above for 7 or more replicate runs.
2. Determine the flow rate for each replicate.
3. Determine the average flow rate for the replicate runs.
4. Determine the standard deviation for the replicate runs.
5. Determine the relative standard deviation for the replicate runs.

Using the following example values:

Average = 0.502 mL/min

Standard Deviation = 0.004

Relative Standard Deviation (RSD) = 100 × SD/Average

Yields:

RSD = 100 × (0.004/0.502 mL/min)

RSD = 100 × 0.00797

RSD = 0.797%

Specification

Typical results for flow precision are less than 1% (absolute). However, results can vary based on temperature, mobile phase, and flow rate.

GRADIENT PERFORMANCE

Gradient performance is measured by testing gradient linearity and step/compositional accuracy. This procedure consists of running a pump method, which establishes a baseline, runs a linear gradient, and then runs 20% steps. This procedure can be run on a single pair of pump solenoid/switching valves or both pairs for quaternary gradient pumps. Each pair of valves will take approximately 50 minutes.

Depending data system, you will need a way to record the absorbance levels throughout the gradient run. With a computer data system or strip chart recorder this is automatically done for you. However, with an integrator this may require some manual recording of these levels.

HINT: The levels of importance are 0% spiked mobile phase, 100% spiked mobile phase, and each of the 20% steps.

Test Setup

Flow Restrictor:	Deliver 1,000 to 2,000 psi
Mobile phases:	A and/or C: Methanol
	B and/or D: Spiked Methanol with 7 ppm Ethyl Paraben
Flow rate:	0.5 mL/min (or other desired flow rate)
Detection:	UV at 254 nm

Experimental

1. Prepare mobile phases, filter through a 0.45-micron filter, and degas.
2. Purge pump lines with the appropriate mobile phase.
3. Purge entire system with methanol.
4. Setup the following gradient method for the pump:

NOTE: This procedure is for a quaternary pump. For a binary pump simply eliminate the gradient profile for C & D.

5. Run the gradient profile of step 4.
6. Record the 0%, 100%, and each 20% absorbance level of the spiked methanol.
6. Subtract the 0% level from all other recorded levels.
7. Determine 1% absorbance level of spiked methanol from the recorded 100% level.
8. Record each of the 20% absorbance levels of spiked methanol.

HINT: Record data after the particular step has stabilized.

Specification

The factory specification is that the linear gradient falls within ±1%. Each step must fall within ±1% of it's respective theoretical value.

Pump: P4000 Quat Gradient
Minimum Pressure: 0 PSI
Maximum Pressure: 6000 PSI
Delay Volume: 0.00 mL
Solvent Profile: Linear
Equilibration Time: 0.00 minutes

Time	A(%A)	B(%B)	C(%C)	D(%D)	Flow
0.00	100.0	0.0	0.0	0.0	0.50
5.00	100.0	0.0	0.0	0.0	0.50
20.00	0.0	100.0	0.0	0.0	0.50
25.00	0.0	100.0	0.0	0.0	0.50
25.10	20.0	80.0	0.0	0.0	0.50
30.00	20.0	80.0	0.0	0.0	0.50
30.10	40.0	60.0	0.0	0.0	0.50
35.00	40.0	60.0	0.0	0.0	0.50
35.10	60.0	40.0	0.0	0.0	0.50
40.00	60.0	40.0	0.0	0.0	0.50
40.10	80.0	20.0	0.0	0.0	0.50
45.00	80.0	20.0	0.0	0.0	0.50
45.10	100.0	0.0	0.0	0.0	0.50
50.00	100.0	0.0	0.0	0.0	0.50
50.10	0.0	0.0	100.0	0.0	0.50
55.00	0.0	0.0	100.0	0.0	0.50
70.00	0.0	0.0	0.0	100.0	0.50
75.00	0.0	0.0	0.0	100.0	0.50
75.10	0.0	0.0	20.0	80.0	0.50
80.00	0.0	0.0	20.0	80.0	0.50
80.10	0.0	0.0	40.0	60.0	0.50
85.00	0.0	0.0	40.0	60.0	0.50
85.10	0.0	0.0	60.0	40.0	0.50
90.00	0.0	0.0	60.0	40.0	0.50
90.10	0.0	0.0	80.0	20.0	0.50
95.00	0.0	0.0	80.0	20.0	0.50
95.10	0.0	0.0	100.0	0.0	0.50
102.00	0.0	0.0	100.0	0.0	0.50

Injector: AS3000 Variable Vol
Injection Volume: 1.0 uL
Viscosity: Normal
Equilibration Time: 0.00 minutes
Needle Height: 2.0 mm
Injection Mode: PushLoop
Flush Volume: 400 uL
Oven Off
Tray Temperature Off

Time Function	Time	State
Time Function #1 (TF1)	0.00	Off
Time Function #2 (TF2)	0.00	Off
Time Function #3 (TF3)	0.00	Off

Figure 1.11 Gradient Profile

Specifications

P4000:	Gradient pump using a dual in-line and floating piston design, bayonet-mounted liquid ends, and patented ceramic check valves.
Physical:	14.5" (37 cm) × 6" (15 cm) × 18.5 (47 cm) (H × W × D) 38.6 lb. (18 kg)
Wetted Surfaces:	316 stainless steel, analytical and narrow-bore PEEK, semi-prep other surfaces for all pumps: Teflon®, Tefzel®, sapphire, polyethylene
Delay Volume:	< 800 µL, analytical < 500 µL, narrow-bore (P4000)
Flow Rate Range:	0.01 - 10.00 mL/min, analytical or narrow-bore pumps 0.01 - 30.00 mL/min, semi-prep PEEK pumps
Flow Accuracy:	< 1% at 1.0 and 4.0 mL/min, analytical or semi-prep < 1% at 0.2 and 1.0 mL/min, narrow-bore
Flow Precision:	Typically < 0.2% at 0.5 mL/min or greater
Gradient Linearity:	< 1.0% at 1.0 mL/min from 5 - 95% composition, analytical < 1.0% at 0.5 mL/min from 5 - 95% composition, narrow-bore
Compositional Accuracy:	< 1.0% at 1.0 mL/min from 5 - 95% , composition analytical < 1.0% at 0.5 mL/min from 5 - 95% composition, narrow-bore
Compositional Precision:	Typically < 1.0% at 1.0 mL/min analytical Typically < 0.2% at 0.3 mL/min, narrow-bore
Pressure Range:	42 MPa or 420 bar or 6000 psi, analytical or narrow-bore 28 MPa or 280 bar or 4000 psi, semi-prep
Pressure Pulsation:	Typically < 1.0% at 1 mL/min
Method Files:	9 method + 1 Shutdown + 1 Develop, P4000
Communications:	Remote Inputs: Ready, Run, Stop Timed Events Analog Pressure output RS-232 (P4000)
Environmental	10-40 °C 5-95% RH noncondensing
Power:	115/230 VAC, 50/60 Hz T4A 200VA
Safety/EMC Compliance:	CSA, TÜV, FCC, CE Mark, EMC and Low Voltage Directives

2

A Quick Example

Introduction

This chapter provides you with the concepts you'll need for using your gradient pump. It also introduces you to the instrument's pump's screens and menus. In this chapter you will set up a few typical options, purge your solvent lines, and run a flow stability test.

If you already feel comfortable with how to move through menus and displays, just scan this chapter and proceed to Chapter 3. If you want more practice with the pump, follow the instructions in this chapter closely. Since the object of this chapter is to become familiar with the keypad and menus, we won't provide detailed explanations of the examples shown. More information can be found in one of the succeeding chapters.

If you haven't installed your pump, be sure that you read the Safety Information section and follow the procedure in Chapter 1.

Throughout our explanations, we encourage you to explore the general architecture of the instrument's menus and screens. Use the menu trees in the front pocket of the manual as your guide.

Learning Your Way Around

AS EASY AS 1-2-3!

It's easy to learn your way around a SpectraSYSTEM pump. Just remember these three easy rules:

1. The ([∧], [∨], [<], [>]) move the cursor in the direction printed on the key.

   HINT: Press [MENU] to jump quickly to the top of the menu structure.

2. The shape of the cursor determines how you make a selection:
   - If a triangular cursor appears, press [ENTER].
   - If a blinking square cursor appears, press the [+] or [-] keys to scroll up or down through preset choices, or to increase or decrease alphanumeric entries

3. There are four ways to accept (and automatically save) an entry. Just move the cursor out of the field by any of the following methods:
    - Pressing [ENTER]
    - Using the arrow keys
    - Pressing [MENU]
    - Pressing [STATUS]

NOTE: You won't be able to leave a menu if errors are present or if you haven't filled in all the necessary entries.

Several visual clues help you move through the pump's menus and enter values.

1. Top-level menu choices are displayed in all-capital letters; lower-level menu choices are displayed in upper- and lower-case letters.
2. A field's square cursor changes to an underscore cursor when you're scrolling through preset choices or entering numerical values and characters.
3. A solid down-arrow (▼) on the right side of some displays indicates that the current menu continues on additional screens. To access additional menu lines, press the down-arrow key, [∨].
4. The last line of a longer menu is frequently a blank line (without a solid down-arrow).

Instrument Control

Take a look at the keypad and two-line display located on the front panel (Figure 2.1). This is the command center from which you'll access menus and control the instrument's operations. A brief explanation of the keys and the main menus and screens follows.

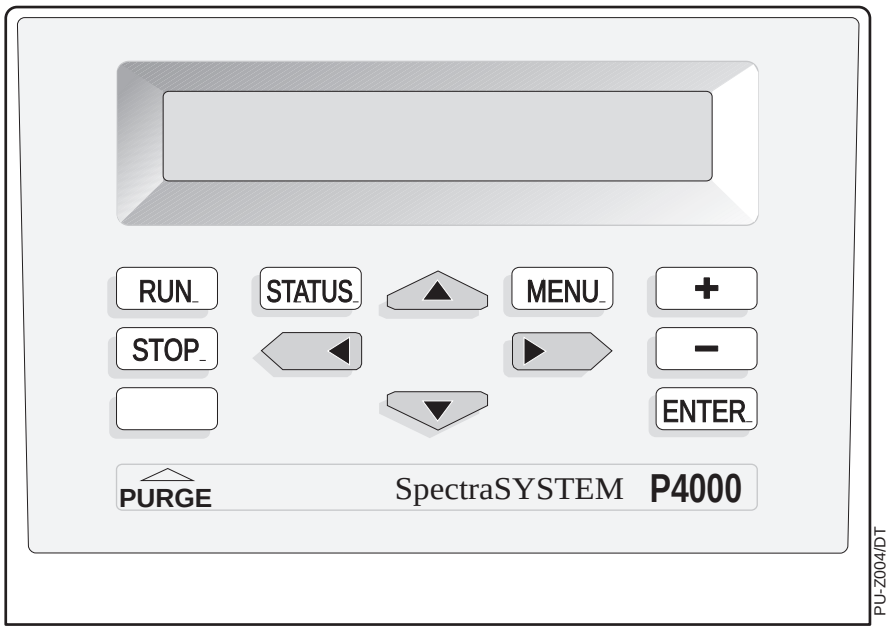


Figure 2.1 The P4000 pump's keypad.

The keypad of each SpectraSYSTEM instrument consists of twelve keys. Four keys directly control the instrument's operation: [RUN], [STOP], [STATUS], and, on the pump, a blank key called [PURGE]. The remaining keys ([MENU], [ENTER], [∧], [∨], [<], [>], [+], [-]) either access commands or are used to set parameters and move around the display. The function of each is explained below.

[RUN]

Generally, pressing [RUN] starts a run or sets up the conditions specified for the beginning of a run.

The specific [RUN] operation depends on the instrument's state:

1. If the pump's state is STOP, pressing [RUN] automatically prepares the last file loaded to be run and sets the pump to the conditions specified for the start of the run (t_0 conditions).
2. If the pump's state is READY, (that is, the initial conditions already exist), pressing [RUN] starts the run.

[STOP]

Pressing stop halts an operation in progress. (Specifically, the [STOP] key aborts a run in progress by stopping solvent flow through the pump.)

[STATUS]

Pressing [STATUS] displays the Status Screen (Figure 2.3). From the status screen you can monitor the run in progress. You can also access the Status Menu. See page 29 for more information.

[PURGE]

The unlabeled key is the only variable key in the whole SpectraSYSTEM family. On the pump, the blank key is the [PURGE] key. The key's name appears on the nameplate below the key.

The [PURGE] key brings the PURGE Screen to the display. Purge parameters can be changed, and the purge operation started from this display. Refer to *Purging Solvent Lines* in Chapter 1 and to *Priming and Purging the Pump* in Appendix A for complete information.

[MENU]

Pressing [MENU] displays the Main Menu (Figure 2.2). Each main menu item is explained in detail in the rest of this manual. For FILES and COMMANDS see Chapter 3, for QUEUE and OPTIONS and TESTS, see Chapter 4.

[ENTER]

Pressing [ENTER] accepts a selected choice or menu entry. The [ENTER] key also advances the cursor to a new field, either on the same line of the display or in the line below.

ARROWS

Pressing any arrow key (up, down, left, or right) moves the cursor in the direction indicated on the key. If the cursor is on the first or the last line of a menu, the up- and down-arrow keys move you "up" or "down" in the menu structure.

[+] and [-]

Pressing the [+] and [-] keys scrolls you through a field's available choices or changes the value of alphanumeric entries. Holding down either key will continuously scroll the list of choices forward or backward until you release the key.

In fields that require numeric entries, the value of each digit is increased or decreased by one unit each time you press the [+] or [-] key. In fields that accept *either* numeric or alphabetic entries, such as the File Name field, the [+] and [-] key scroll through the alphabet from A to Z, then through the numbers 0 to 9, and finally to a slash, hyphen and blank space.

In other fields, the [+] key advances you through a preset list of choices while the [-] key takes you back through the list.

MENUS AND SCREENS

Your pump has two kinds of displays: menus and screens. Menus require you to make selections or enter specific values. Screens display information that cannot be edited. The Menu Tree in the front pocket illustrates the structure and content of the pump's menus and screens.

Main Menu

The Main Menu (Figure 2.2) is the top level of the menu structure. It gives you access to five other menus: FILES, QUEUE, TESTS, COMMANDS, and OPTIONS. To see the Main Menu, press the [MENU] key at any time.

```
>FILES                ·QUEUE                ·TESTS
          ·COMMANDS             ·OPTIONS
```

Figure 2.2 Main Menu (P4000)

From the Files Menu you can edit, load, copy, or delete files. The Commands Menu lets you hold, continue, or reset the pump. In the Options Menu, you can set up or change your instrument's configuration. From the Queue Menu you can edit or change the order and number of files in the queue. Refer to Chapters 3 and 4, and Appendices B and C for more information on any of the instrument's menus.

Status Screen

The Status Screen (Figure 2.3) appears whenever you turn on the instrument or press the [STATUS] key. The P4000's Status Screen displays the pump's state, flow, current pressure, and the maximum pressure setting. Two additional lines show the current solvent composition and the elapsed time of a run. Below the Status Screen is the Status Menu, described next.

```
Status          Flow          PSI          MaxP
 READY          1.00           0           3000▼
```

Figure 2.3 The first two lines of the P4000 Status Screen

Status Menu

Just below the Status Screen is the Status Menu. To access the Status Menu, press the down-arrow key from the Status Screen. The Status Menu lets you review and edit run parameters during a run. Chapter 3 discusses the Status Menu in more detail.

MESSAGES

There are three different kinds of messages that can appear on the pump's display: user messages, confirmation messages, and error messages.

User messages

User messages (Figure 2.4) tell you about an existing instrument condition or ask for further action. Some of these will only appear on the display for three seconds. An example of a message requiring further action is shown in Figure 2.4.

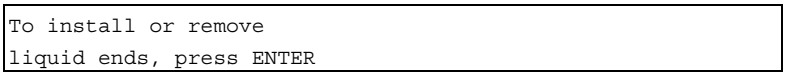


Figure 2.4 Example of a user message

Confirmation messages

Confirmation messages (Figure 2.5) indicated on the display by asterisks, appear for one second after an operation has been carried out successfully.

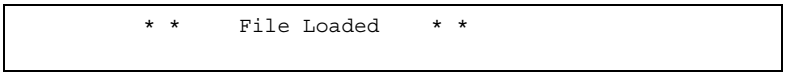


Figure 2.5 An example of a confirmation message

Error messages

Error messages (Figure 2.6), indicated on the display by exclamation points, are displayed whenever an undesirable condition exists that prevents the instrument from carrying out an operation. Error messages remain on the display until you press a key.

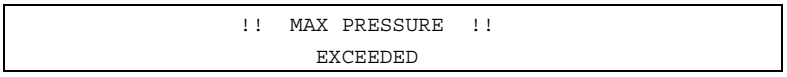


Figure 2.6 An example of an error message

Practice Examples

This section will take you step-by-step through four operations:

1. Relabeling two solvents so that the display shows H_2O and MeOH instead of A and B (P4000 only).
2. Changing pressure units.

3. Purging these two solvent lines.
4. Running a flow stability test.

These examples assume that the pump is properly installed and that the bypass valve's outlet is routed to a solvent waste container. [The solvents you actually use need not be water and methanol (MeOH), but the solvents connected to lines A and B must be miscible. If they are not, skip the example in Purging Lines A and B; it calls for mixing the two solvents which may damage the liquid ends.]

LABELING SOLVENTS

The pump display can be changed to suit your own needs. The solvent labels in the P4000 are a good example.

To change solvent labels:

1. Press [MENU].
2. Use the right-arrow key [>] to move the cursor to /OPTIONS/. Press [ENTER] to access the Options Menu.
3. The cursor is next to Solvent Selection. Press [ENTER] to select /Solvent Selection. A display similar to the one in Figure 2.7 should appear:

```
                    Select Solvents
[  A  ]          [  B  ]        [  C  ]        [  D  ]
```

Figure 2.7 Solvent Selection Menu

4. The cursor should be in the [A] field. Press [+] until H_2O appears. If you accidentally go past it, press [-] to go back.
5. Press [ENTER]. The cursor should now be in the [B] field.
6. Again, press [+] until MeOH appears. The display should now look like the one below:

```
                    Select Solvents
[ H2O ]          [ MeOH ]       [  C  ]        [  D  ]
```

Figure 2.8 Labels assigned to solvents A and B

You have just relabeled the two solvents A and B. Wherever a display would ordinarily show A and B, it will now show H2O and MeOH instead. C and D will remain unaffected. You will notice this as you continue with the examples.

CHANGING PRESSURE UNITS

To change pressure units on your display:

1. Press [MENU] and move the cursor to /OPTIONS/. Press [ENTER].
2. From the Options Menu, press [∨]. Select /More/, and press [ENTER]. The display now looks like Figure 2.9.

Pressure Units	PSIv
Purge Mode	Flow

Figure 2.9 The More Menu

3. The cursor should be in the Pressure Units field. Press [+] until the field shows MPa. You have just changed the display so that all pressures will be shown in megapascals. If you prefer other units, press [+] until you see another preset choice that suits you.
4. Move the cursor down to the Purge Mode field. Use [+] to see the Pressure selection (pressure). Press [+] once more, to change the purge mode selection back to Flow.
5. You may exit the More Menu in several ways. This time, press [STATUS] to exit the menu.
6. Use [∨] to look at the Status Menu, which shows the run file.

PURGING LINES A AND B

You might already be familiar with the purge operation from Chapter 1. If so, skip this example and go on to *Running a Flow Stability Test* page 34.

In this example you will purge two solvent lines simultaneously, using equal compositions.

1. Open the bypass valve on the pump. Make sure that the bypass valve outlet is routed to a solvent waste container.
2. Press [STATUS]. The Status Screen should show that the pump is stopped.
3. Notice that by pressing [∨] you can see the current solvent compositions, and the H_2O and MeOH labels you set earlier. Your Status Screen and Status Menu should be similar to Figure 2.10.

Status	Flow	MPa	MaxP
STOP	1.50	1	21

Stat	H_2O	MeOH	C	D
0.0	100.^{0}	0.^{0}	0.^{0}	0.^{0}

(the pressure units displayed may be different depending on your selection in the example above.)

Figure 2.10 Status Screen and Status Menu (P4000)

4. Leave the Status Screen and go to the Purge Menu by pressing [PURGE] (the blank key).
5. The cursor should be in the Purge field (Figure 2.11). Press [+] until the word BLEND appears.

Purge	Flow	Time
1 H_2O	1.50	0.0

Figure 2.11 Purge Menu (P4000)

6. Press the down-arrow key. The display will show four solvents (Figure 2.12).

H_2O	MeOH	C	D
100	0	0	0

Figure 2.12 Blend solvent compositions (P4000)

7. Enter 50 (percent) for the composition of the first solvent using the [-] key. Notice that the composition of the second solvent automatically changes so that the total remains 100%. The display now looks similar to Figure 2.13.

H_2O	MeOH	C	D
50	50	0	0

Figure 2.13 Equal compositions of two solvents (P4000)

8. Press [∧]. You will return to the top of the Purge Menu.
9. To begin the purge cycle, move the cursor to the Time field by pressing [ENTER] as many times as necessary. Enter 2.0 minutes in the Time field, then press [ENTER] again.

The pump will purge the A and B solvents for two minutes at the flow rate shown in the Flow field. You can stop the purge operation at any time by pressing [STOP]. After the purge is complete, the pump will automatically initialize the run file.

Remember to close the bypass valve immediately following the purge so that solvent flow returns to the LC.

RUNNING A FLOW STABILITY TEST

The flow stability test is a common test of the pump's performance. The pump must be in a READY, EQUIL, or RUN state for this test to be initiated, and the flow rate must be greater than 0 mL/min. If you need to change the flow rate, edit the flow setting from the Status Menu, then proceed with the steps below.

1. When the Status Screen shows READY, press [MENU] and select /TESTS/.
2. Select /Diagnostics/ from the Tests Menu.
3. The cursor should be on /Flow Stability/. Press [ENTER].

After a short time, the flow stability rating followed by a numerical value will appear. STABLE flow corresponds to a reading between 0 and 25, ACCEPTABLE is between 26 and 90, and UNSTABLE is a value greater than 90.

NOTE: The Flow Stability test may show unstable flow during gradient compositional changes or during column equilibration.

3

Basic Operations

Introduction

The type of chromatographic analyses you do will determine how you choose to use your pump. The P4000 is a gradient pump used for methods development, and characterized by automatically varying solvent compositions during a run. This chapter contains a pump theory of operation, some recommended LC pump practices, and describes how the pump is used to perform basic operations: editing, loading and running a file, purging the pump, and viewing the pump's status.

Theory of Operation

The pump is typically the second of six components in an LC system (solvent degasser is the first). A pump delivers a steady flow of one or more solvents to a sample-injection instrument (generally an autosampler). This solvent flow continues through the column and on to a detector. From the detector, a signal is passed to an integrator, a recorder or another kind of data system capable of collecting the data and allowing the data of the injected sample to be analyzed.

A gradient pump works by first pulling a filtered and degassed solvent into a proportioning valve. The P4000 pump has four proportioning valves. Solvents are measured by percentages, specified by you, and mixed inside the pump. The solvents then travel to the pump head where a piston meters the flow of the mixture to an outlet tube. The pump's outlet tubing then connects the solvent stream to an automatic or manual injector.

SpectraSYSTEM P4000 Pump

The SpectraSYSTEM P4000 pump has been designed for ease of use and unsurpassed performance. It can be used as a stand-alone pump or as a module in a totally automated LC system.

The P4000 pump provides low-pressure quaternary mixing for accurate proportioning of binary, ternary, or quaternary gradient mobile-phase compositions and solvent switching when used for isocratic applications. The P4000 pump contains as many as 210 lines in as many as 9 method files (40 lines per file maximum).

Table 3.1 File Characteristics for P4000 pump

File Characteristics	P4000
Time Lines/File	40
Total Number Files	9 + Shutdown + Develop
Total Time Lines	210 lines

The SpectraSYSTEM P4000 pump is engineered for reliability and ease of maintenance. Easy maintenance helps to ensure that your chromatography results are accurate and remain accurate. A built-in, patented Maintenance Log (Chapter 5) allows you to follow the life span and use of seals, pistons, and check valves. If service is ever required, the resident diagnostics and modular design of the pump will keep downtime to a minimum. The simplicity and durability of the pump means that a minimum of spare parts need to be kept on hand.

OPTIONS

Narrow-bore

LC refers to the use of narrow-ID, 2.0-3.0 mm columns for LC separations. To optimize instruments for narrow-bore LC, standard LC hardware must be modified to reduce extra-column volume and gradient delay. Specific hardware modifications include minimizing the pump's dead volume, the detector flowcell's volume, and the volume contained in any interconnecting tubing and fittings. The lower system volume of narrow-bore LC increases sample concentration, which results in greatly improved sensitivity. Decreased solvent consumption reduces operating costs as compared to standard (4.0-4.6 mm ID) LC separation techniques.

NOTE: For narrow-bore applications we recommend you use a 20 µL sample loop in Thermo Scientific autosamplers.

Narrow-bore Hardware Modifications

Pump: For the SpectraSYSTEM gradient pump, the following hardware changes have been made to create a compatible pump for narrow-bore applications.

- The solvent inlet line (from the gradient valve to the inlet check valve) is changed from 0.060-inch to 0.030-inch ID tubing and shortened from 12 cm to 10 cm.
- All interconnecting tubing has been changed from 0.020-inch ID to 0.010-inch ID.
- The outlet liquid end is changed from the standard to the low-volume design.

Autosampler: For the SpectraSYSTEM narrow-bore autosampler the pump-to-autosampler tubing (~ 6 inches long) and pre-heat tubing (~ 24 inches long) has been changed from 0.020-inch ID to 0.007-inch ID.

Operational Considerations

Injection Mode: We recommend that you consider the following in setting up your methods and LC instrumentation for most narrow-bore applications. Due to the manner in which the pump firmware stores certain values and references the hardware, the minimum operating pressure for these pumps is 200 psi.

We recommend that you use the PushLoop® mode for narrow-bore applications; however, depending on your sample volume, other injection modes may be more beneficial (see below) in some cases.

The following algorithms define the amount of sample needed for your injection volume:

PushLoop	(Injection Volume + 15 µL)
Pull Loop	(Injection Volume + 1.1 µL)
Full Loop	(Injection Volume x 1.33) + 70 µL

In PushLoop mode, the smallest setable sample volume is 0.1 µL. Allowed volume increments are also 0.1 µL. You should not inject more than 10 µL without changing the standard 20 µL loop. The autosampler is supplied with a 20 µL loop.

Reducing Extra Column Volume: Minimizing extra column volume maximizes analytical efficiency. Band-broadening can occur in several ways. With narrow-bore columns, it is vital that good plumbing connections are made. Otherwise, a dead volume created by a bad fitting can result in much lower than expected efficiency and peak resolution. Use zero dead volume (ZDV) fittings only with the proper nuts and ferrules. Cut tubing with the proper tools. If possible, electropolish metal tubing after cutting. For polymer tubing (*for example* PEEK), use a "guillotine" cutter to ensure straight, right-angle cuts.

Instrument Startup

Be sure you have installed the pump according to Chapter 1 and have completed the Start-up Checklist.

When you turn on the pump, the Status display appears. This allows you to check the instrument settings before entering your parameters. As described in Chapter 2, it is possible to edit a file under the Status Menu. If you have turned on the pump before, and have used this editing capability, the Status Menu will contain the parameters last saved before the pump was turned on.

At powerup, there are no messages to alert you that a file is being loaded, but you can check the file number and name by pressing the [STATUS] key. Then use the down-arrow key to scroll to the file listing. (Both the [STATUS] key and the creation of files are discussed later in this chapter.)

Some Routine Operations

Ordinarily, you will probably perform these operations with your pump every day:

- Edit a file (or create a new file) and/or load a file to run
- Run your samples
- Purge the solvent lines
- Check the pump's status
- Monitor pump performance
- Shut down the pump at the end of the day

The rest of this chapter is devoted to explaining these basic operations. If you need to, refer to the keypad "rules" in Chapter 1. You may also want to refer to the basic menu structure on the quick reference card in the front pocket of your manual.

The File(s) Menu

In this section we describe how to set up the files that control solvent composition, pressures, flow rates, and run times. Each of the file operations accessed from the Files Menu is briefly defined, then described in detail.

To access the File(s) Menu, press [MENU] and select /FILES/. The Files Menu is shown in Figure 3.1.

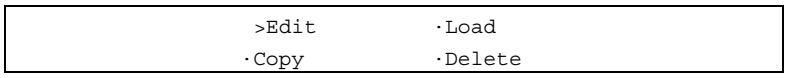


Figure 3.1 The Files Menu (P4000)

Edit

Select /Edit/ if you want to change the name and/or parameters in an existing file, or create a new file. Select the file by file number or letter (file S is the Shutdown file, file D is the Develop File™).

Load

Select /Load/ to load the file you want the pump to use when in operation. Make a file selection by file number. We refer to a loaded file as the run file.

Copy

Select /Copy/ to copy the contents of one file into another. Select both files by file number.

Delete

Select /Delete/ to delete the contents of a file and return all file parameters to their default values. An information message allows

you to cancel the delete operation before the file is deleted. Once deleted, the file cannot be retrieved.

EDITING A FILE

Each file consists of four parts: File Name, Solvent Program, Options, and Timed Events. Selecting /Edit/ gives you access to these areas (Figure 3.2).

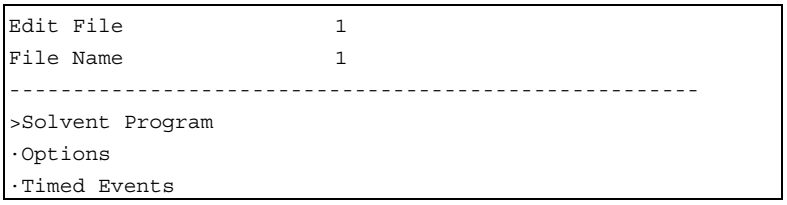


Figure 3.2 The Edit Menu

To use the Edit Menu, select the file number in the Edit File field using the [+]/[-] keys. The P4000 pump has file numbers 1 through 9. The P4000 pump has a Shutdown file (file number S), discussed on page 51. In addition, the P4000 has a Develop file (D) which is explained in detail in Chapter 4.

If a particular file cannot be accessed, the File Protect feature for that file has been turned on, preventing changes or deletions. When the File Protect feature has been turned on, a message appears on the display, indicating that the file cannot be edited. (File Protection is discussed in OPTIONS, More Menu in Chapter 4.)

File Name

You can enter or change the File Name using the [+]/[-] keys. The File Name field is eight characters long. Any name you enter is automatically remembered by the pump, so you do not need to formally "save" the name. All of the parameters discussed below are automatically remembered by the pump when you edit a file from the Files Menu.

NOTE: When changing the parameters of a run file from the Status Menu, you must select /Save File/ in order for the pump to remember your changes for future runs. The pump will, however, use your changes until the next time the file is re-initialized, regardless of whether the file has been saved. Editing a run file from the Status Menu is discussed in more detail later.

The Solvent Program Menu

The Solvent Program Menu contains time lines used to construct gradient or isocratic solvent parameters. Time lines consist of a time, a percentage composition (in whole and half percents in the P4000), and a flow rate (in mL/min). When /Solvent Program/ is selected, the display shows (Figure 3.3):

Time	A	B	C	D	Flow
0.0	100	0.0	0.0	0.0	1.00

Figure 3.3 A P4000 solvent program

NOTE: If you used the Solvent Selection Menu (P4000) to label solvents as described in Chapter 2, the display will show the assigned labels in place of A, B, C, and D. This is true for all displays showing solvents.

If you are not going to use one of the solvents set up for your pump, you can change the display so that it shows only three solvents. Complete information for changing the solvents displayed is found under *Display Solvents* on page 49.

General Rules for Entering Time Lines

A file should contain a minimum of two time lines. If a file contains one time line, the pump will not be able to maintain a RUN state, although it will show READY.

The solvent compositions and flow for the zero (0.0) time line are always editable. The Time field 0.0 itself is not editable; it remains as time zero. To add new times you must create new time lines.

To create a new time line, use the down-arrow key to go to a blank line, or press [ENTER] until a blank line appears, then press the [+] key. The new line, automatically incremented one minute past the

previous line, will be displayed. The cursor will appear in the Time field of the new line. The Time field in any new line is editable.

HINT: Don't add all your time lines to the file at once. Enter values for solvent composition and flow in the first time line, then add a new time line. All of the solvent composition and flow rate values will be copied to the new line automatically. In fact, a new line is always identical to the one just before it, with the exception of the Time field as discussed earlier. Solvent composition values can be incremented easily then, from the previous values.

The solvent compositions in each time line must always add to 100. As soon as one percentage is changed, the pump automatically changes the remaining solvents to keep the total equal to 100.

Solvent compositions can be set to whole (X.0) or half (X.5) percents in the P4000.

You can enter times in any order you choose. The pump will automatically rearrange the time lines on the display so that they are in chronological order as soon as you move the cursor off the Time field.

Gradients occur between adjacent pairs of time lines. The pump continually adjusts the solvent composition to achieve the composition shown in the next time line by the time specified.

One of twenty-one preset gradient curves can be selected from the Options Menu (P4000). Creating step gradients is explained on page 48.

Changing Solvent Composition — A P4000 Example

Start with the display shown in Figure 3.4.

Time	A	B	C	D	Flow
0.0	100	0.0	0.0	0.0	1.00
1.0	100	0.0	0.0	0.0	1.00
2.0	100	0.0	0.0	0.0	1.00

Figure 3.4 Example P4000 solvent program

Then decrease solvent A by 20, solvent B automatically increases so that the total is 100 (Figure 3.5).

1.0	100	0.0	0.0	0.0	1.00
2.0	80.0	20.0	0.0	0.0	1.00

Figure 3.5 Example P4000, continued

Then, if you decrease solvent B by 15, the difference is applied to solvent C (Figure 3.6), and so on.

1.0	100	0.0	0.0	0.0	1.00
2.0	80.0	5.0	15.0	0.0	1.00

Figure 3.6 Example P4000, continued

If all solvent fields have been filled in, a change made to one field will cause the adjacent field, to the right, to reflect the difference. If that next solvent goes to 0, the solvent after it is affected, and so on.

In this example the pump would pump 100% solvent A until 1.0 minute into the run. After one minute the pump changes the composition such that at 2.0 minutes into the run solvent A would be at 80%, and solvents B and C at 5% and 15%, respectively.

The Options Menu Under Files, Edit

The Options Menu consists of maximum and minimum pressure levels, an equilibration time, and, in the P4000, a gradient curve type and option to display certain solvents. You are not required to set any values in this menu, although you should always set a maximum pressure level, since the pump will warn you whenever operating pressures exceed this level.

You can access the Options Menu from /FILES/, /Edit/ (Figure 3.7) or by pressing [ENTER] or [∨] on the blank line at the bottom of the

Solvent Program, then selecting /Options/. The Options Menu is shown in Figure 3.7.

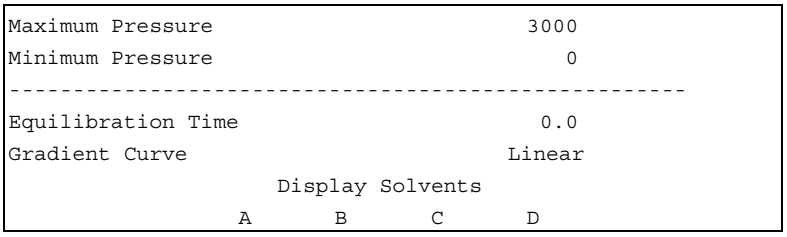


Figure 3.7 The Files, Options Menu (P4000)

Maximum, Minimum Pressures

Change the pressure levels as desired. The maximum value is 6000 psi, the minimum is 0 psi. (The allowed values depend on the pressure units, selected in /OPTIONS/, /More/, Pressure Units.) The maximum pressure value must be greater than the minimum pressure. The pump will not allow you to set maximum and minimum pressures that are inconsistent. If you are unable to edit one value, try to edit the other.

NOTE: Pressure units (psi, bar, or MPa) are selected from the Main Menu, /OPTIONS/, /More/, described in Chapter 4.

During operation, the pump continuously monitors the actual operating pressure and displays this on the Status Screen. If the actual pressure ever exceeds the maximum pressure level, or falls below the minimum pressure level set here, an error message will alert you to this circumstance. For more information about using this error feature, refer to Chapter 4, Options, Error Recovery.

Equilibration Time

If desired, enter a value for the equilibration time. The equilibration time is the time that the pump will maintain the conditions (solvent composition and flow) specified on the first line of a run file (the zero time line), before showing READY on the Status Screen.

Gradient Curve

The P4000 pump has the ability to automatically run a Gradient Curve between time lines. There are twenty-one choices in this field: Linear, Convex 1 to Convex 10, and Concave 1 to Concave 10. These shapes are shown in Figure 3.8. The curve is applied to all solvents participating in the gradient.

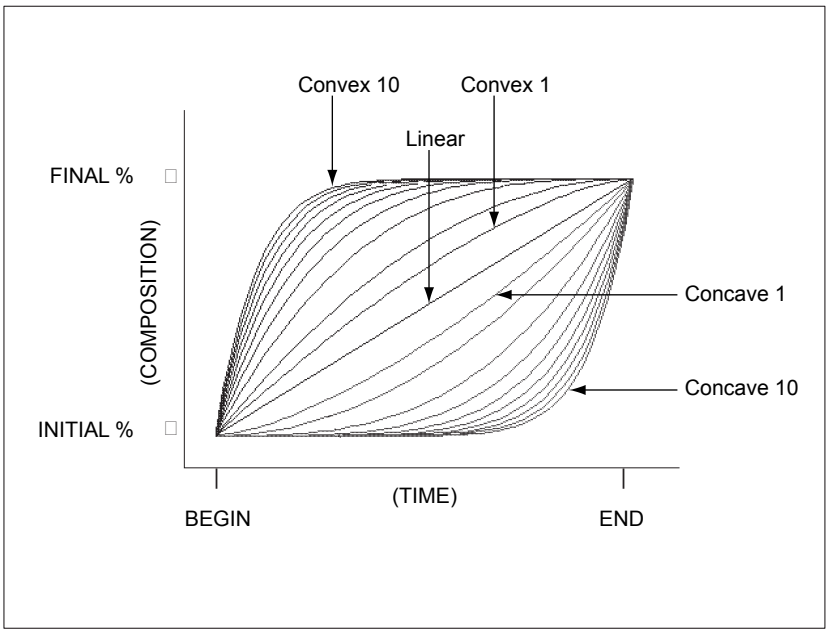


Figure 3.8 Gradient Curves in the P4000

The selected curve operates between adjacent pairs of time lines of the Solvent Program. Gradient curves are usually used by specifying only two time lines (a zero time line and one other time line). The pump automatically runs the chosen gradient curve between these two time lines.

Concave gradients begin with a small and end with a high rate of change. Convex gradients begin with a high rate of change and end with a small rate of change. Linear gradients have a constant rate of change between solvent compositions.

For example, if the Solvent Program contained the time lines shown in Figure 3.9, and the gradient curve were Convex 3, then, over 12 minutes, solvent A would follow the shape of Convex 3, starting at a composition of 20%, and ending with a composition of 100%. Solvent C would follow a complimentary shape, starting at a composition of 80%, and ending with a composition of 0% (Figure 3.10).

```
 0.0      20.0    0.0    80.0    0.0    1.00
12.0      100     0.0    0.0     0.0    1.00
```

Figure 3.9 A two-solvent program

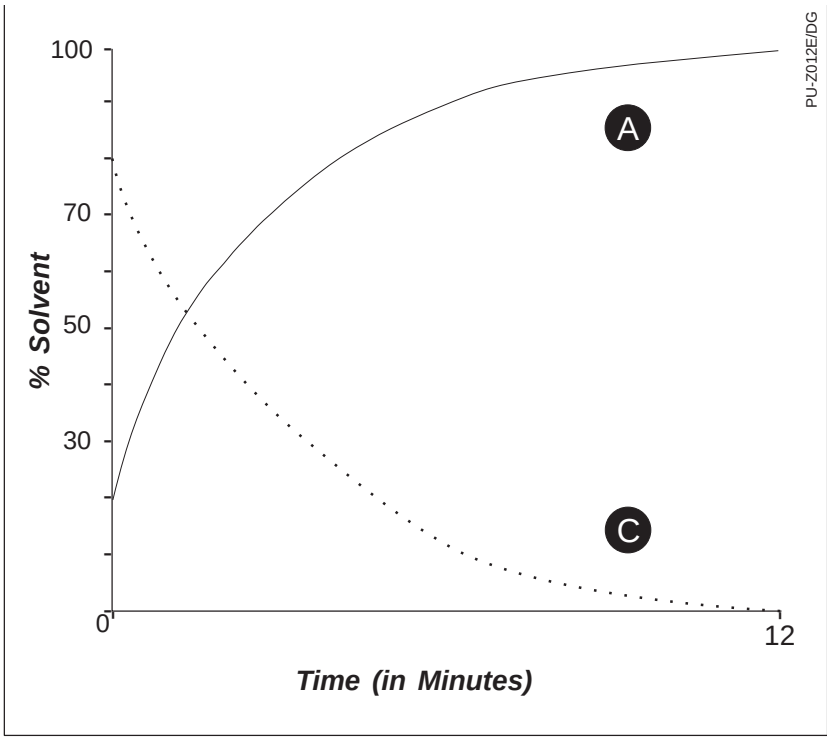


Figure 3.10 Solvents A and C, using a Convex 3 gradient curve.

When three solvents are involved, as in the Solvent Program shown in Figure 3.11, Solvent A follows the designated curve from 0% to 100% over 12 minutes. Solvents B and C follow a similar curve (remember, it is the rate of change that defines the curve shape), from 70% to 0%, and 30% to 0%, respectively, over the same 12 minutes (Figure 3.12).

0.0	$0.^0$	$70.^0$	$30.^0$	$0.^0$	1.00
12.0	100	$0.^0$	$0.^0$	$0.^0$	1.00

Figure 3.11 A three-solvent program

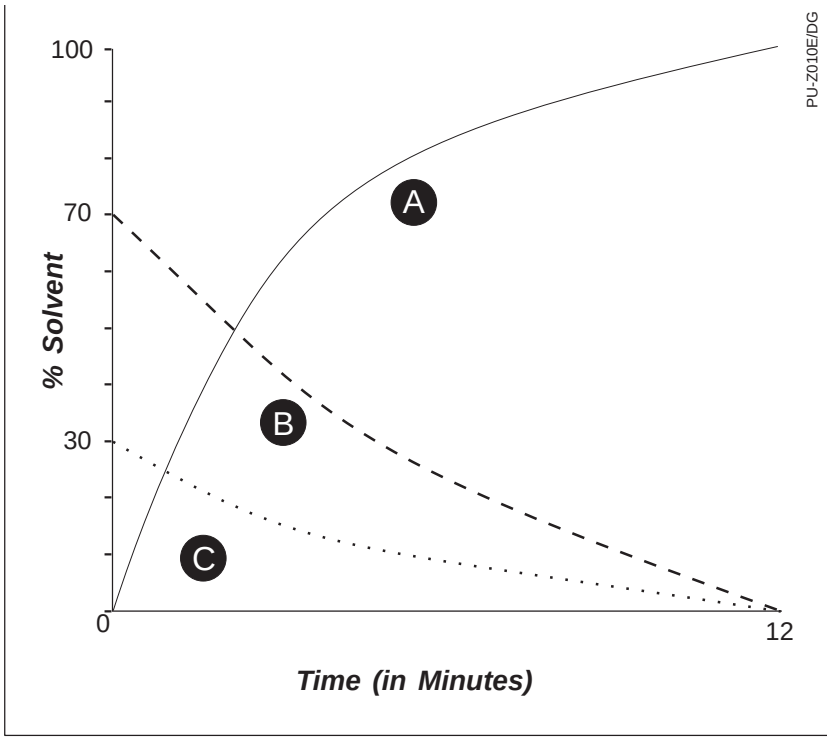


Figure 3.12 Solvents A, B, and C using a Convex 3 gradient curve

The most common gradient is linear. When Linear is selected in the Gradient Curve field, solvent composition follows a straight line, ramping from one solvent percentage to the next, between time lines. Figure 3.13 shows the composition profile of the solvent program shown in Figure 3.11.

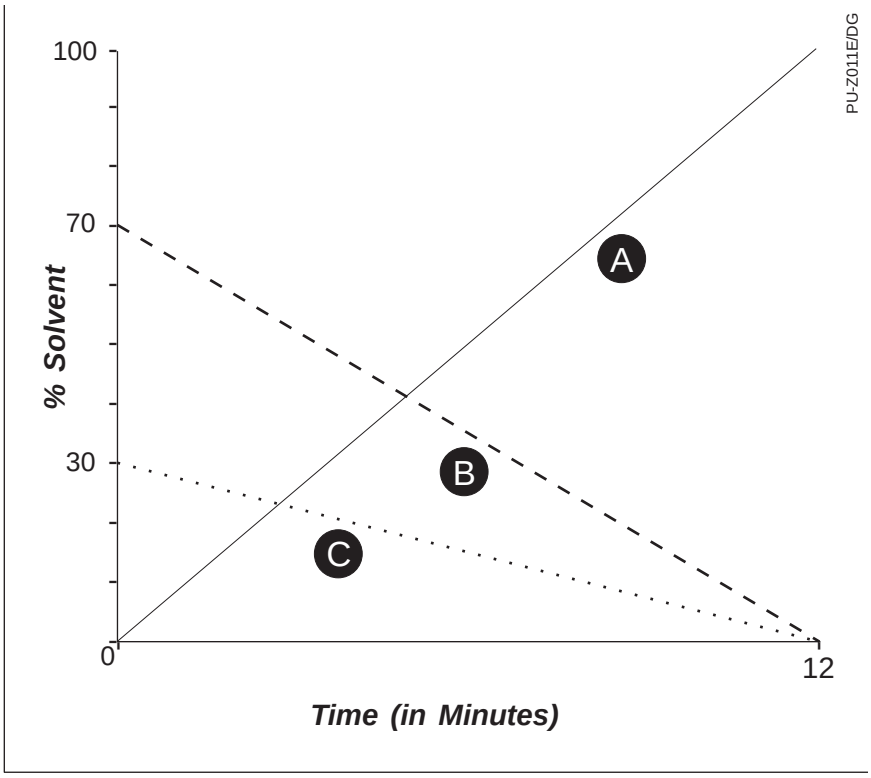


Figure 3.13 Solvents A, B, and C using a Linear gradient curve

Creating Step Gradients

Creating step gradients is easy. Figure 3.14 is an example showing a simple step gradient solvent program.

Time	A	B	C	D	Flow
0.0	100	0.^{0}	0.^{0}	0.^{0}	1.00
5.0	100	0.^{0}	0.^{0}	0.^{0}	1.00
5.1	80.^{0}	20.^{0}	0.^{0}	0.^{0}	1.00
10.0	80.^{0}	20.^{0}	0.^{0}	0.^{0}	1.00
10.1	60.^{0}	40.^{0}	0.^{0}	0.^{0}	1.00
15.0	60.^{0}	40.^{0}	0.^{0}	0.^{0}	1.00
15.1	30.^{0}	70.^{0}	0.^{0}	0.^{0}	1.00
20.0	30.^{0}	70.^{0}	0.^{0}	0.^{0}	1.00

Figure 3.14 A two-solvent step gradient solvent program (P4000)

Each "step" is created by specifying a very short amount of time (0.1 minutes) in which the gradient is allowed to operate. In the example, the pump establishes the conditions in the Time = 0.0 min time line, and maintains this until the Time = 5.0 min time line (because both time lines have identical compositions). The first step takes place between 5.0 and 5.1 minutes. The pump establishes the conditions in the Time = 5.1 minutes time line and maintains them

until 10.0 minutes, when the next step occurs. The last step occurs at 15.0 minutes.

HINT: System baseline may take time to stabilize.

Display Solvents

In the Display Solvents field you can change the number of solvents displayed in the file. You should be certain that the composition for any undisplayed solvent is zero in all time lines before you try to turn off its display. The pump automatically checks the solvent composition field for each solvent and prevents you from turning off the display for any one whose composition is not zero.

Use the [+] / [-] key to see and select the set of solvents that suits your needs. A minimum of three solvents must be displayed at all times. Automatic solvent proportioning rules apply only to the solvents that are displayed.

HINT: It is simpler to program a three-solvent display since the automatic solvent proportioning rules make binary program creation straightforward (e.g. if you select Display Solvents ABC and want to program only A and B, then you only need to select values in the A field. If you want to program B and C, then you only need to select values in the B field. If you want to program A and C, then select values in the C field.)

Timed Events Menu

You can access the Timed Events Menu from /FILES/, /Edit/, or by pressing [ENTER] or [∨] on the bottom line of the Options Menu, then selecting /Timed Events/. The Timed Events Menu is shown in Figure 3.15.

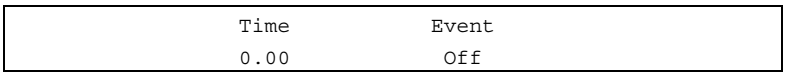


Figure 3.15 The Timed Events Menu

A timed events output is available at the external events port. This output can be used to control an external device such as a column switching valve. The output can be turned on or off up to six times per run.

The time on the first line of the menu remains 0.0. Additional timed events are added much like time lines in the Solvent Program. Use the down-arrow key to reach a blank line, then press [+]. As many as five additional lines can be added. The Time field in any added line is editable. Enter times and turn the output Off or On in any of the six Event fields using the [+] / [-] keys.

For example, Figure 3.16 shows a timed events example.

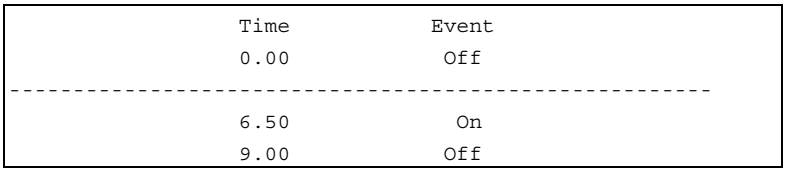


Figure 3.16 Timed events example

In this example, a timed event output (signal) would be sent (turn on) at 6.5 minutes into the pump's run and become inactive (turn off) two and a half minutes later, at 9.00 minutes.

To delete a line, move the cursor to the time field, then press and hold [-]. The time field will eventually become blank, and the line will disappear. If the time has more than one non-zero digit (for example 6.50), then decrement each digit, starting with the left-most digit. Release the [-] key after each field becomes blank and decrement the next digit.

THE SHUTDOWN FILE

The Shutdown file (file number S) is an easy way to assist you with the proper maintenance of the pump. Since your pump and column should never be allowed to sit idle with salts or corrosive materials in them (including water), the Shutdown file lets you automatically flush the pump and the column at the completion of a series of samples.

Whenever the pump detects that it has been in a READY state, without a run being initiated, for a specified period of time, it automatically loads, initializes, and runs the Shutdown file.

NOTE: The pump's clock is reset anytime the [RUN] or [STOP] key is pressed, or anytime the /Reset/ command is issued.

Some instances when you might want to use a shutdown file are:

- To keep solvent at a very low flow rate flowing through the LC
- To clean the column and keep the pump running
- To clean the column, then stop solvent flow through the LC

The Shutdown file's Solvent Program, Options, and Timed Events Menus are exactly the same as those for other files with one exception: in the last line of the Options Menu, the display shows a Time from READY field instead of the Equilibration Time field (Figure 3.17). Gradient curves are not available in the Shutdown file.

```
Minimum Pressure                          0
Time from READY                         Off
```

Figure 3.17 Last lines of the Options Menu of the Shutdown file

The Shutdown file will be automatically loaded and run if the current run file has not been started, either manually or remotely, in the time specified in the Time from READY field. There are ten preset times (in minutes) that can be selected in this field: 5, 10, 20, 30, 45, 60, 90, 120, 240, or 480. The Shutdown feature can be turned off completely by selecting "Off." The Time from READY timer starts as soon as the Status Screen shows READY.

If the Shutdown file is loaded automatically, the pump will do one of the following when it reaches the last time line of the Shutdown file:

- If the flow rate is greater than zero (> 0) the pump will maintain (Hold) the last time line's solvent composition and flow rate indefinitely.
- If the flow rate is zero (= 0) then the pump stops, and STOP is shown on the Status Screen. In addition, the pump remembers the run file that had been used just before the Shutdown file was automatically invoked, and restores that file to the run file position.

If the Shutdown file is loaded manually (/FILES/, /Load/), then it remains the run file until another file is loaded.

NOTE: If the pump will be operated unattended for an extended period of time, ensure that the solvent reservoir and waste containers have sufficient capacity.

If you use an autosampler, specify the time interval to be longer than the cycle time on the autosampler (for example., [1.5 × cycle time], or [cycle time + 20 minutes]). If you perform manual injections, set the time interval to the maximum time likely between injections. Depending on your own circumstances, you may wish to turn the Time from READY to "Off."

LOADING A FILE

When you select /Load/ the display shown in Figure 3.18 appears.

```
>Load File   1:(filename)
```

Figure 3.18 The Load display

Select a file by number, then press [ENTER]. A message will confirm that the file was loaded. A loaded file is referred to as the "run file."

As soon as a file is loaded, the pump will initialize the file, i.e., bring the pump to the conditions specified on the zero (0.0) time line of the file. The Status Screen shows INIT until these conditions are achieved. If an equilibration has been specified, the pump will then show EQUIL until the equilibration time has been reached.

After initialization and equilibration, the Status Screen then shows that the pump is READY. The pump's clock will not start unless a run is triggered either manually by pressing [RUN], from a properly connected (hardwired) autosampler, or from a data system.

COPYING A FILE

When you select /Copy/ the display shows (Figure 3.19):

```
Copy File 1:(filename)
  to File 2:(filename)
```

Figure 3.19 The Copy display

Select both files by number. Press [ENTER] after both files are specified only if you are sure you want to copy the parameters from the top file into the other file. The Copy command overwrites the selected file. Once overwritten, previous values cannot be retrieved. If you do not want to copy a file, leave the display by pressing [MENU] or [STATUS], or the up-arrow key. Press [ENTER] to proceed with the copy operation, and a message confirms its completion.

If you try to copy to a protected file, a message will appear indicating that the file is protected. You will then be returned to the Copy display.

DELETING A FILE

When you select /DELETE/ the display shows (Figure 3.20):

```
>Delete File 1:(filename)
```

Figure 3.20 The Delete display

Select a file by number, then press [ENTER]. A message is displayed, asking you to confirm the selection. Press [ENTER] only if you are sure you want to delete the file. Once deleted, a file cannot be retrieved. If you do not want to delete the file, leave the display by pressing [MENU] or [STATUS], or the up-arrow key. A confirmation message concludes a successful file deletion.

When you delete a file you are actually returning all parameters in the file to their default values.

If you try to delete a protected file, a message will appear indicating that the file is protected. You will then be returned to the Delete display.

Purging Solvent Lines

If none of the solvent lines has solvent in it, refer to Priming the Pump in Chapter 1.

Air will slowly diffuse through the thin-wall Teflon inlet tubing, and into the solvent. If the pump flow has been turned off or if any of the solvent lines from the solvent reservoirs to the proportioning valve have not been used in the past several hours, those lines should be purged with degassed solvent before use.

The purge operation can be activated when the pump is in any state.

NOTE: Open the bypass valve prior to purging, or else ensure that your chromatographic column can withstand the purge parameters you set before performing any purge.

THE PURGE MENU

Pressing the [PURGE] key brings the Purge Menu to the display (Figure 3.21).

NOTE: DO NOT move the cursor out of the Time field using the [ENTER] key until your LC is ready to start a purge.

```
Purge              Flow                   Time
   A               1.00                   0.0
------------------------------------------------------------
(If BLEND is selected under Purge for P4000)
   A       B               C              D
   100      0               0              0
```

Figure 3.21 The Purge Menu

Purging can be accomplished in one of three purge modes: Flow, Pressure, or Both. In Flow mode, purging is regulated by the flow rate from the pump. In Pressure mode purging is accomplished at a specific fluid pressure. In Both mode purging is accomplished using both flow and pressure parameters. Internal limits are designed to protect your LC system: in flow mode, the pump will accept rates of 0.1 to 10.0 mL/min. The maximum pressure the pump allows in flow mode is the maximum pressure value from the current run file; in pressure mode, the maximum flow the pump will reach in attaining the set pressure is 6 mL/min.

NOTE: Purging in Pressure or Both mode with the bypass valve open may not allow sufficient pressure to be generated in the system. The pump will operate at maximum flow, but the target pressure may not be achieved. Ensure that your analytical column can withstand the purge pressure (or use

a flow restrictor or old column), and do not open the bypass valve.

The purge mode can be changed from /OPTIONS/, /More/, Purge Mode, described in Chapter 4. The top line of the Purge Menu will show either Flow or Pres, depending on the purge mode you select.

Purge (field)

There are six choices available in the Purge field for the P4000 (A, B, C, D, All, Blend). When Blend is selected, one additional line can be accessed by pressing the down-arrow key (not the [ENTER] key!). These are used to set solvent compositions. You should enter values for the solvent composition in these fields before you initiate a purge.

Initially, the compositions will be copied automatically from the first line of the Run File. These solvent fields follow the same proportioning rules as the fields in the Solvent Program; they must total 100. Unlike the solvent program however, all solvents are automatically displayed, regardless of the selections made from Display Solvents.

Flow or Pres

The flow rate is taken from the last line of the run file. Use this field to select a flow rate. If the purge mode is Pres (pressure) instead of Flow, the pressure is automatically set to one-half (50%) of the maximum pressure level set in the run file.

NOTE: To change the purge mode you must go to /OPTIONS/, /More/, Purge Mode. Refer to Chapter 4 for complete information.

Time

The Time field is used to set the length of time you want the pump to purge. If the field remains 0.0, purging, once started, will continue until stopped.

NOTE: When you check the purge operation from the Status Screen, the P4000 displays PURGE. The time remaining to complete the purge is shown on line 4.

Starting a Purge

To initiate a purge cycle, move the cursor out of the Time field by using the [ENTER] key.

If the time stays set to 0.0, purging will continue until you press [STOP], or a file is initialized. If a time is entered, the pump will purge until the time set. After completing a purge, the pump automatically initializes the run file.

When running in a timed purge mode, the Status Screen will indicate the time *remaining* to purge (that is, counts backwards toward 0.0). If the [PURGE] key is pressed once more, the Purge Menu is again displayed and the time countdown continues, uninterrupted, unless the cursor is moved out of the Time field by pressing [ENTER], at which point the timer will be reset and purging will restart.

The pump will retain the selections made in the PURGE display as long as the power to the pump is on.

If power is turned off, the pressure parameter must be re-entered. However, the flow rate setting is retained in NOVRAM.

Stopping a Purge

There are three ways to stop a purge cycle:

- Press the [STOP] key
- Load (initialize) a file by selecting a file using /FILES/, /Load/
- Allow the pump to complete the purge (if a time has been specified), at which point the pump will automatically initialize the last run file.

Running the Pump

To perform a run:

- purge the solvent lines if necessary,
- establish a READY state (may require the pump to INIT, and/or EQUIL)
- inject the sample, and
- press [RUN].

If you are performing a manual injection, fill the injection loop, check that the Status Screen shows READY, inject the sample, and press [RUN] in succession. If an autosampler is hardwired to the pump, simply initiate the autosampler run sequence.

NOTE: Generally, when operated manually, if the pump is stopped (i.e., the Status Screen shows STOP), the run file can be initialized by pressing [RUN], and then started by pressing [RUN] again when the Status Screen shows READY.

ESTABLISHING READY

The READY state means that the pump has reached the conditions specified on the first line of the solvent program and is ready to start a run.

Initializing a file

To achieve the READY state, initialize a run file, by any of the following methods:

- Load a file by selecting /FILES/, /Load/, a file number, then pressing [ENTER]. This simultaneously loads the run file and initializes it.
- If the pump is stopped (Status Screen shows STOP), press [RUN]. This initializes the run file, without starting the run.
- If the pump is stopped or in run, reset the run file by selecting /COMMANDS/, /Reset/. This initializes the run file without starting the run.

Wait for the pump to reach zero time line conditions. If an equilibration time was specified in the file, the Status Screen will show EQUIL for this period of time.

PRESSING [RUN]

As soon as the pump shows READY, begin the run by pressing the [RUN] key. This starts the pump's clock.

NOTE: Even though the pump may be ready, your column and the rest of your LC system may not be! Take into account your own LC application and ensure that your column is at chemical equilibrium and that the other instruments in your system are ready before your proceed with any injection.

As soon as the [RUN] key is pressed, the pump begins to operate based on the time lines in the Solvent Program of the run file, and the Status Screen is displayed. The Status field on the P4000 Status Screen shows RUN, indicating that the pump is operating from the parameters in the run file. Line 4 shows the time into the run. Status is fully described on page 61.

STOPPING THE PUMP

There are a number of ways to stop the pump, depending on what you wish to do next. More information regarding the Commands Menu, referred to below, is found on page 60.

Using a Hold Command

If you want to stop the pump's clock momentarily (but not stop solvent flow), and plan to resume the run where it was stopped, press [MENU], and select /COMMANDS/, /Hold/. To resume, select /COMMANDS/, /Continue/.

By Resetting the Pump's Clock

If you want to restart the current run, press [MENU] and select /COMMANDS/, /Reset/. This stops the pump's clock and returns to the zero time line, automatically initializing the file. Restart the run by pressing [RUN] after the pump shows READY.

By Pressing [STOP]

If you want to completely stop the pump, press [STOP]. This aborts the run and stops solvent flow through the pump. If you want to resume with the same file, you must initialize the run file by pressing [RUN], waiting for the pump to show [READY], then pressing [RUN] again.

WHILE THE PUMP IS RUNNING

There are several messages which can appear in the Status field. These are discussed in detail in the *Status* section below.

While the pump is running you may do several things without disturbing pump operation:

- edit files (/FILES/, /Edit/).
- check some pump performance parameters.
- edit the run file from the Status Menu. (This has an effect on the current run - see page 63 for more information.)

The Commands Menu

The Commands Menu is reached by pressing [MENU] and selecting /COMMANDS/.

When /COMMANDS/ is selected the display shows (Figure 3.22):

```
>Reset
·Hold
------------------------------------------------------
·Repeat
```

Figure 3.22 The Commands Menu

RESET

Reset is used when the pump is in RUN and you want to abort the run without stopping the pump's flow. Reset reinitializes the file *(i.e.,* resets the timer to zero). The result is that the pump reestablishes the conditions of the zero time line, and returns the pump to a READY state. Pressing [RUN] restarts the run.

HOLD/CONTINUE

Hold is used to stop and hold the pump's clock. It causes the pump to maintain the operating conditions used at the moment the Hold command was issued, including flow rate. These conditions are maintained indefinitely unless:

- A Continue command is used from the Commands Menu, at which point the timer continues from the point at which it was held.
- The [STOP] key is pressed.
- The Reset command is selected.

Whenever a Hold command is issued, the word Continue will replace the word Hold in the Commands Menu.

To select any of the commands on the Commands Menu, move the cursor to the desired line, and press [ENTER] to issue the command. The display returns to the Status Screen.

REPEAT

The Repeat command initiates two different events, depending on what the pump is doing when the command is issued:

1) If a queue is running, the /Repeat/ repeats the run current in progress. (To skip to the *next* run, select /Reset/.)
2) If a queue is not running, then /Repeat/ is identical to /Reset/.)

Status

The Status Screen appears whenever the pump is powered on, a file is initialized, or the [STATUS] key is pressed. The Status Screen, consisting of four lines in the P4000, shows the pump's current operating values. Below the Status Screen is the Status Menu, where you can view and, if necessary, edit parameters of the run file.

STATUS MESSAGES

The Status field of the Status Screen can show any of the following messages:

Table 3.2 Status Messages

(time)	• The time into the run (or time remaining if a timed purge).
EQUIL	• The pump is equilibrating the LC by maintaining the conditions on the first line of the run file for the equilibration time specified in the file.
HOLD	• /COMMANDS/, /Hold/ has been selected. The pump is maintaining the conditions that existed when the Hold command was issued, including the flow rate. To continue, select /COMMANDS/, /Continue/.
INIT	• The pump is initializing a file.
NRDY	• (Not Ready) The pump is waiting for a signal from another instrument.
PURGE	• The pump is purging.
Q *(time)*	• The file listed in Order 1 of the queue is running. The time into the current run is shown.
QEQUIL	• The pump is equilibrating the LC based on a file listed in the queue.

Table 3.2 Status Messages, *continued*

QREADY	• A queue has been loaded and the run specified in Order 1 can be started.
Q RUN	• Appears briefly when a queue is run.
QSTOP	• A run listed in the queue has been stopped.
READY	• The pump has achieved the conditions on the first line of the run file, and the equilibration time has elapsed (if set). A run can be started. READY, shown in Status.
RUN	• The pump is running the file.
STOP	• All mobile-phase flow through the pump is stopped.
SYNC	• This is a remote communications message that appears briefly whenever a run is started. At lower flow rates, it may be seen for longer periods of time.

P4000

```
Status            Flow           PSI     MaxP
 READY            1.00             0     3000♦
-------------------------------------------------------
Stat       A       B       C       D
 0.0      100.    0.0     0.0     0.0

File 1:EXAMPLE
Time       A       B       C       D      Flow
 0.0      100     0.0     0.0     0.0     1.00
 1.0      100     0.0     0.0     0.0     1.00
 2.0      80.0    5.0     15.0    0.0     1.00

Maximum Pressure                          3000
Minimum Pressure                             0
Equilibration Time                         0.0
Gradient Curve                          Linear
>Save File
(appears only if the run file is changed)
```

Figure 3.23 Example P4000 Status Screen and Status Menu

The first and second lines of the P4000's Status Screen show the state, flow, pressure, and maximum pressure setting.

The third and fourth lines of the Status Screen show the pump's time and current solvent composition.

Solvent compositions are displayed to tenths of a percent (0.1).

EDITING A RUN FILE

The remaining lines, which comprise the Status Menu, show the file name, solvent program, and options of the run file. If the solvent program and options do not appear, the Status Lock feature has been turned on. (Status Lock is described in Chapter 4, under the OPTIONS, More Menu.)

All time lines of the run file's solvent program may be edited while the pump is running, as may the maximum and minimum pressures, and the equilibration time. Any changes take effect as soon as the cursor leaves each field. However, the changes are saved only when the /Save File/ command is selected below Gradient Curve (P4000).

HINT: Any time the parameters of the Run File are changed, the /Save File/ command will appear at the bottom of the Status Menu. Note that you cannot save changes using the /Save File/ command if file protection for that file has been turned "On".

When changing the parameters of a run file from the Status Menu, you must select /Save File/ in order for the pump to remember your changes. The pump will, however, use your changes until the next time the file is initialized by selecting /FILES/, /Load/. Use the same methods described on page 41 and on page 43 to edit the parameters of a run file. New lines may be added to the run file. Timed events may only be edited using /FILES/, /Edit/, /Timed Events/, and will not take effect until that file is loaded (becomes the run file).

Monitoring Pump Performance

The pump has the capability to automatically monitor its own performance and warn you if a flow problem exists. The way the pump responds to error conditions is set in the Error Recovery menu of OPTIONS, selected from the Main Menu. These options (not to be confused with File Options), are described in detail in Chapter 4.

Certain flow and pressure conditions are monitored continuously. For example, if a time line with a zero flow rate has been encountered, the pump responds with the appropriate error message shown below. As mentioned earlier, you can select the pump's response to certain conditions.

```
! !  ZERO FLOW RATE  ! !
```

```
! !  MAX PRESSURE EXCEEDED  ! !
```

Figure 3.24 Example error messages resulting from flow problems

"ZERO FLOW RATE" indicates that a time line with a zero flow rate has been encountered. "MAX PRESSURE EXCEEDED" is a condition whose pump response is user-selectable in /OPTIONS/, /Error Recovery/.

The pump can also initiate a Flow Stability Test. This test is run by selecting /TESTS/, /Diagnostics/, /Flow Stability/. The results are continuously displayed until another key is pressed. The results consist of 2 parts. The first is a summary of the performance evaluation (STABLE, ACCEPTABLE, or UNSTABLE) and the second is a number that indicates a position in each range. Further explanation of this test can be found in Appendix A.

Shutting Down at the End of the Day

Some shut down suggestions when you conclude your work with the pump for the day:

- Do not leave buffers in the pump or in your LC; purge the pump (50/50 MeOH/water is a good solvent) if it has just concluded a run using buffered solutions. (Don't leave 100% H_2O in your LC.)
- Leave the column full of a solvent recommended by the column manufacturer.
- Make use of the Shutdown file. The pump will automatically maintain the conditions specified on the last line of the file. This is particularly useful if you prefer to have a small but continuous flow of solvent through your LC system while it is idle.

4

Advanced Operations

Introduction

This chapter focuses on two menu items: Options, used to set some important features, and Queue, used to build a list of files to run. This chapter also describes what the P4000 pump's Develop File is and how to use it. The majority of this chapter discusses the Develop File.

The Options Menu

The Options Menu (accessed from the Main Menu, not from /FILES/), contains seldom-changed features such as the pump's response to certain electrical and flow conditions, user-selected display and operational preferences, and file protection.

```
·FILES                    ·QUEUE                    ·TESTS
          ·COMMANDS                        >OPTIONS
```

Figure 4.1 The Main Menu with /OPTIONS/ selected

The P4000 Options Menu is shown in Figure 4.2.

```
>Solvent Selection
·Error Recovery
------------------------------------------------------------
·More
```

Figure 4.2 The P4000 Options Menu

ERROR RECOVERY MENU

The pump continuously checks pressure so that problems can be indicated on the display immediately. The pump can also sense a power failure or power interruption. The Error Recovery Menu (Figure 4.3) is used to preset the pump's response to detecting error conditions in any of these three operating parameters.

```
AC Power Fail                          Stop
@Maximum Pres                          Stop
```

Figure 4.3 The Error Recovery Menu

Each field in the Error Recovery Menu can be set to one of three selections:

Selection	Pump's Response
Stop	The pump stops immediately if the condition is encountered
Continue	The pump continues as if the condition had not occurred
Shutdown	The pump immediately stops, then loads and runs the Shutdown file.

AC Power Fail

The pump may sense a power interruption at any time. Select Stop, Continue, or Shutdown in the AC Power Fail field to stop, continue, or shut down the pump as soon as power is restored.

NOTE: If the power switch is turned off while the motor is running, the pump considers this a power failure and will respond accordingly as soon as power is restored.

@Maximum Pres

In a file's Options Menu, you can change the maximum pressure level (the default is 6000 psi). If the pump's operating pressure ever exceeds this value, the pump will operate based on your selection in the @Maximum Pres field. Select Stop, Continue, or Shutdown to set the pump's response to sensing operation at maximum pressure.

MORE MENU

To access other options, select /More/ (Figure 4.4).

```
·Error Recovery
>More
```

Figure 4.4 Selecting /More/

The More Menu (Figure 4.5) contains additional, miscellaneous, user preferences, such as the units the pressure is displayed in and how quickly field choices scroll when the [+] and [-] keys are pressed and held.

```
Pressure Units                              PSI
Purge Mode                          Flow
-------------------------------------------------------
Delay Volume                                0.0
Cursor Speed                            Medium
Status Lock                                 Off
Ready Output Active                          Hi
File Name                        Protect
1:(filename)                                Off
2:(filename)                                Off
3:(filename)                                Off
4:(filename)                                Off
5:(filename)                                Off
6:(filename)                                Off
7:(filename)                                Off
8:(filename)                                Off
9:(filename)                                Off
```

Figure 4.5 The More Menu

Pressure Units

Select either PSI, BAR, or MPa as your preferred units. All menus and screens that show pressure units will reflect the selection.

Purge Mode

Select either Flow, Pressure, or Both in the Purge Mode field. Your selection is reflected on the Purge Menu and is used as the primary purge parameter. Select Flow if you wish to purge based on a flow rate, Pressure if you want to purge based on an operating pressure, or

Both if you wish both flow and pressure parameters to govern purging.

Purging in pressure mode requires a certain amount of back pressure in the system. Ensure that your analytical column can withstand the purge pressure (or use a flow restrictor or old column), and do not open the bypass valve.

Delay Volume

The gradient delay volume is the volume of mobile phase that the pump will pump before allowing a SpectraSYSTEM autosampler to proceed with an injection. This prevents an injection from occurring before the gradient has reached the column. Determine the delay volume of your system, and enter this value (in mL) in the Delay Volume field. (The pump must be properly hardwired to the autosampler for the delay volume to be recognized by the autosampler. The pump sends a signal to the autosampler's Inject Hold input while the delay volume is being pumped.)

Cursor Speed

Cursor Speed is used to change how quickly choices scroll on the display when the [+] and [-] keys are pressed and held, and how quickly a menu scrolls (up and down) when the arrow keys are pressed and held. Select Fast, Medium, or Slow.

Status Lock

Status Lock prevents a run file from being edited from the Status Menu. When Status Lock is On, the Status Menu only shows the run file name (and number). The rest of the run file cannot be accessed.

Status Lock is different from File Protection (below). A protected file cannot be saved from the Status Menu (using the /Save File/ command), although it can be viewed (from the Status Menu), nor can it be edited from /FILES/. Status Lock, on the other hand, prevents a run file from being seen (and hence edited) from the Status Menu. The file remains editable from /FILES/.

The Status Screen is unaffected by Status Lock; it can always be viewed.

Ready Output Active

The Ready output, located on the back of the pump, continuously sends an electrical signal to any device hardwired to it. Use the Ready Output Active field to choose whether the signal is either a 5V signal (Hi) or a 0V signal (Lo) whenever the pump is in a READY state. If the pump is not READY, the other signal is output. Whenever the pump's Ready Output is hardwired to a SpectraSYSTEM autosampler to coordinate injections, it should be set to provide "Hi" voltage in the READY state.

File Protection

Each numbered file can be safeguarded against accidental or unauthorized changes by turning on the file protection feature. When File Protection for a specific file is turned On, that file cannot be edited, deleted, or copied to. Initially, all files are editable (file protection is Off.) Use the [+] or [-] key to select Off or On in the Protect field.

SOLVENT SELECTION MENU

The Solvent Selection Menu displays four solvent labels (A, B, C, and D), each of which can be changed to one of thirteen preset solvent names. Matching a label to the actual solvents that are connected to the four inlet lines is a convenient way to avoid confusion. When you label solvents, each of the pump's displays that would normally show lettered solvent labels A, B, C, and D will instead show the name you set. The labels available are: H_2O, MeOH, ACN, Phos (Phosphate), Acet (acetate), TFA, Buff (Buffer), Acid, Base, THF, IPA, MeCl, and HEX.

To change a label, select /OPTIONS/, /Solvent Selection/. The display in Figure 4.6 appears.

```
                  Select Solvents
[  A  ]           [  B  ]        [  C  ]        [  D  ]
```

Figure 4.6 Solvent Names display

Use the [+]/[-] keys to select a label in any of the four fields. Figure 4.7 shows an example.

```
                  Select Solvents
[ H2O ]           [ MeOH ]       [ IPA ]        [  D  ]
```

Figure 4.7 Solvent labels for A, B, and C chosen

NOTE: If your solvent isn't in the list of available choices, retain the default letter as the solvent label.

The Queue Menu

The Queue Menu is used to edit, load or delete a chronological list of files the pump will run, and the number of times each file is run. By linking several files together by means of a queue you can match specific pump files with injections in your sequence. Any regular file (numbers 1 - 9 in the P4000) can be put into the queue. (The Shutdown and Develop files are not queue-able.)

You can create a queue with as many as ten lines. Access the Queue Menu by pressing [MENU] and selecting /QUEUE/ (Figure 4.8).

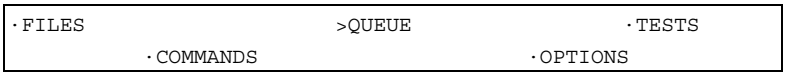


Figure 4.8 The Main Menu, showing /QUEUE/ selected

When you select /QUEUE/ the display shown in Figure 4.9 appears.

Figure 4.9 The Queue Menu

This section describes how the queue works, and how to edit, load, delete, and run a queue. It also explains how a running queue can be paused, stopped, or edited.

HOW THE QUEUE WORKS

The pump looks at the first line of the queue to determine which file to run. It then runs that file as many times as specified, with each run being initiated by a manual or remote RUN command.

To use the queue:

1. Edit the Queue.
2. Load the Queue.
3. Initiate the [RUN] manually or remotely each time a new injection/run needs to be started.

NOTE: Set the autosampler cycle time equal to the pump run time.

The pump will run based on the files listed in the queue. For the example queue shown in Figure 4.10, the pump would run File #4 ten times, then File #2 five times, and then File #1 twenty times, for a total of 35 runs. Note that the file number is not the same as the file's Order (chronological position) in the queue.

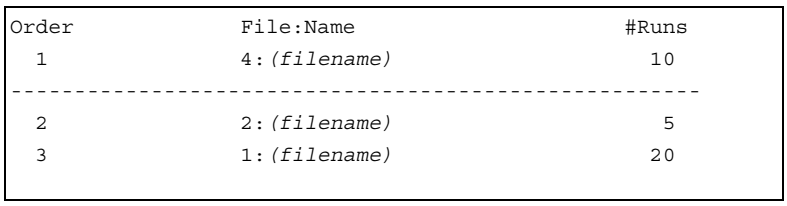


Figure 4.10 An Example queue

Editing, loading, and deleting a queue are explained on the following pages.

EDITING THE QUEUE

To view, build, or change the queue, select /Edit/. A display similar to Figure 4.11 appears.

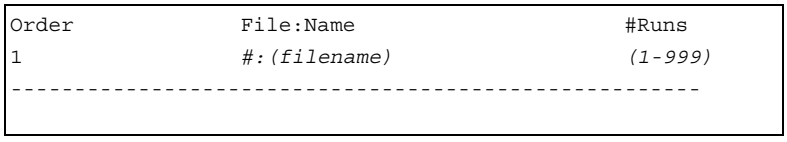


Figure 4.11 The Queue's Edit Menu

Order

The field is not editable. As you add more lines to the queue, this field automatically displays the numerical order of lines in the queue.

File:Name

The File:Name field is used to select the name of each file to be run. Use the [+] / [-] keys to select one file for each line.

#Runs

The field is used to enter the number of times you want a particular file to be run before the next file (Order 2) is loaded.

Adding Lines to the Queue

Once the File:Name and #Runs fields for Order 1 are filled in, you can add an additional line to the queue by pressing the down-arrow key or [ENTER]. The cursor will move to the Order 2 line. For each line that you add, select a file name and enter a value for the number of times you want the file to run. You may add as many as nine lines (for a total of 10 lines).

```
Order              File:Name                          #Runs
1                  #:(filename)                       (1-999)
-----------------------------------------------------------
2                  2:EXAMPLE                            5
```

Figure 4.12 Adding lines to the queue

HINT: You can repeat a file name several times in the queue.

Deleting Lines from the Queue

To delete lines from the queue, put the cursor in the File:Name field, and press [-] until the File:Name field is blank. The remaining entries will be re-sorted as soon as the cursor is moved off the line.

LOADING THE QUEUE

The Queue Menu's Load command simultaneously loads the queue into the pump and begins running the queue by initializing the file in Order 1. When /Load/ is selected, the display prompts you to confirm the operation (Figure 4.13). Press [ENTER] if you wish to proceed. (If you do not want to load the queue, exit the display by pressing [MENU], or [STATUS], or the up-arrow key.) A confirmation message will appear, completing the load operation.

```
                    >Load Queue
```

Figure 4.13 Loading the queue

If you load a queue while another file is running, the queue will immediately take over the pump's operation. The Order 1 file becomes the run file and is initialized.

Once the queue is loaded, you are returned to the Status Screen. The Status Screen will show QREADY, as soon as the pump is ready to begin a run using the file specified in Order 1.

DELETING THE QUEUE

The Queue Menu's command is used to erase the *entire* queue. When /Delete/ is selected, the display prompts you to confirm the operation (Figure 4.14). Press [ENTER] only if you wish to delete the *entire* queue. A confirmation message completes the operation.

If you do not want to delete the queue, press the up-arrow key, or [STATUS] or [MENU].

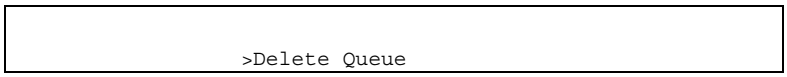


Figure 4.14 Deleting the queue

If you only want to delete certain lines of the queue, use the Edit Menu. Refer to the paragraph Deleting Lines from the Queue on page 72, for more information.

You can delete the queue at any time, regardless of whether or not it is running. If you delete a running queue, the current run is completed. The file that had been in Order 1 remains as the run file.

NOTE: Deleting a queue has no effect on the files themselves, it simply erases the list of files.

The contents of the queue is lost whenever the pump is switched off or a power failure occurs. The file that had been in Order 1 will be the run file when power is restored.

RUNNING A QUEUE

To run a queue, simply load it by selecting /QUEUE/, /Load/. When the pump's Status Screen shows QREADY, you can begin running the first file in the queue by pressing [RUN] or by having another LC instrument trigger the run. The pump will continue to run the file in Order 1 each time a new run is started, until it has been run the number of times specified in the #Runs field. The pump then loads the file designated in Order 2 and uses that file the number of times specified in that line, and so on, until the entire queue has been run.

CHECKING A QUEUE'S PROGRESS

You can track the progress of a running queue from the Queue Menu. To view the progress of a running queue:

1. Press [MENU].
2. Select /QUEUE/. Note that when a Queue is loaded, the Queue Menu (Figure 4.15) changes. The /Load/ selection is replaced by /Pause/.

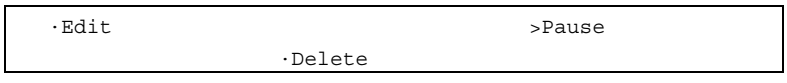


Figure 4.15 The Queue Menu when a queue is loaded (running)

3. Select /Edit/ to view the running queue. The display will look similar to Figure 4.10.

While the queue is running, you can see the #Runs field automatically decrease by one with each run (injection). When the last run is made for a file, the queue is automatically re-sorted. The information for Order 2 is moved up to Order 1, and the information for Order 3 is moved up to Order 2. This process continues until the queue becomes empty, is paused, or is deleted.

You can also see the progress of the current run in the queue from Status. When a queue is running in the P4000, a Status Screen similar to Figure 4.16 will be displayed. Note that the Status Screen shows Q RUN on line 2.

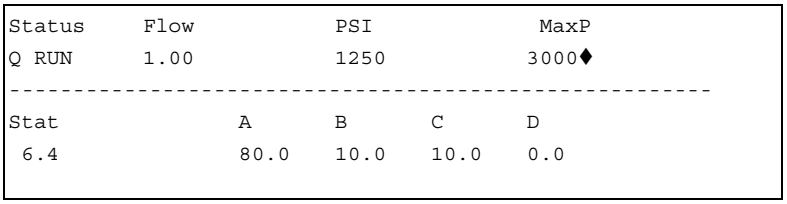


Figure 4.16 A P4000 Status Screen when a queue is running

As always, the Status Menu shows the run file. The run file can be edited from the Status Menu (if Status Lock is Off), as normal.

EDITING A RUNNING QUEUE

You can edit a running queue in order to add, delete, or edit lines (File, #Runs). All lines of a running queue except the Order 1 line are editable. Refer to the procedure outlined in the Edit section on page 71 to edit the queue. If you need to make a change to the Order 1 line of a running queue, you must first pause the queue as described in *Using a Pause Command* on page 75. Note that the pump will always finish the current run before pausing.

EDITING A FILE IN THE QUEUE

You can edit any file in the queue that has not yet been run by selecting /FILES/, /Edit/. Since the pump only loads the file in Order 1 once, any changes made to the file specified in Order 1 do not take effect while the queue is running. If the same file is specified later in the queue, then the changes will be recognized, since the edited file is loaded at a later time. To edit the file shown in Order 1, you can either edit the run file from the Status Menu, or pause the queue (see page 75) then edit the file. If you pause the queue (and the #Runs for Order 1 is greater than 1), the edited file will be loaded as soon as the queue is re-loaded.

LOADING OTHER FILES

When a queue is running, you may not load any other file from the Files Menu without first pausing or deleting the queue. If you try to load a file while a queue is running, the information message shown in Figure 4.17 appears. You are then returned to the Files Menu. As described on page 75 you can load another file *into the pump* by first pausing the queue. You can load another file *into the queue* by editing the queue.

```
** Queue Loaded **
 Cannot Load File
```

Figure 4.17 File load error message when the queue is loaded

STOPPING A QUEUE

There are several ways to stop a queue, depending on what you wish to do next.

NOTE: You do not need to stop the queue in order to edit it.

Using a Hold Command

If you want to stop the pump's clock momentarily and plan to resume the run in the queue, press [MENU], and select /COMMANDS/, /Hold/, to hold the pump at the current compositions. The pump will hold until a Continue command is issued.

To resume, select /COMMANDS/, /Continue/.

Using a Pause Command

If you want to finish the current run, but then pause the queue so that the pump can run another file, or so that you can edit the Order 1 line of the queue, press [MENU] and select /QUEUE/, /Pause/. The /Pause/ selection is only present if the queue is running. Remember, you can always edit the queue itself to move a particular file into the queue, but you cannot edit the first line of the queue if the queue is running. Whenever the queue is paused, the letter Q will disappear from the Status Screen.

Use /Pause/ if you need to interrupt the running of a queue for the purpose of relegating the pump to another task.

To resume running the queue, re-load the queue by selecting /QUEUE/, /Load/. When the Status Screen shows QREADY, press [RUN] to start the queue.

By Pressing [STOP]

You can stop the current run in the queue by pressing the [STOP] key. The pump will immediately stop, and the clock will be reset to zero. You may restart the same run by initializing the run file by pressing [RUN]. (The run file will be the last file in Order 1 - remember, the queue automatically re-sorts the queue after all the runs of a file have been performed.) When the pump shows QREADY, restart the run as you normally would. The pump continues its operation based on the queue.

By Resetting the Pump's Clock

Another way to reset the pump's clock and to restart the current run is to press [MENU] and select /COMMANDS/, /Reset/. When the Status Screen shows QREADY, restart the run by pressing [RUN].

By Aborting a Queue

You may abort the queue by deleting it. To do this select /QUEUE/, /Delete/. The current run of a deleted queue will be completed and the file in Order 1 will remain the run file.

The Develop File

INTRODUCTION

The Develop File is a flexible and powerful tool that can help you automate methods development. Available only on the P4000, this file is used to "program" a comprehensive set of fixed and changing parameters that includes solvent composition, gradient curves, and run time. By letting the pump systematically increment these parameters, and by studying the resulting chromatogram(s), you can identify the optimum set of conditions for obtaining your best chromatogram or for focusing on a single peak. We recommend that only chromatographers experienced in methods development use the Develop File.

The Develop File sets up the beginning and ending solvent composition parameters, and specifies a solvent interval (isocratic mode) or curve (gradient mode) by which those conditions will change from run to run. Essentially, you "program" the Develop File to perform the first run at one set of conditions and to automatically

increment those conditions through successive runs. All solvent proportioning and switching is done based on the "program" you set up in the file.

For example, in isocratic mode, using two solvents, you might specify that solvent A start at 20% and end at 100% (the pump automatically adjusts solvent B's composition to begin at 80% and end at 0%), with the time interval set at 10%. The pump would then set up nine runs, with each isocratic composition being different from the previous one. Table 4.1 illustrates the resulting solvent composition for each run in an isocratic, two-Solvent Develop file.

Table 4.1 Solvent Composition For Each Run In An Isocratic, Two-Solvent Develop File

Run #	Solvent A%	Solvent B%
1	20	80
2	30	70
3	40	60
4	50	50
5	60	40
6	70	30
7	80	20
8	90	10
9	100	0

The flow rate, run time, and equilibration time you specify remain constant from run to run.

You won't build a file whose menu, line by line, looks at all like Table 4.1, though. Instead, you will simply specify all the starting and ending parameters and any interval, along with other typical file parameters such as flow rate and run time. From these, the pump will automatically deliver solvents at the proper proportions.

The example in Table 4.1 is very basic. We will present many examples in this chapter to help you understand exactly what can be accomplished with the Develop File.

You should become familiar with an isocratic develop file before you read about creating a gradient develop file. The examples in the first half of this section are designed to provide you with a foundation of isocratic program editing and then continue to the more advanced editing features of gradient programs.

At the end of this chapter are form sheets for you to copy and use in planning your own Develop File. They list all of the fields shown in each display. There are six different sheets: three for isocratic development (2, 3, or 4 solvents) and three for gradient development (2, 3, or 4 solvents).

Creating a Develop File Program

To edit the Develop File, select [MENU], /FILES/, /Edit/ and choose file D which is called DEVELOP. This filename is permanent and cannot be changed (Figure 4.18).

```
Edit File                                    D
File Name                                DEVELOP
-------------------------------------------------------
>Setup
·Options
·Timed Events
·Preview
```

Figure 4.18 The Edit Menu of a Develop File

The menu selections are similar to those found in regular file editing, except that Solvent Program is replaced by Setup, and a Preview selection is added (Figure 4.18). The Setup Menu should be edited first, as the Options Menu is affected by selections made in the Setup Menu.

Program Modes

The Develop File operates in either of two program modes: isocratic or gradient. The fields displayed in the Setup Menu will vary based on the mode you select on the first line of the Setup Menu, and on the number of solvents you specify on the second line.

SETUP

When you select /Setup/, a display similar to the one shown in Figure 4.19 appears.

```
Program                                  Isocratic
Number of Solvents                        2
```

Figure 4.19 The first lines of a Develop File's Setup Menu

Program
Number of Solvents

Select either Gradient or Isocratic in the Program field. Then choose the number of solvents you want to use in the Number of Solvents field. You must always use at least two solvents.

NOTE: Verify the program mode and the number of solvents you intend to use before going any further into Setup or going into the Options Menu. Although there are some common fields between Setup Menus, not all entries you make will be retained if either the program mode or the number of solvents is later changed. You cannot retrieve your previous menu settings by returning the Program/Number of Solvents settings to their earlier values.

Common Fields in the Setup Menu

The Setup Menu in gradient (grad) mode is similar to the Setup Menu in isocratic (iso) mode, but it is not identical. There are minor differences between the Options Menu in both modes, too. In several instances menus contain fields with identical names, in other instances, field names are different, but imply a very similar function.

The fields of an isocratic, two-solvent program (Figure 4.20) are described below. These fields provide the foundation for all other isocratic and gradient programs. Other fields, specific to certain Program/Number of Solvent combinations, are explained below each menu in the *Other Isocratic Programs* (page 80) and *Gradient Programs* (page 84) sections.

Iso / Two Solvents

The two-solvent isocratic Setup Menu is shown in Figure 4.20. All of the fields in this Setup Menu are explained below.

```
Program                              Isocratic
Number of Solvents                           2
-----------------------------------------------------------
Solvent 1                           A
Solvent 2                           B
Start      %       (Solvent 1)                0
End        %       (Solvent 1)              100
% Interval                          20
Flow Rate                          1.0
Run Time                                  10.0
```

Figure 4.20 The Develop File's Setup Menu showing some common fields

Solvent 1

Select one of the four solvents to be designated as your first solvent.

Solvent 2

Select one of the four solvents to be designated as your second solvent.

NOTE: The pump does not allow you to select the same solvent for Solvent 1 and Solvent 2.

Start %
End %

Enter values for the beginning and ending percentages of the first solvent. This would be solvent A in the menu shown in Figure 4.20 because A was selected to be Solvent 1.

% Interval

Set an interval by which the first solvent will change (increase or decrease, depending on the start and end percentages) for every successive run. The pump will automatically set up each successive run to increase or decrease the first solvent by this percent. The pump automatically adjusts the second solvent accordingly.

Flow Rate
Run Time

Enter values as desired.

OTHER ISOCRATIC PROGRAMS

This section describes fields that are specific to three- and four-solvent isocratic Setup Menus.

Iso / Three Solvents

When using three solvents in an isocratic program, the Setup Menu (Figure 4.21) asks for more solvent information than when only a two-solvent program is used.

```
Program                              Isocratic
Number of Solvents                           3
-------------------------------------------------------
Solvent Not Used                             D
Solvent 1                           A
Start      %       A                         0
End        %       A                       100
% Interval                          20
Solvents 2,3                 B               C
Relative Start %            50              50
Relative End %               0              100
Relative % Interval                          20
Flow Rate                          1.0
Run Time                                  10.0
```

Figure 4.21 The three-solvent isocratic Setup Menu

Solvent Not Used

Select the solvent that you will not use.

NOTE: The pump does not allow you to select the same solvent for Solvent Not Used and Solvent 1.

Solvents 2,3

The Solvents 2,3 field is filled in automatically, based on the solvents remaining after selections have been made in the Solvent Not Used field and the Solvent 1 field. This line simply labels to Solvents 2 and 3 for you.

RelativeStart %

There are two fields on the RelativeStart % line. These correspond to Solvents 2 and 3. The relative start percentages for the two solvents always add to 100. You enter a value for Solvent 2. Solvent 3 is entered automatically. The values in these fields are *relative* to Solvent 1's composition.

For example, if the Start % of Solvent 1 were 60%, then the remaining 40% would be comprised of a combination of solvents 2 and 3. If you want the relative ratio of Solvents 2 and 3 to be 50 / 50, you would enter a RelativeStart % of 50 for Solvent 2. Then, the *actual* proportions delivered by the pump would be:

Solvent 1	60%	
Solvent 2	20%	(40% × 0.5 = 20)
Solvent 3	20%	(40% × 0.5 = 20)

The total of all solvents is 100%.

If solvents 2 and 3 were not entered as 50 / 50, but 20 / 80 instead, then the *actual* proportions delivered by the pump would be:

Solvent 1	60%	
Solvent 2	8%	(40% × 0.2 = 8)
Solvent 3	32%	(40% × 0.8 = 32)

You do not need to make these calculations yourself to see what the pump will actually do. After you complete your program in the Setup Menu, you can preview the composition of the resulting runs by selecting /Preview/ as described in conjunction with several examples beginning on page 87.

Relative End %

Enter a value for the relative end % of Solvent 2. Solvent 3 is entered automatically.

Relative % Interval

Enter a value for the relative percent increment from run to run of Solvents 2 and 3. If you do not wish to increment Solvents 2 and 3, make sure the Relative End % is the same as the RelativeStart %.

More Information About Relative Percentages

When you set up a program to increment the relative percents of solvents 2 and 3, you are creating a "sub-loop" in the program. This "sub-loop" is created because Solvent 1, itself, has been set up to increment from run to run.

The set of runs that the Develop File creates in these situations goes like this:

- In the first run, the proportions of solvents 1, 2, and 3 are taken directly from the Setup Menu.
- In the next runs, Solvent 1 will maintain its initial composition, while Solvents 2 and 3 increment. Solvent 1 will maintain its initial proportion for as many runs as are necessary, while Solvents 2 and 3 are systematically incremented.
- Only when the "sub-loop" is complete (*i.e.,* when Solvents 2 and 3 reach their specified Relative End % values), will Solvent 1 change by its increment value.
- Then the sub-loop of Solvents 2 and 3 begins again, as Solvent 1 remains fixed at its new composition.

Iso / Four Solvents

The isocratic four-solvent case is similar to the isocratic three-solvent case. However, when four solvents are specified (Figure 4.22), one of the four solvent compositions must remain fixed. The entries made in the Setup Menu determine how Solvents 1, 2, and 3 vary to comprise the percentage difference (100% - Fixed Solvent %) for each run.

```
Program                              Isocratic
Number of Solvents                           4
-----------------------------------------------------
Fixed Solvent                                D
Fixed Solvent %                             20
Solvent 1                             A
Start      %       A                         0
End        %       A                       100
% Interval                          20
Solvents 2,3                  B              C
RelativeStart %             100              0
Relative End %                0            100
Relative % Interval                         20
Flow Rate                           1.0
Run Time                                  10.0
```

Figure 4.22 The four-solvent isocratic Setup Menu

Fixed Solvent
Select the solvent whose composition will remain fixed.

Fixed Solvent %
Enter a value for the fixed solvent composition.

OPTIONS MENU (ISOCRATIC)

The isocratic Options Menu (Figure 4.23) is used to set all of the same parameters as that of normal files, with three additions. Note that the Options Menu in isocratic mode is different than in gradient mode. The Options Menu for gradient programs is described on page 86.

Access the Options Menu by selecting /Options/ from the Edit Menu. The Options Menu is the same for any isocratic program, regardless of the number of solvents specified in the Setup Menu.

Equilibration Time	0.0
#Runs/Interval	1
#Develop Cycles	1
Maximum Pressure	3000
Minimum Pressure	0
Total # of Runs	*(Not editable, filled in automatically)*

Figure 4.23 An isocratic program's Options Menu

Equilibration Time
Enter a value for the equilibration time. It is very important to set an equilibration time in the Develop File so that the column can equilibrate prior to the beginning of the next run.

#Runs/Interval (Develop File)
Enter the number of times you want each run repeated, before the solvents' compositions are incremented. (An interval, in this case, refers to one set of solvent compositions.)

#Develop Cycles (Develop File)
Select the number of times (9 maximum) the entire program will be executed. Usually this is only once (1), but you might enter another number if you want to run the entire program again using a different column, or, if you have a column oven, at a different column temperature.

Maximum Pressure
Minimum Pressure
Enter values for maximum and minimum pressure levels, if desired.

Total # of Runs

The Total # of Runs field is filled in automatically, based on the program entered in the Setup Menu, and the values specified in the #Runs/Interval and the #Develop Cycles fields. The pump can contain as many as 999 runs in the Develop File.

GRADIENT PROGRAMS

The gradient program mode is used to program the pump to perform a series of varying gradient runs. Unlike the isocratic mode where you increment Solvent 1 by a percentage, in the gradient mode you specify the total number of gradient curves you want performed. During each successive run, the pump follows a different gradient shape (for example., Linear, Convex 2, then Concave 2). Note that this is not the same as specifying the curve itself (that is., Concave 7 or Convex 4), as you do in regular files.

You vary the relative percentages of Solvents 2 and 3 just as you do in the isocratic program mode, except that, when the pump performs the gradient, the other solvents also follow gradient shapes.

This section describes the fields that are specific to two-, three-, or four-solvent gradient Setup Menus. The fields that are common to all Setup Menus are described on page 79.

Grad / Two Solvents

```
Program                                  Gradient
Number of Solvents                              2
-------------------------------------------------------------
Solvent 1                               A
Solvent 2                               B
Start      %       A                            0
End        %       A                          100
#Curves                                         1
Flow Rate                              1.0
Run Time                                     10.0
```

Figure 4.24 The two-solvent gradient Setup Menu

#Curves (Develop File)

Select the total number of gradient shapes you want the pump to run. If, for example, you select 1, as in Figure 4.24, then the curve would be "Linear" - a straight line from 0% to 100% over the course of the run. If, instead, you select 3, then three runs would be performed: Linear, Concave 2, and Convex 2. shows the curves that correspond to each numerical selection. Refer to Chapter 3 to see the shape of each curve.

Table 4.2 The Number Of Curves Selected And The Corresponding Gradient Shapes Performed

#Curves	Shapes, Performed
1	Line
3	Line, Cvx2, Ccv2
5	Line, Cvx2, Ccv2, Cvx3, Ccv3
7	Line, Cvx2, Ccv2, Cvx3, Ccv3, Cvx5, Ccv5
9	Line, Cvx2, Ccv2, Cvx3, Ccv3, Cvx5, Ccv5, Cvx7, Ccv7
11	Line, Cvx2, Ccv2, Cvx3, Ccv3, Cvx5, Ccv5, Cvx7, Ccv7, Cvx10, Ccv10

(Line = linear, Ccv = concave, Cvx = convex)

Grad / Three Solvents

During a three-solvent gradient run, the *ratio* of solvents 2 and 3 remains constant. As the gradient is generated, it is Solvent 1's ratio to Solvent 2 and its ratio to Solvent 3 that changes. This is commonly called a solvent strength gradient. In successive runs, Solvents 2 and 3 take on a new ratio, which is maintained as the gradient is generated.

```
Program                                  Gradient
Number of Solvents                              3
-------------------------------------------------------
Solvent Not Used                                D
Solvent 1                                A
Start      %       A                            80
End        %       A                            20
Solvents 2,3                B                    C
RelativeStart %           100                    0
Relative End %              0                  100
Relative % Interval                             20
#Curves                                          1
Flow Rate                                 1.0
Run Time                                       10.0
```

Figure 4.25 The three-solvent gradient Setup Menu

All the fields in this menu have been explained in the previous pages.

Grad / Four Solvents

When four solvents are specified (Figure 4.26), one of the four solvents' compositions must remain fixed. The entries made in the Setup Menu determine the ratios between Solvents 1, 2, and 3 that will be maintained as each gradient curve is performed.

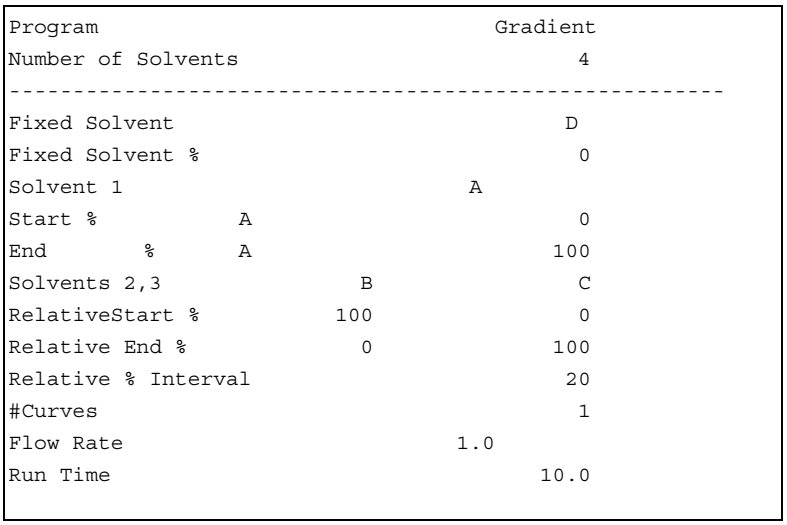


Figure 4.26 The four-solvent gradient Setup Menu

All fields in this menu have also been explained previously.

OPTIONS MENU (GRADIENT)

The gradient Options Menu (Figure 4.27) is used to set all of the same parameters as those found in an isocratic Options Menu, with two differences, #Runs/Curve and Run Time Increment. To access the Options Menu select /Options/ from the Files Menu. The Options Menu is the same for all gradient programs, regardless of the Number of Solvents specified in the Setup Menu. (Note the Options Menu for an isocratic program is different from a gradient program. Refer to page 83 for the isocratic Options Menu.)

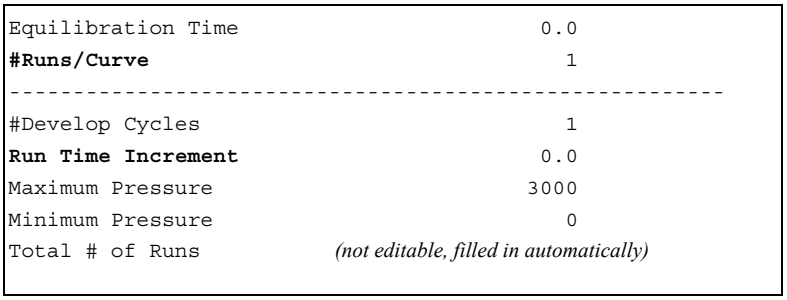


Figure 4.27 A gradient program's Options Menu

#Runs/Curve (Devlop File)

Select the number of times you want each run of a gradient shape performed, before the solvent parameters are incremented.

Run Time Increment

Enter a value (in minutes) to increment the run time of multiple develop cycles. The Run Time Increment only operates if there is

more than one develop cycle (#Develop Cycles). After the pump completes an entire cycle of runs, it increments the Run Time by the time entered here.

For example, if you specify five develop cycles, a run time of 10.0 minutes, and a Run Time Increment of 5.0 minutes. The resulting run times for each develop cycle will be those shown in Table 4.3.

Table 4.3 An example showing the use of Run Time Increment and five develop cycles

Cycle	Run Time
Each run in Cycle 1	*10.0 minutes*
Each run in Cycle 2	*15.0 minutes*
Each run in Cycle 3	*20.0 minutes*
Each run in Cycle 4	*25.0 minutes*
Each run in Cycle 5	*30.0 minutes*

Develop File Examples

This section describes the Preview Screen and presents seven different examples of isocratic and gradient programs. The Setup Menu for each example program is presented, along with a preview of each run, and a graph showing solvent compositions from run to run. The last section of this chapter contains form sheets for planning and recording your own Develop File, as well as a Preview Record on which you can record the composition of each run shown in the Preview Screen.

PREVIEW SCREEN

Once you have built a program using the Setup and Options Menus, you can preview the programmed run compositions by selecting /Preview/ (Figure 4.28).

```
Edit File                                   D
File Name                                DEVELOP
-------------------------------------------------------
·Setup
·Options
·Timed Events
>Preview
```

Figure 4.28 The Develop File's Edit Menu, with Preview selected

The Preview Screen differs, depending on the program mode. An isocratic Preview Screen is shown in Figure 4.29; a gradient Preview Screen is shown in Figure 4.30.

```
Isocratic          Cyc:1              IntRun#1
80.0       20.0           0.0            0.0     #  1
```

Figure 4.29 An isocratic Preview Screen

```
Gradient                  Cyc:1              CrvRun#1
0.0        100            0.0            0.0     #  1
-------------------------------------------------------
100.0       0.0           0.0            0.0     Line
```

Figure 4.30 A gradient Preview Screen

Selecting Runs

There is only one active cursor location (field) in the Preview Screen: the far-right field, underneath the word IntRun# (isocratic) or CrvRun# (gradient). Use the [+] / [-] keys in this field to select a run number to preview. The solvent compositions for each run will be displayed to the left, in the solvent composition fields, as you select run numbers. Note that the figures showing Preview Screens in the rest of this chapter list subsequent runs under a dashed line. (Normally, the dashed line indicates that you press [∨] to view subsequent lines.)

The gradient Preview Screen contains one additional line for each run number, accessed by using the down-arrow key. This line shows the ending solvent compositions and, in the far-right corner, indicates the gradient curve shape for that run. Refer to Chapter 3 for a description of gradient curve shapes. "Ccv" denotes concave, "Cvx" denotes convex, and "Line" denotes linear.

Program Mode

The program mode is shown in the left-hand corner of the first line.

Cycle

The number corresponding to the develop cycle for the selected run is shown in the middle of the top line.

IntRun# , CrvRun#

The number immediately following the word "IntRun#" and "CrvRun#" at the top, right-hand corner, changes automatically if more than one run per interval or one run per curve has been selected. You will see this number change as you increment and decrement the run number.

The Preview Screen shows all runs for a given program even when the Develop File is running. The preview screen only changes when the Develop File is edited.

EXAMPLE 1
ISO / TWO SOLVENTS

This example shows a two-solvent isocratic program (Figure 4.31). Solvent A starts at 20% and will increment 20% for each run until it reaches the last run at 80%. The total number of runs is four (4).

Setup

This example assumes that the #Runs/Interval = 1 and the #Develop Cycles = 1.

```
Program                                  Isocratic
Number of Solvents                               2
-------------------------------------------------------------
Solvent 1                               A
Solvent 2                               B
Start      %       A                            20
End        %       A                            80
% Interval                             20
Flow Rate                             1.0
Run Time                                      10.0
```

Figure 4.31 The Setup Menu for Example 1

Preview

The Preview Screen for this program is shown in Figure 4.32.

```
Isocratic          Cyc:1                IntRun#1
20.0       80.0            0.0              0.0     #  1
-------------------------------------------------------------
40.0       60.0            0.0              0.0     #  2
60.0       40.0            0.0              0.0     #  3
80.0       20.0            0.0              0.0     #  4
```

Figure 4.32 The Preview Screen for Example 1

The graph below shows how solvents A and B change each time a new run begins. Note that the dashed line in the isocratic graphs indicate that the pump changes solvent composition between the end of one run and the beginning of the next. Solvent composition does not change during the run in an isocratic program.

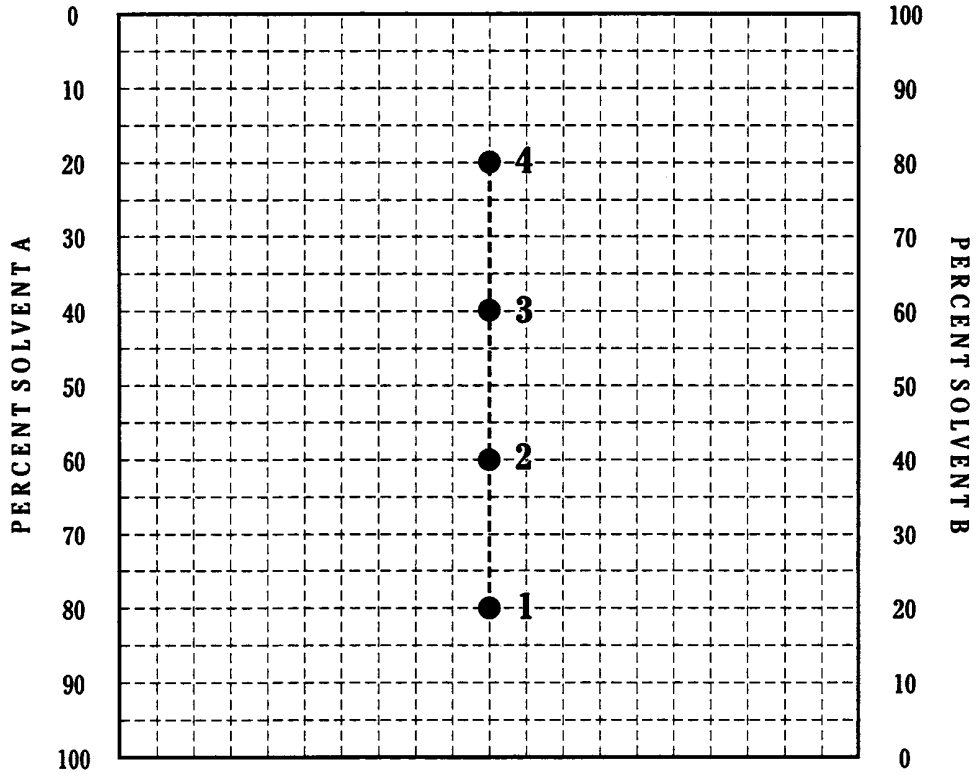


Figure 4.33 The two-solvent composition path for Example 1

EXAMPLE 2
GRAD / TWO SOLVENTS

This example shows a simple gradient program using two solvents (Figure 4.34). Solvent A starts at 80% and ends at 20%. One curve shape, linear, is performed over the 10.0 minute run time. The total number of runs in this program is one (1).

Setup

This example assumes that the #Runs/Curve = 1, the #Develop Cycles = 1, and the #Curves = 1.

```
Program                                  Gradient
Number of Solvents                              2
-----------------------------------------------------------
Solvent 1                               A
Solvent 2                               B
Start      %       A                           80
End        %       A                           20
#Curves                                         1
Flow Rate                              1.0
Run Time                                     10.0
```

Figure 4.34 The Setup Menu for Example 2

Preview

```
Gradient                  Cyc:1                  CrvRun#1
80.0       20.0           0.0            0.0     # 1
-----------------------------------------------------------
20.0       80.0           0.0            0.0     Line
```

Figure 4.35 The Preview Screen for Example 2

Since only one curve is specified, the Develop File in this example is identical to a two time-line Solvent Program, which specifies a linear gradient curve.

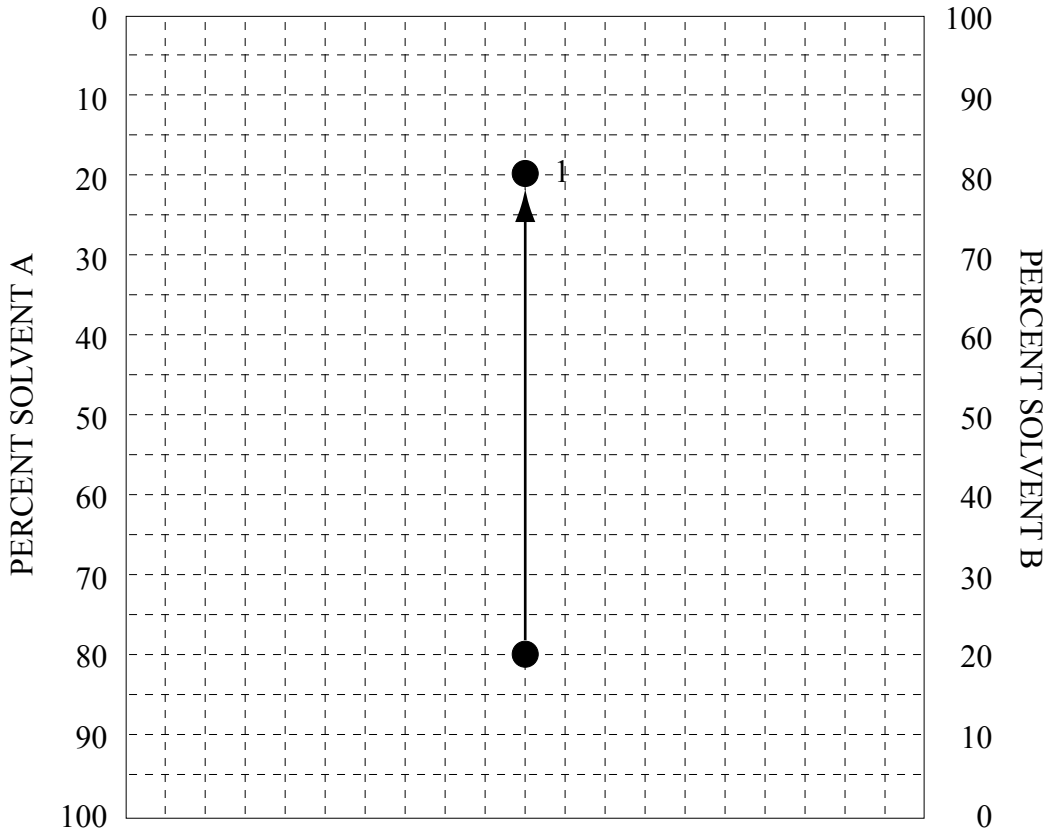


Figure 4.36 The two-solvent gradient composition path for Example 2

Notice that the solid line in the gradient graphs indicates that the pump changes solvent composition during the run.

EXAMPLE 3
GRAD / TWO SOLVENTS WITH MULTIPLE CURVES

Example 3 (Figure 4.37) is similar to Example 2, except that five curves are specified. The total number of runs in this program is five (5).

Setup

This example assumes the #Runs/Curve = 1, the #Develop Cycles = 1, and the #Curves = 5.

```
Program                                  Gradient
Number of Solvents                            2
-------------------------------------------------------------
Solvent 1                               A
Solvent 2                               B
Start      %       A                          100
End        %       A                            0
#Curves                                         5
Flow Rate                              1.0
Run Time                                     10.0
```

Figure 4.37 The Setup Menu for Example 3

Preview

```
Gradient                    Cyc:1                 CrvRun#1
100.0        0.0             0.0             0.0     # 1
  0.0      100.0             0.0             0.0     Line
-------------------------------------------------------------
 100.        0.0             0.0             0.0     # 2
  0.0      100.              0.0             0.0     Cvx2
-------------------------------------------------------------
 100.        0.0             0.0             0.0     # 3
  0.0      100.              0.0             0.0     Ccv2
-------------------------------------------------------------
 100.        0.0             0.0             0.0     # 4
  0.0      100.              0.0             0.0     Cvx3
-------------------------------------------------------------
 100.        0.0             0.0             0.0     # 5
  0.0      100.              0.0             0.0     Ccv3
```

Figure 4.38 The Preview Screen for Example 3

If, in the Options Menu, we had specified the #Runs/Curve = 2, then a total of ten runs would have been seen in the Preview Screen instead of five.

The graph in Figure 4.39 illustrates the gradient curves performed in this example.

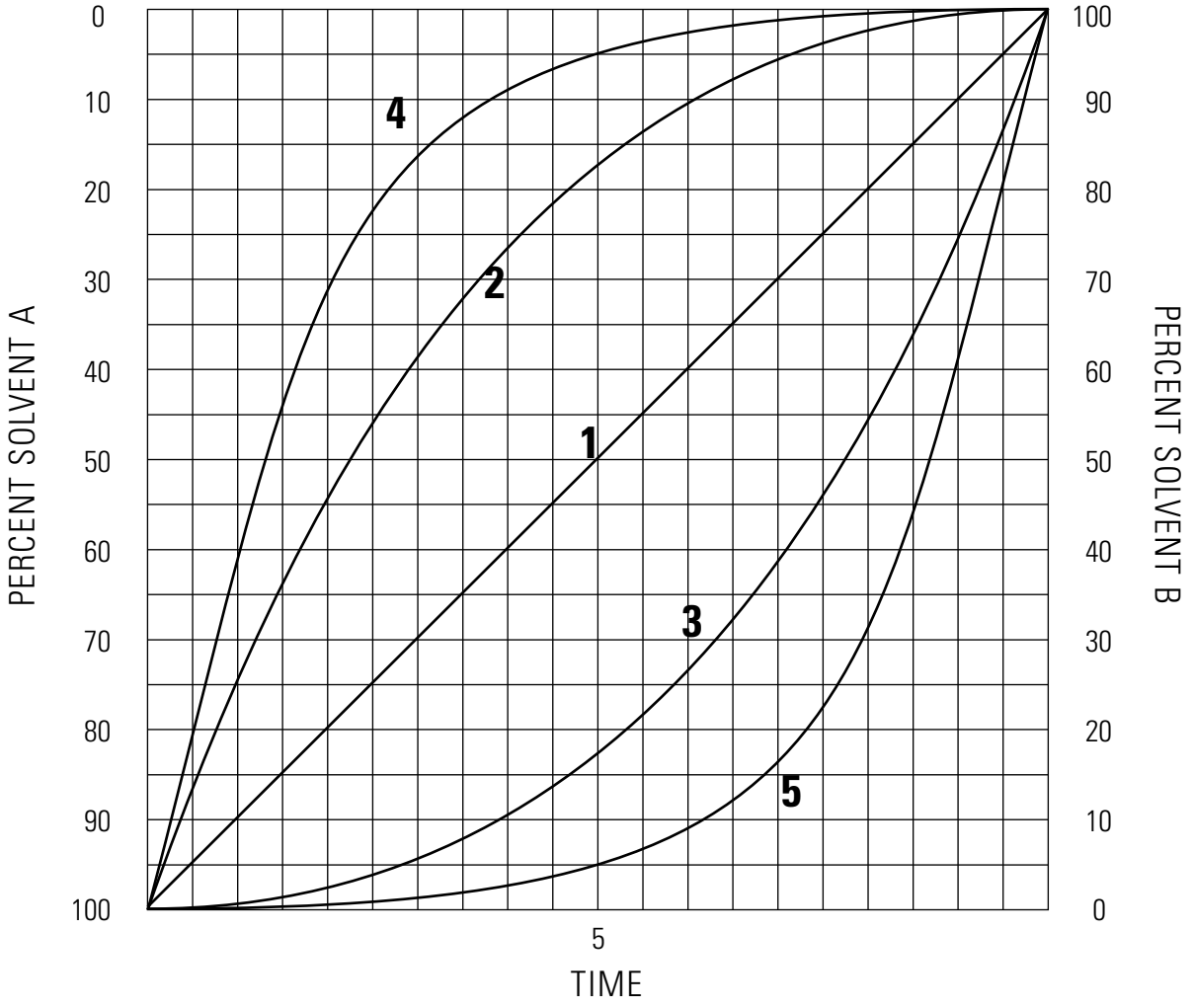


Figure 4.39 The two-solvent gradient curve composition path for Example 3

EXAMPLE 4 GRAD / TWO SOLVENTS, WITH RUN TIME INCREMENT

Example 4 (Figure 4.40) shows how a run time increment can be used in the gradient program mode. The Setup Menu is similar to Example 4, but the Options Menu in this example specifies a Run Time Increment of 10.0 minutes, and four (4) Develop Cycles. The total number of runs created by this program is four (4).

Setup

This example also assumes the #Runs/Curve = 1, and the #Curves = 1.

```
Program                                Gradient
Number of Solvents                            2
-------------------------------------------------------------
Solvent 1                             A
Solvent 2                             B
Start      %       A                         80
End        %       A                         20
#Curves                                       1
Flow Rate                             1.0
Run Time                                   10.0
```

Figure 4.40 The Setup Menu for Example 4

Options

```
Equilibration Time                          0.0
#Runs/Curve                                   1
-------------------------------------------------------------
#Develop Cycles                               4
Run Time Increment                         10.0
Maximum Pressure                           3000
Minimum Pressure                              0
Total # of Runs                               4
```

Figure 4.41 The Options Menu for Example 4

Preview

```
Gradient                    Cyc:1                CrvRun#1
80.0        20.0            0.0          0.0     # 1
20.0        80.0            0.0          0.0     Line
(10.0 minute run time)
------------------------------------------------------------
80.0        20.0            0.0          0.0     # 2
20.0        80.0            0.0          0.0     Line
(20.0 minute run time)
------------------------------------------------------------
80.0        20.0            0.0          0.0     # 3
20.0        80.0            0.0          0.0     Line
(30.0 minute run time)
------------------------------------------------------------
80.0        20.0            0.0          0.0     # 4
20.0        80.0            0.0          0.0     Line
(40.0 minute run time)
```

Figure 4.42 The Preview Screen for Example 4

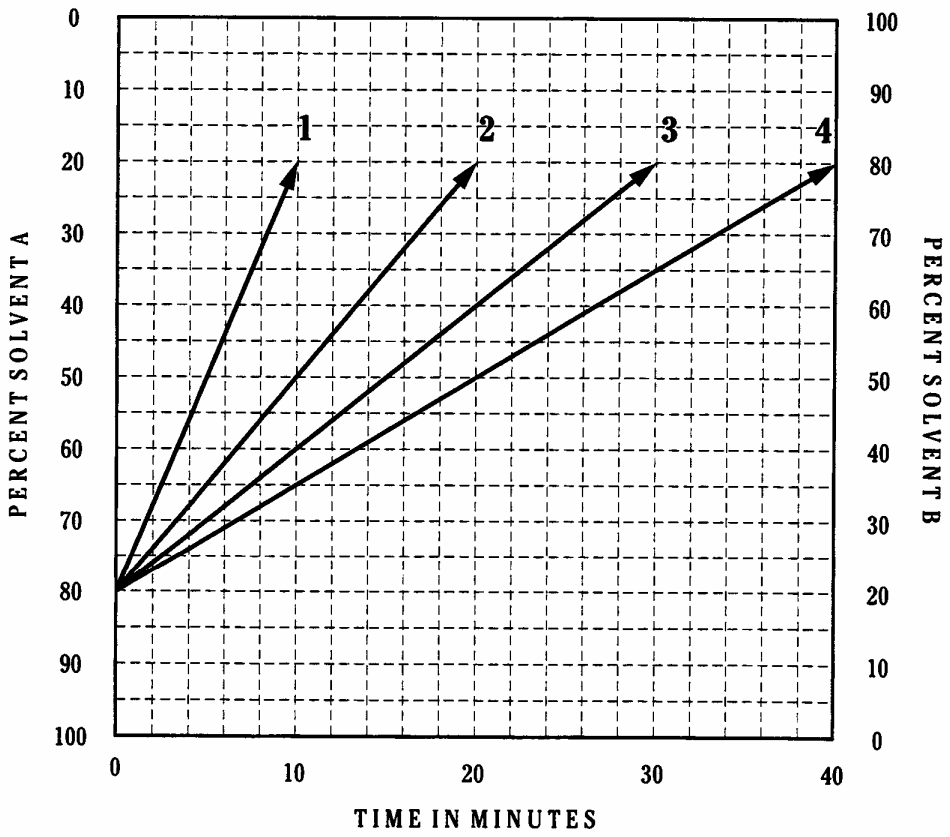


Figure 4.43 The composition paths for a two-solvent gradient with run time increment for Example 4

Running the Develop File

LOADING THE DEVELOP FILE

Load the Develop File just as you would any other file (/FILES/, /Load/). If the total number of runs programmed by the Develop File exceeds 999, a message will indicate that this maximum has been reached, preventing the file from being loaded successfully. If the message appears, edit the Develop File to reduce the total number of runs. The following fields affect the total number of runs:

% Interval	(Setup Menu)
Relative % Interval	(Setup Menu)
#Curves	(Setup Menu)
#Develop Cycles	(Options Menu)
#Runs/Interval or #Runs/Curve	(Options Menu)

After you make changes you can double-check the total number of runs from the Options Menu.

STATUS

The Status Screen for an example Develop File is shown in Figure 4.44. Note that all four solvents are automatically displayed, regardless of the Number of Solvents specified in the Setup Menu.

```
Status            Flow            PSI           MaxP
 READY            1.00           1250           6000▼
-------------------------------------------------------
Stat         A              B              C       D
 0.0       20.0           80.0            0.0     0.0

File  D:DEVELOP

Time         A              B              C      Flow
0.0        20.0           80.0           0.0     1.00
10.0       70.0           30.0           0.0     1.00

Maximum Pressure                                 6000
Minimum Pressure                                    0
Equilibration Time                                0.0
Gradient Curve                                Convex 3
#Run/Total#Runs                                  04/10
```

Figure 4.44 An example Status of a gradient Develop File

Status Menu

The Status Menu shows one new line, #Run/Total#Runs. This line displays the current run number and the total number of runs programmed in the file. The Gradient Curve field shows the curve shape of the current run.

The same Status Menu editing capabilities of a run file also apply when a Develop File is loaded, except that the run file may not contain more than two time-lines.

COMMANDS

All commands function as they would for any other file. However, the pump sees the Develop File as one file, not as a series of runs. This is important if you reset the file, using /COMMANDS/, /Reset/. When the reset command is issued, the pump will return to the initial conditions of Run #1. The entire program then restarts as soon as the pump receives a RUN command.

Develop File Review

The examples presented in the preceding pages give you a good idea of the flexibility and power of the Develop File. You can combine many of the concepts presented in these examples into your own Develop File, depending on your needs.

For instance, in a gradient program you might combine the run time increment with three or more curves, instead of one. The pump would perform all the curve shapes, then increment the run time and run all the shapes again.

In an isocratic program you might specify two develop cycles, instead of one, and run the second cycle with a different column. You could do the same thing with a gradient program.

The Develop File can greatly assist you in automating methods development. The form sheets supplied in the following pages can help you plan and document the conditions necessary for optimizing your chromatography.

Develop File Record

Date: _____

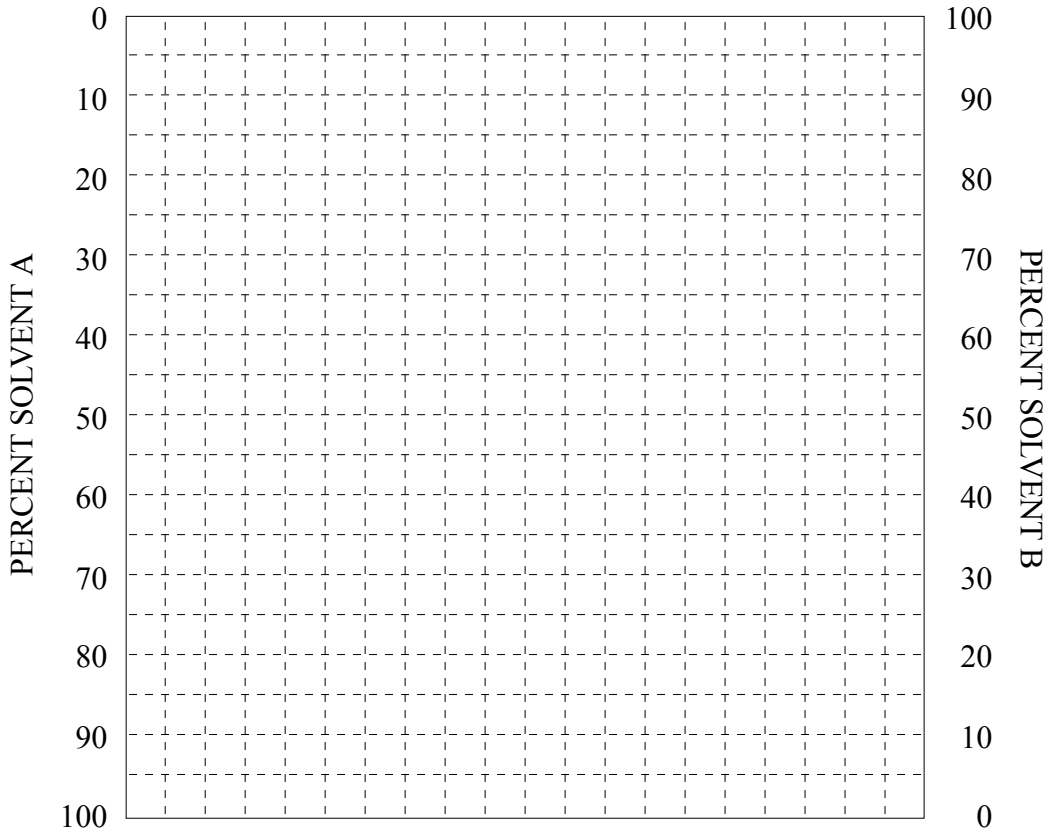
Setup

Program	**Isocratic**	
Number of Solvents	**2**	
Solvent 1		_____
Solvent 2		_____
Start % *(Solvent 1)*	*(0 - 100)*	_____
End % *(Solvent 1)*	*(0 - 100)*	_____
% Interval	*(1 - 100)*	_____
Flow Rate		_____
Run Time	*(0.0 - 650)*	_____

Options

Equilibration Time	*(0.00 - 99.9)*	_____
#Runs/Interval	*(1 - 9)*	_____
#Develop Cycles	*(1 - 9)*	_____
Maximum Pressure	*(1 - 6000 psi)*	_____
Minimum Pressure	*(0 - 5999 psi)*	_____
Total # of Runs	*(fill in from display)*	_____

NOTES:


PERCENT SOLVENT A
0
10
20
30
40
50
60
70
80
90
100
PERCENT SOLVENT B
100
90
80
70
60
50
40
30
20
10
0

Develop File Record

Date: _____

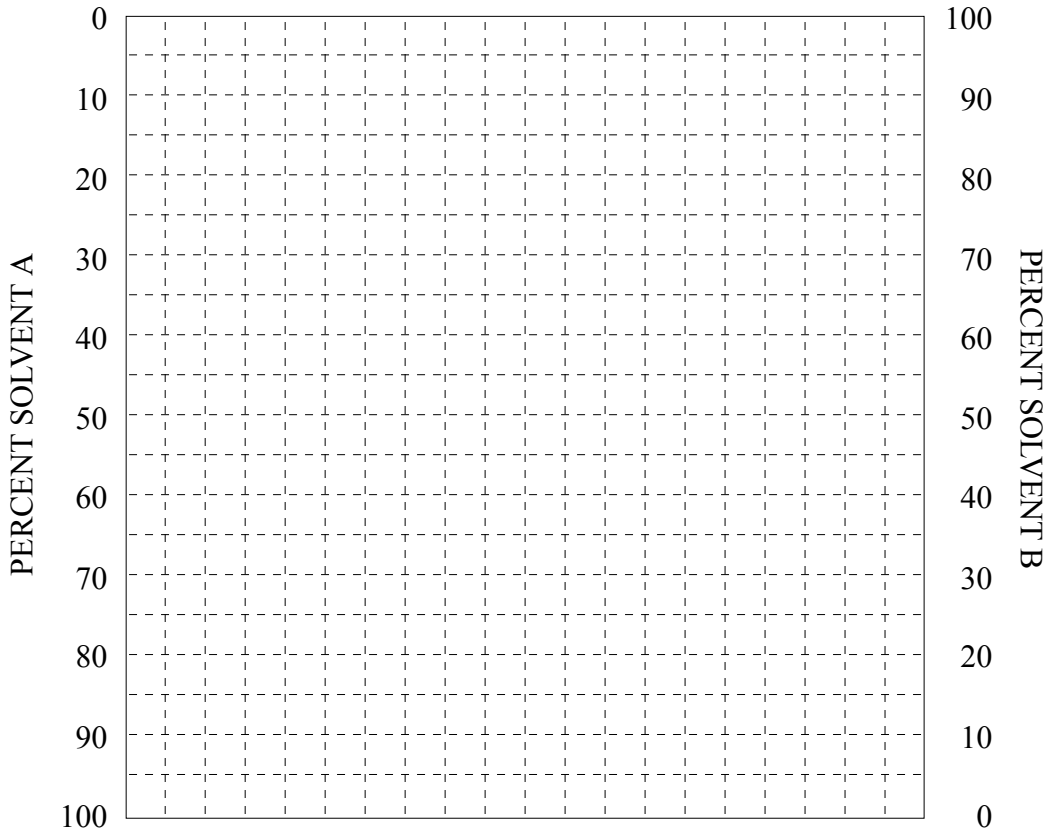
Setup

Program	**Gradient**	
Number of Solvents	**2**	
Solvent 1		_____
Solvent 2		_____
Start % *(Solvent 1)*	*(0 - 100)*	_____
End% *(Solvent 1)*	*(0 - 100)*	_____
#Curve	*(1,3,5,7,9,11)*	_____
Flow Rate		_____
Run Time	*(0.0 - 650)*	_____

Options

Equilibration Time	*(0.00 - 99.9)*	_____
#Runs/Curve	*(1 - 9)*	_____
#Develop Cycles	*(1 - 9)*	_____
Run Time Increment	*(0.0 - 650)*	_____
Maximum Pressure	*(1 - 6000 psi)*	_____
Minimum Pressure	*(0 - 5999 psi)*	_____
Total # of Runs	*(fill in from display)*	_____

NOTES:


PERCENT SOLVENT A
0
10
20
30
40
50
60
70
80
90
100
PERCENT SOLVENT B
100
90
80
70
60
50
40
30
20
10
0

Develop File Record

Date: _____

Setup

Program **Gradient**

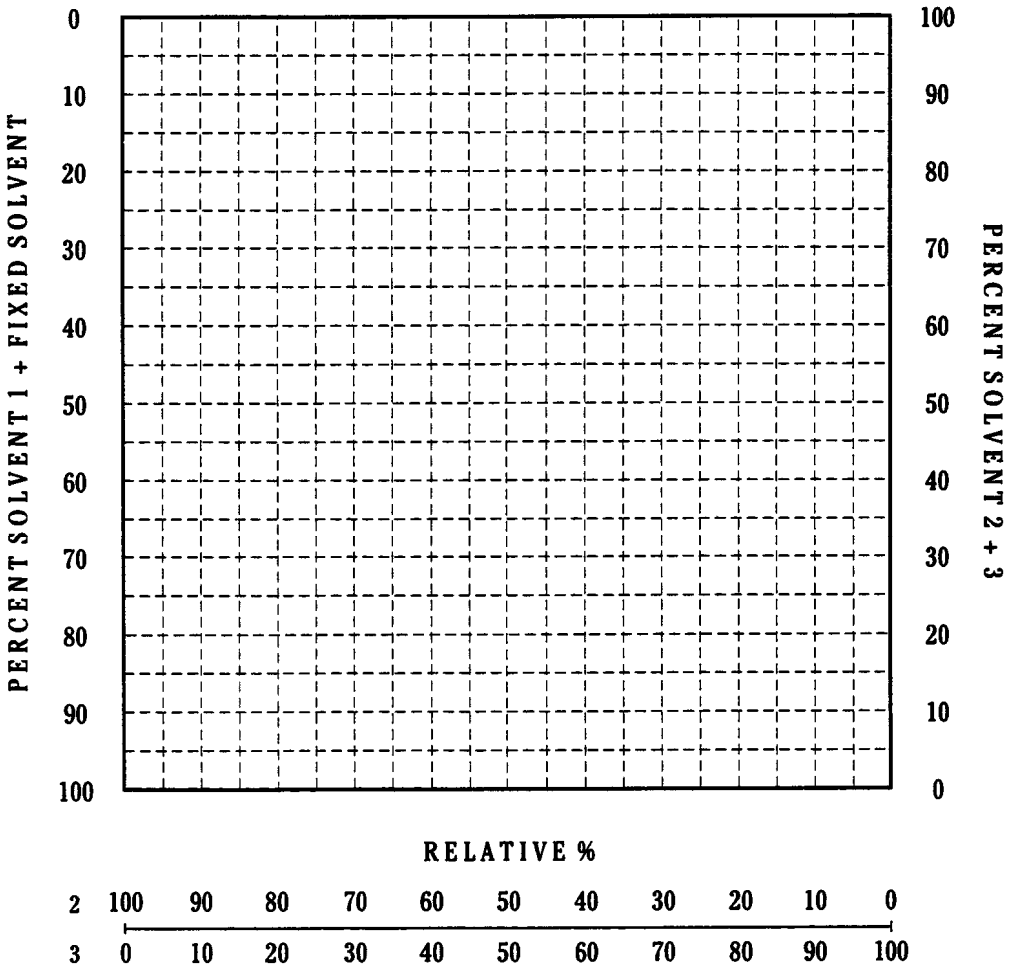
Number of Solvents **4**

Fixed Solvent			_____
Fixed Solvent % *(0 - 100)*		_____	
Solvent 1			_____
Start % *(Solvent 1)* *(0 - 100)*			_____
End % *(Solvent 1)* *(0 - 100)*			_____
Solvents 2,3		( )	( )
RelativeStart % *(0 - 100)*	_____	_____	
Relative End % *(0 - 100)*	_____	_____	
Relative % Interval *(1 - 100)*			_____
#Curves *(1,3,5,7,9,11)*		_____	
Flow Rate			_____
Run Time *(0.0 - 650)*			_____

Options

Equilibration Time *(0.00 - 99.9)*		_____
#Runs/Curve *(1 - 9)*		_____
#Develop Cycles *(1 - 9)*	_____	
Run Time Increment *(0.0 - 650)*		_____
Maximum Pressure *(1 - 6000 psi)*		_____
Minimum Pressure *(0 - 5999 psi)*		_____
Total # of Runs *(fill in from display)*	_____	

NOTES:



Fixed Solvent = _____, _____ %

Preview Record

Date: _____

Program Mode: _____ # Develop Cycles: _____

A: (_____)	B: (_____)	C: (_____)	D: (_____)	Run # or Shape

A: (_____)	B: (_____)	C: (_____)	D: (_____)Run # or Shape

The Tests Menu

The Tests Menu allows you to access the pump's built-in diagnostics, part of the pump's advanced features. This section assumes that the source of the problem is known to be the SpectraSYSTEM pump. If you are not certain that the pump is the source of trouble, refer to *General LC System Troubleshooting*, on page 159.

To access the Tests Menu, select /TESTS/ from the Main Menu (Figure 4.45):

Figure 4.45 Main Menu with /TESTS/ selected

The Tests Menu (Figure 4.46) consists of five items. Tests are divided into three specific menus, for convenience: diagnostic tests, calibration tests, and service tests. The Maintenance Log is described fully in Chapter 5.

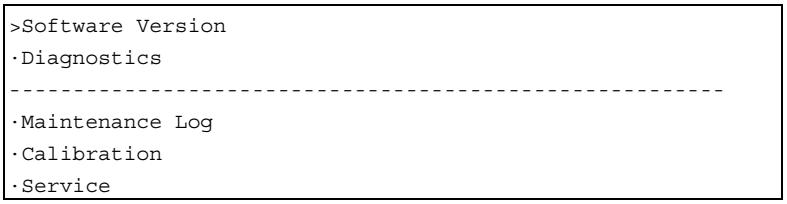


Figure 4.46 Tests Menu

ABOUT RUNNING TESTS

Tests are internal computer programs that exercise the pump's hardware and circuitry and verify operation. If any abnormal behavior is found it is reported as a message or an electronic circuit board failure code. In most cases the test isolates the problem to the failed module or component.

Active and Passive Tests

There are active and passive tests. Passive tests can be initiated at any time as they do not affect either file memory or pump performance. Passive tests are usually initiated by pressing [ENTER]. Active tests require that the pump be idle before being initiated, since the pump's valves and motor maybe engaged during the test. Usually, active tests are initiated by pressing [RUN], and are stopped by pressing [STOP]. Some tests stop by themselves. An active test should not be performed while the pump is in operation, as it will interfere with pump operation.

NOTE: Pressing [STOP] during a passive test can interrupt pump operation.

Initiating Tests

To initiate a test, move the cursor to the test's name and press [ENTER]. Always follow the instructions displayed on the pump when you initiate a test. In some cases the message, "Pump must be stopped to run test" may be displayed if you attempt to run an active test while the pump is in RUN. Alternately, if the pump is stopped when you initiate a test you may see the message, "The Pump Must Be Running to Perform This Test." In most cases, the pump will initiate an active test if the pump is in EQUIL or READY.

Usually you will press [ENTER] to initiate a passive test, or [RUN] to initiate an active test. Some tests display instructions. In these cases, follow the instructions in the message to proceed.

Test Results

After each test is run a message appears advising you of the results of the test. In most cases, if trouble is found, the message indicates the failure or failed component. Specific test menu descriptions begin below.

Flow Stability and Hardware Series Test Routines

Under most circumstances the Flow Stability and Hardware Series tests will provide a thorough evaluation of the condition of your pump. We recommended that these two tests be used first if the performance of the pump is in question. The Flow Stability test is described on page 110 and the Hardware Series Tests is described on page 121.

SOFTWARE VERSION (PASSIVE)

Selecting /Software Version/ displays the version of software contained in the pump. The particular version of software resident in your pump will vary depending upon the date of manufacture or upon the date of software upgrade.

THE DIAGNOSTICS MENU

The Diagnostics Menu (Figure 4.47) contains three items commonly used to evaluate the pump and an additional selection to allow the pressure transducer's output to be zeroed.

```
>Flow Stability
·Measured Parameters
------------------------------------------------------
·Check Valve Test
·Zero Pressure
```

Figure 4.47 The Diagnostics Menu

Flow Stability

Your pump is constantly monitoring its flow stability while pumping. An internal software program allows the pump to determine when flow stability has been adversely affected by leaking check valves, out-gassing solvents, or other abnormal conditions.

The Flow Stability test (a passive test) evaluates how even the solvent flow is through the pump. The pump must be pumping solvent for this test to be run. The test can be initiated when the Status Screen shows any one of these states: EQUIL, or READY (all pumps). In addition, *time*, QEQUIL, or QREADY can be displayed on the P4000 pump. Completion of the test however, is dependent on the pump being in a READY state. When the pump is not in a ready state the Flow Stability screen will read, "Not Ready". When flow stability is being evaluated, the screen will read, "Test in Progress. Please Wait."

The pump's cam must go through at least 10 pump cycles (cam revolutions) to accurately assess stability. The value displayed is affected by the compressibility of the solvent being pumped and the compliancy of the hardware (tubing, column, and so on).

Therefore, the results are reported in two ways: a) an overall judgment of the pump performance which appears in the upper left-hand corner of the display, and b) a number indicating where, within the range, the result lies. This number is shown in the upper right-hand corner of the display. Three flow stability readings are possible:

Stable	(0- 25)
Acceptable	(25 - 90)
Unstable	(> 90)

Unless a very volatile or compressible solvent is being pumped, for example hexane, a number near the higher end of the range (60-90) probably indicates that the system is not ideal, and the results of further troubleshooting might improve the flow stability.

NOTE: The Flow Stability Test may show unstable flow during column equilibration.

If the results of the flow stability test are abnormal for your LC application, follow these steps to locate the problem:

1. Test the integrity of the inlet and transducer check valves by running a Check Valve test. (See page 112.)
2. Verify that the mobile phase solvents are adequately degassed.
3. Refer to the recommendations of *General LC System Troubleshooting Techniques* in Appendix A, page 159.

The flow stability assessment will remain displayed until you stop the test by pressing any one of the following keys: [ENTER], [∧], [MENU], or [STATUS].

Measured Parameters

Measured Parameters (a passive test) shows the measured flow rate (Figure 4.48). The number in parentheses indicates the flow rate setting in the run file. The calculated flow is based on the flow calculated during the last 360° of motor rotation.

```
Calc Flow          (2.00)          1.95
```

Figure 4.48 An example of the Measured Parameters Menu

The measured parameters will remain on the display until the test is stopped by pressing any one of the following keys: [ENTER], [∧], [MENU], or [STATUS].

Check Valve Test

The Check Valve test (an active test) can help you further diagnose the source of flow stability problems. If the results indicate a defective check valve, the test should be repeated to ensure the results were not due to an isolated transient condition, such as a single air bubble.

This test affects flow accuracy while the test is running. Do not run the test during an analysis. The pump can be in EQUIL, or READY and the flow rate must be 2.5 mL/min or less (for standard liquid ends). If the test is activated and the current flow rate is greater than 2.5 mL/min, a message will be displayed, prompting you to reset the flow rate. In general, the flow rate must be set to one-quarter or less of the maximum flow rate of the liquid ends.

HINT: Defective check valves tend to perform better at higher column pressures. Lowering the column pressure by decreasing the flow rate increases the test's sensitivity of marginally operating check valves.

NOTE: The inlet check valve is located at the base of the inlet liquid end. The transducer check valve is located at the base of the pressure transducer.

To activate the test select /Check Valve Test/. Follow the displayed instructions. A message will inform you of the 8 - 10 pump cycle delay before test completion. During this time, the condition of the inlet and transducer check valves of the pump are monitored. When the monitoring period is complete, pumping returns to normal constant flow control and a message is displayed, showing the results. Messages are listed in Table 4.1.

NOTE: Changing mobile phase concentration may cause the test to report a good check valve as defective. Stabilize composition before running the test.

Pressing [RUN] after the results are displayed will rerun the test. Stop the test by pressing [ENTER] or [∧].

Table 4.4 Check Valve Test Results

`Both check valves good`	Both check valves are performing well.
`Transducer check valve is defective`	The transducer check valve should be replaced. See Chapter 5, *Required Maintenance* for instructions.
`Inlet check valve may be defective`	The inlet check valve may be defective. An air bubble lodged in the check valve or piston seal or a slight leak in an inlet fitting may cause this message to be displayed. Verify that solvents are adequately degassed and that fittings are tight. Purge the pump and rerun the test to verify the message. If this same message is displayed, replace the inlet check valve. See Chapter 5, *Required Maintenance* for instructions.
`Bubbles or leaks likely. Check degas`	The check valves are not the cause of flow problems. Verify that solvents are adequately degassed and that fittings are tight. Observe the inlet tubing while purging the pump. If air bubbles are seen, increase the helium flow rate (if helium degassing), or tighten the leaking fitting. Tighten bottle caps. Ensure solvent supply is vented.

NOTE: Pulse dampeners should not be used with SpectraSYSTEM pumps. The flow is dynamically controlled and will be adversely affected by compliant loads.

`Test aborted, Pump not referenced in 10 Cycles`	The pump is not able to establish a reference column pressure within 10 pump cycles. The pump has serious flow problems. Verify that the solvents used are miscible in all concentrations encountered. If possible for your column, increase the column pressure by raising the flow rate. The check valves require more than 100 psi column pressure to operate properly. Defective check valves will usually operate well enough at higher pressures to allow the test to run.
`Test aborted By Operator`	The test was stopped before the pump could count 8 cycles.

Zero Pressure

The pump allows the pressure transducer's output to be zeroed automatically without the need for adjustment of potentiometers. The pump should be stopped before selecting /Zero Pressure/. The display will show:

```
Release system pressure,
then press ENTER key.
```

Figure 4.49 Release system pressure prior to zeroing the pressure transducer's output

Release the system pressure by opening the bypass valve or removing the column from the system to ensure that the transducer is actually sensing zero system pressure. Otherwise, a message showing "Unable to Zero" will appear. Follow the instructions to complete zeroing the pressure. To abort the test, press [ENTER], or [∧], or [STOP]. This will return the zero setting to its previous value.

THE MAINTENANCE LOG

The Maintenance Log is fully described in Chapter 5, *Required Maintenance*.

THE CALIBRATION MENU

The Calibration Menu (Figure 4.50) contains a Flow Calibration "test". The flow Calibration can be run in one of three modes.

```
·Flow Calibration
```

Figure 4.50 The Calibration Menu

Flow Calibration

The Flow Calibration Menu performs some internal calculations based on user-measured values. This is a passive test but it uses the [RUN] key. Unless run incorrectly, this test will not interfere with pump operation. Select /Flow Calibration/ to access the flow calibration menus.

The test is run in one of three modes. Select either Meter, Fixed Volume or Fixed Time from the Flow Calibration Menu (Figure 4.51). Depending on your selection, the display will allow you to enter values that enable the test to be completed. These are described in Figure 4.51.

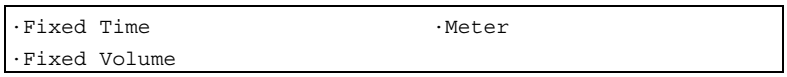


Figure 4.51 Flow Calibration Menu

Table 4.5 Flow Calibration Modes

Calibration Mode	Value to Enter
Fixed Time	Measured Volume (mL)
Fixed Volume	Measured Time (min)
Meter	Measured Flow

Fixed Time: Measured Vol (mL)
This calibration mode assumes that you have collected and measured a specific volume pumped during a fixed time period. Selecting Fixed Time displays the Fixed Time Menu (Figure 4.52).

Figure 4.52 The Fixed Time Menu
(NOTE: The Measured Vol. value was artificially input to demonstrate the menu function.)

1. Enter the theoretical volume for the fixed time period in the Calculated Vol. field.
2. Enter the measured volume for the fixed time period in the Measured Vol. field.
3. Press [RUN] to initiate the test. During the test, the pump determines a new flow correction factor to compensate for the inaccuracy in the flow rate. The Flow Correction Menu displays the results of the test. Figure 4.53 shows example results.

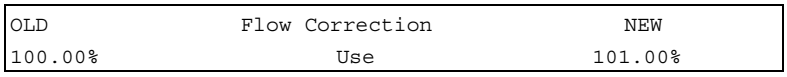


Figure 4.53 The Flow Correction Menu with example fixed time calibration results

OLD: The previous flow rate correction factor default or from a previous flow calibration test.

NEW: The new flow rate correction factor based on the Measured Vol. value.

Flow Correction: Use the [+]/[-] keys and press [ENTER] to select one of three choices:

Use: Use NEW flow correction factor. The factor remains in effect until you turn off the pump or initiated the NOVRAM.

Save: Save NEW flow correction factor to NOVRAM. This factor remains in effect until you save a different value over it, or until you reinitialize the NOVRAM (TESTS/SERVICE/Test 271). Test 271 replaces this value with the default flow correction factor.

Scrap: Discard the NEW (previous) flow calibration factor and keep the current value.

Press [RUN] to complete the test.

Fixed Volume: Measured Time (min)
This calibration mode assumes that you have externally timed the period in which a specific volume has been pumped.

Selecting Fixed Volume displays the Fixed Volume Menu (Figure 4.54).

·Calculated Time.	·10.00 min
·Measured Time	9.50 min

Figure 4.54 The Fixed Volume Menu

(NOTE: The Measured Time value was artificially input to demonstrate the menu function.)

1. Enter the theoretical time period for the fixed volume in the Calculated time field.
2. Enter the measured time for the fixed volume in the Measured Time field.
3. Press [RUN] to initiate the test. During the test the pump determines a new flow correction factor to compensate for the flow rate inaccuracy.

OLD	Flow Correction	NEW
100.00%	Use	101.00%

Figure 4.55 The Flow Correction Menu with example fixed volume calibration results

OLD: The previous flow rate correction factor default or from a previous flow calibration test.

NEW: The new flow rate correction factor based on the Measured Vol. value.

Flow Correction: Use the [+]/[-] keys and press [ENTER] to select Use, Save, or Scrap as described above.

4. Use the [+]/[-] keys to choose Use, Save, or Scrap.
5. Press [RUN] to complete the test.

Meter: Measured Flow (mL/min)
This calibration mode assumes that you have externally measured the precise flow rate that the pump is operating at while set at a specific flow rate.

Selecting /Meter/ displays the Meter Menu (Figure 4.56).

```
·Selected Flow.                    ·1.00 Mn
·Measured Flow                      1.00 Mn
```

Figure 4.56 The Meter Menu

(NOTE: The Measured Flow value was artificially input to demonstrate the menu function.)

1. Enter the set flow in the Selected Flow field.
2. Enter the measured flow in the Measured Flow field.
3. Press [RUN] to initiate the test. During the test the pump determines a new flow correction factor to compensate for the inaccuracy in the flow. The Flow Correction Menu displays the results of the test. Figure 4.57 shows example results.

```
OLD              Flow Correction              NEW
100.00%                Use                  101.00%
```

Figure 4.57 The Flow Correction Menu with example fixed volume calibration results

4. Use the [+]/[-] keys to choose Use, Save or Scrap as described for the Fixed Time Menu.
5. Press [RUN] to complete the test.

To exit the flow calibration menus without entering any values, press [∧] until you return to the Calibration Menu.

NOTE: Do not press [STOP] unless you have already entered a new value in one of the flow calibration menus. If the pump is in RUN, doing so will interfere with your analysis. Use ***[∧]*** *to exit the flow calibration menus instead.*

THE SERVICE MENU

The Service Menu (Figure 4.58) contains several service-related tests, including the Hardware Series test.

```
>Current History
·Lifetime History
-------------------------------------------------------
·ROM Test                          (200)
·RAM Test                          (201)
·Cycle Step Count                  (205)
·External Inputs                   (206)
·Display Test                      (208)
·Transducer Range                  (209)
·Motor Step/Valve                  (211)
·Hardware Series                   (220)
·Initialize NOVRAM                 (271)
```

Figure 4.58 The Service Menu

The numbers in parentheses refer to a similar test found in earlier SpectraSYSTEM pumps. They are included for the convenience of users and service personnel familiar with this previously-used numbering scheme.

Current History

By selecting /Current History/ you access a chronological list of operating state changes. The negative number on the far left indicates the time (in minutes) between the time the Current History Menu was accessed and the state change occurred. More specific information about reading the Current History Menu is found in the *SpectraSYSTEM Pumps Field Repair Manual*.

To exit the Current History, press [ENTER].

Lifetime History

By selecting /Lifetime History/ you access a log of five measured items relating to the entire time the pump has been in operation. An example Lifetime History Menu is shown in Figure 4.59.

The top line shows 1) the total time that the pump's motor has been running in hours (Hr) and 2) the total number of strokes in thousands (kSt) taken by the cam; the bottom line shows 3) the number of times the pump has been powered-on (on), 4) the number of times the pump has been powered-down (off), and 5) the number of times an error occurred when NOVRAM was written to upon power-down (bad).

Press any one of the following keys to exit the Lifetime History screen: [ENTER], [∧], [MENU], or [STATUS].

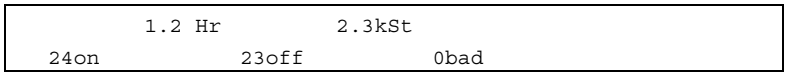


Figure 4.59 The Lifetime History Menu

ROM Test (Passive)

The ROM test (200) verifies the integrity of the ROM (Read Only Memory) in your pump. The ROM is where all of the built-in programs for the pump operation are stored. If faults are found in any part of ROM, a message indicating that the test has failed will be displayed. Press [STOP] to stop this test. Do not to press [STOP] more than once or else pump operation will be interfered with.

If a failure is indicated, contact Thermo Fisher Scientific.

RAM Test (Active)

The RAM test (201) verifies the integrity of the RAM (Random Access Memory) in your pump. The RAM is where your pump files are stored and where temporary calculations are performed. The pump must be stopped (STOP) for this test to be implemented. If any faults are found with RAM, the message shown in Figure 4.60 is displayed.

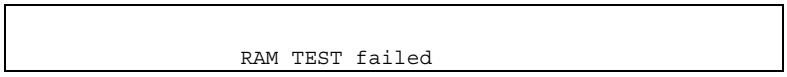


Figure 4.60 RAM Test failure message

Contact Thermo Fisher Scientific if this test indicates a failure.

Cycle Step Count (Passive)

Normally, 12,800 motor drive pulses are required for one revolution of the pump motor, as detected by the cam sensor. The Cycle Step Count test (205) displays a count of the number of pulses required for the last complete motor revolution.

The Cycle Step Count test is a dynamic measurement of the number of steps counted, the lag amount (the number of steps the count has shifted since the last revolution), and the number of seconds required for the last revolution.

Generally, the number of steps should be 12,800 ± 64. The lag value varies due to the load on the pump. This number should be steady, or fluctuate no more than ±120 steps.

Start the test by selecting /Cycle Step Count/. To stop the test, press any one of the following keys: [ENTER], [∧], [MENU], or [STATUS].

NOTE: The Cycle Step Count test requires that a full cam revolution has occurred.

External Inputs (Passive)

The External Inputs test (206) allows you to conveniently monitor the status of two of the external input lines, STOP and RUN. The STOP line causes the pump to stop pumping when momentarily grounded. A momentary ground at the RUN input line causes the run time clock to begin. Use this test if you are having difficulty interfacing your pump to a controlling device, such as a SpectraSYSTEM autosampler.

To run the test, select /External Inputs/. The display continuously shows the current state of the STOP and RUN inputs (updates every 0.5 second). "Lo" means the input is grounded (active) and "Hi" means the input is "high" (inactive).

To stop the test, press either [ENTER], [∧], [MENU], or [STATUS].

Display Test (Passive)

The Display test (208) exercises the pump's display. When initiated, the display shows staggered alphanumeric characters that scroll from left to right. Pressing [STOP] freezes the display; pressing [RUN] resumes movement.

This test is also a keyboard test. Pressing the cursor keys will cause the alphanumeric display to scroll in that direction.

Other keys can be tested by first pressing [ENTER] to access the key test. The display will verify other keys such as [STATUS] or [MENU], as soon as each is pressed.

To return to the scrolling alphanumeric characters, press [RUN], [RUN]. To stop the test, and return to the Service Test Menu, press [STOP], [STOP].

Contact your representative if the display appears unusual.

Transducer Range (Active)

Transducer Range is not truly a test. It contains a field where you must enter the calibration value (in mV) for a replaced pressure transducer. The range is located on the replacement transducer wire. Your pump features advanced circuit designs which allow the pressure transducer range adjustment to be set by entering a value from the keyboard. No adjustment of potentiometers is necessary. Your pump comes from the factory preset to the proper range. The value is stored in a NOVRAM. Do not change the transducer calibration setting unless the pressure transducer or System PCB are replaced. The calibration number is recorded on a tag attached to the transducer cable. The System PCB and pressure transducer are not user-serviceable parts. A qualified service representative must perform any repair or replacement.

Motor Step/Valve (Active)

The Motor Step/Valve test (211) exercises the pump motor and the switching valve on the pump. When activated, the pump motor is continuously stepped and each switching valve is sequentially opened and closed at a rate of 1 valve per 0.512 seconds. This test is useful for detecting an intermittently failing switching valve or pump motor.

Select /Motor Step Valve/ to initiate the test. Follow the instructions. Each open valve is shown dynamically on the display. The test will continue until one of the following keys is pressed: [ENTER], [∧], [STOP], [MENU], or [STATUS].

Hardware Series (Active)

The Hardware Series test (220) is an extensive evaluation of the System Printed Circuit Board (PCB), switching valve, pump motor, and pressure transducer. The System PCB contains all of the circuitry for the operation of the pump, except for the display functions. Once activated, the test exercises and diagnoses the condition of various circuits. The pump must be idle (not pumping) before activating the test. This test will not affect pump files.

NOTE: The external events connector (if present) must be removed from the rear of the pump before initiating the Hardware Series test. Otherwise, "Board Failure: Code 8" may occur.

To activate the test, select /Hardware Series/. Follow the instructions given on the display. Typically, the display will show:

```
Release system pressure
Then press ENTER.
```

Figure 4.61 Initial Hardware Series test message

Open the column bypass valve or otherwise remove column pressure from the transducer since the pump will operate during the test and an excessively high column pressure might be generated if not bypassed. Press [ENTER] to continue the test.

Once the test is activated the display will appear as in Figure 4.62.

```
          Test in Progress
```

Figure 4.62 Hardware Series test message, after pressing [ENTER]

The pump's components are tested in the following order.

1. Pressure transducer and circuitry
2. Input/output ports
3. Solvent switching valve and circuitry (P4000 only)
4. Motor drive circuitry
5. Cam marker and circuitry
6. Motor revolution and sine/cosine circuitry

As each portion of the test is completed a message is displayed. If all components and circuitry are within specifications, the messages shown in Figure 4.63 are displayed during the test.

If a failure is detected during the test, the failure message is displayed. It remains displayed until [ENTER] is pressed (the test resumes).

To stop the test, press [STOP], or press [∧] to return to the Service Menu.

```
TRANSDUCER TEST PASSED
-------------------------------------------------------
PORT TEST PASSED
-------------------------------------------------------
VALVE TEST PASSED ** (P4000 only)
-------------------------------------------------------
MOTOR DRIVE TEST PASSED
-------------------------------------------------------
CAM MARKER TEST PASSED
-------------------------------------------------------
REVOLUTION TEST PASSED
-------------------------------------------------------
Hardware series tests completed - PASSED
```

Figure 4.63 No problems found during the Hardware Series test.

If problems are found during the test, a message suggesting the most likely failure is displayed, although in some cases other failures are possible. The messages shown in Table 4.6 indicate a possible System PCB failure.

Table 4.6 Errors Indicating A Possible System PCBA Failure

BOARD FAILURE: CODE XXX	If the failure is identified as a component on the system printed circuit board or if the test cannot determine the failed component, a message is reported where XXX is a 1-, 2- or 3- digit number.
8	Remove the external events connector from the rear of the pump. This test exercises the input lines and may be affected by attached cabling.
1-100	Pertains to failures of the system printed circuit board. Contact your service representative.
101, 102, 103	Indicates the proportioning/switching valve's electrical system (P4000) has failed. Refer to Chapter 5, under *Solvent Proportioning/Switching Valve Replacement*.
104, 105	Possible broken or loose pump motor cable wire.
124	Too many motor steps were needed to complete a cam revolution. You may have a loose motor coupler or faulty System PCB. Contact your local sales/service representative for service information.
125	Too few motor steps were needed to complete a cam revolution. You may have a faulty cam sensor or System PCB. Contact your local sales/service representative for service information.
TRANSDUCER UNPLUGGED	The pressure transducer was not detected. The connector going to the pressure transducer should be checked. Access to this connector requires the removal of the pump's outer protective cover. Because there are safety issues involved in its removal, this should only be performed by a qualified service technician. The location of this connector as well as the proper procedure for removing the outer cover are outlined in the Field Repair Manual.
CANNOT ZERO TRANSDUCER	The transducer circuitry is not able to compensate for the zero offset of the transducer. Make sure that the system is at zero column pressure (column bypassed) before starting the test. If so, replace the transducer. Contact your local sales/service representative for assistance.
CHECK FUSE F1	(P4000) Fuse F1 is used to protect the valve drive circuitry from internally shorted valves which may short internal circuitry on the system PCB. Checking the status of F1 requires the removal of the outer cover of the pump. Because of safety issues involved in the removal of this cover a trained service technician should perform this evaluation. Contact your local sales/service representative for assistance.
CAM MARKER NOT FOUND	This message indicates that the sensor that detects cam revolutions is not operational. Either the motor coupling is loose or the cam sensor is defective. This requires tightening of the motor-to cam coupler if loose, or replacement of the cam sensor. Both of these actions require the removal of the top cover. Because of safety issues involved in the removal of this cover a trained service technician should perform this evaluation.

Table 4.7 Errors Indicating A Possible System PCBA Failure, *continued*

VALVE CABLE UNPLUGGED	(P4000) The test has detected that the solvent proportioning/switching valve cable is disconnected from the System PCB. There are two locations where the valve is connected. The first location is underneath the proportioning valve assembly. To access this location the proportioning valve must first be removed. This is achieved by unscrewing the finger-tight captive fasteners which hold the assembly in place. Once this is accomplished the cable connector is visible. Press together the connector attached to the cable coming from the proportioning valve assembly to the mating connector located nearby in the chassis of the pump. If this fails to remedy the problem then the second cable connection location should be checked. Correction of the problem at this second location requires the removal of the top cover. Because of safety issues involved in the removal of this cover a trained service technician should perform corrective action.
CAM SENSOR FAILURE	The cam sensor cable is disconnected or defective and needs to be replaced. Correction of this problem requires the removal of the top cover. Because of safety issues involved in the removal of this cover a trained service technician should perform corrective action. Contact your local sales/service representative for service information.
NO CURRENT TO MOTOR	The test has detected no current flow through the pump motor. Either the motor cable is unplugged on the System PCB or the entire drive circuitry is defective. Correction of this problem requires the removal of the top cover. Because of safety issues involved in the removal of this cover a trained service technician should perform corrective action. Contact your local sales/service representative for information on obtaining a replacement.

Initialize NOVRAM (Active)

The pump must be stopped for /Initialize NOVRAM/ to be completed.

NOTE: Initialize NOVRAM (271) has a profound effect upon the pump's non-volatile RAM. Do not initialize the NOVRAM unless you fully understand all consequences associated with this action.

By initializing the NOVRAM all files are reset to their default values, with the exception of the run file. All user-preferences set in /OPTIONS/ are also returned to their default values. In addition, any changes that had been made to the Liquid End Type and Flow Calibration parameters are erased, and these selections are returned to their default values. In general, any field value or selection that is normally retained when the pump is turned off and then on will be reset to its default value when the NOVRAM is initialized.

NOTE: The pressure transducer's range value is <u>not</u> erased when the NOVRAM is initialized.

HINT: To retain a single file while initializing the NOVRAM: load it (so that it becomes the run file), stop the pump, initialize NOVRAM, make a change to a value within the run file from Status, then select /Save File/ at the bottom of the Status Menu.

5

Required Maintenance

Introduction

When properly maintained, your Thermo Scientific SpectraSYSTEM pump will provide years of trouble-free operation. It is important that your pump receive routine preventive maintenance to ensure reliability and optimum performance. Properly performed routine, preventive maintenance also helps keep your warranty valid. Your pump is designed to encourage proper maintenance by making maintenance parts easy to access, replace and record.

This chapter describes the Maintenance Log Menu and how to use it. Some hints to help you extend the maintenance period of your pump are also included. Easy-to-follow, step-by-step required maintenance procedures are also contained in this chapter so that you can keep your pump in optimum working condition. A few maintenance tips for parts of the pump not directly involved with solvent flow follow the maintenance procedures. The last section contains replacement procedures for two user-serviceable parts: fuses and the solvent switching valve.

NOTE: Maintenance of the pump is the responsibility of the user. Routine maintenance is not provided under warranty. However, planned maintenance contracts are generally available. Please contact your local representative if you are interested in purchasing a planned maintenance contract.

THE BENEFITS OF PROPER MAINTENANCE

As with most things, there is tremendous benefit in doing things right the first time. For example, an unusually fast seal failure may indicate either incorrect installation or a scratched piston. A scratched piston may be caused by improper installation of the seal or piston, by allowing the pump to sit idle with a buffered eluant in it, or by failing to filter your eluants.

Maintenance Schedule

Table 5.1 Gradient Pump Preventative Maintenance Schedule

Frequency	Procedure	Performed By
Daily	Check waste reservoir. Empty as required.	User
	Check solvent reservoir. Replenish as required.	User
Annually	Replace piston seals.	User
	Replace backflush seal.	User
	Check pistons and Kel-F seals. Sonicate any parts as required.	

Maintenance Log

The Maintenance Log provides a convenient way for you to record maintenance performed on the liquid ends and set intervals for periodic maintenance. When a maintenance interval has been exceeded the pump will automatically display a message indicating that maintenance may need to be performed.

MAINTENANCE LOG MENU

The Maintenance Log is accessed by selecting /TESTS/ from the Main Menu, then selecting /Maintenance Log/ (Figure 5.1 and Figure 5.2).

```
·FILES                    ·QUEUE                    >TESTS
          ·COMMANDS                 ·OPTIONS
```

Figure 5.1 Main Menu with TESTS selected

```
·Software Version
·Diagnostics
------------------------------------------------------
>Maintenance Log
·Calibration
·Service
```

Figure 5.2 Tests Menu with Maintenance Log selected

The Maintenance Log Menu (Figure 5.3) consists of a table, used to record dates and volumes, followed by one field used to enter a value

relating to flow, and two additional menu items. All of these are described in this section.

```
ITEM          DATE        DUE           VOL
Seal1      8 AUG95        200           201
-------------------------------------------------------
Seal2      8 AUG95        200           201
Piston1    18AUG95        600           400
Piston2    18AUG95        600           400
Inlet      8 AUG95        600           201
X-ducer    8 AUG95        600           201
·Maintenance Position
·Liquid End Type
```

Figure 5.3 Maintenance Log Menu

The Maintenance Table

The top half of the Maintenance Log Menu is a table (Figure 5.3).

ITEM, DATE, DUE, and VOL

The ITEM field remains fixed. "Seal 1" and "Seal 2" should be paired with "Piston 1" and "Piston 2", respectively, to identify the inlet and outlet liquid ends. "Inlet" refers to the inlet check valve, while "X-ducer" refers to the transducer check valve.

Enter the date (day/month/year) in the DATE field for the last time maintenance was performed on each item.

The pump keeps "liters pumped" counters, in the VOL (volume) field, for each major maintenance item (the pump seals, pistons, and check valves). You may set a DUE volume in liters for each item. When the DUE volume is exceeded by the volume of liters pumped (VOL), the reminder "MAINTENANCE DUE - SEE PUMP LOG" is displayed. This message will appear each time a file is initialized. You may choose to use this feature to set regular intervals for maintenance, such as seal changes, pump/column cleaning, or simply to serve as a reminder to verify that the system is operating properly. The interval remains set until either the date has been updated or the DUE value has been increased.

In the example shown in Figure 5.3, a fairly complete maintenance was done on 8 Aug 95, when both seals and check valves were replaced.

Setting Intervals

The volume of mobile phase that you can expect to pump before the pump requires maintenance is very dependent upon the eluant being pumped and your adherence to good chromatographic practices. To obtain the maximum lifetime and best performance from your pump,

read *Extending the Maintenance Period* on page 132. Pump pistons and check valves have been known to last for years. Even the seals themselves can last more than a year for some applications.

An initial guideline for setting up your Maintenance Log for the first time is to set both seal DUE counters to 200 liters, and the check valve and piston DUE counters to 600 liters. Setting a value of zero (0) for any DUE interval inactivates the Maintenance Log for that specific item. To inactivate the entire Log, a zero (0) must be entered for all DUE intervals. Your specific maintenance interval can be determined by observing pump performance over time.

When a Maintenance Message is Displayed

Whenever the interval has been exceeded, and the message "MAINTENANCE DUE - SEE PUMP LOG" is displayed, you should either verify that the pump needs maintenance or that the pump is operating properly. If a maintenance interval is exceeded and you find that the pump does not require maintenance, increase the DUE interval by another 50 liters from the previous setting. Once you have established an expected interval for your system, use that interval for routine preventive care.

If you find that the interval before component failure is either unacceptable or variable, then the source of the problem must be identified. Read this chapter and Appendix A. Poor chromatographic practices are by far the most common source of problems. Specific procedures for inspecting and changing parts begin on page 133.

Maintenance Position

Selecting /Maintenance Position/ prepares the pump for liquid end removal or replacement. The display shown in Figure 5.4 appears:

```
To install or remove
liquid ends press ENTER
```

Figure 5.4 Maintenance position message

The maintenance position puts the pump's cam into a position to facilitate liquid end removal.

Liquid End Type

The Liquid End Type Menu allows you to select the proper liquid end for a specific application. Do not change the flow range unless you are installing liquid ends with capacities different from those purchased with the pump.

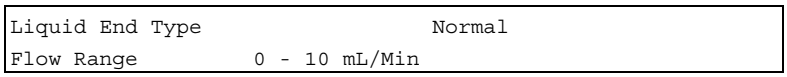


Figure 5.5 Liquid End Type Menu

Selecting Normal, Bio, or SemiPrp

If you select Normal, Bio, or SemiPrp (semi prep) in the Liquid End Type field, the Flow Range field changes automatically to corresponds to the preset ranges for these liquid ends. (Normal = 0 - 10 mL/min, SemiPrp = 0 - 30 mL/min, Bio = 0 - 10 mL/min, and Other.

Selecting Other

If you select Other in the Liquid End Type field, the Flow Range field becomes active, allowing you to enter your own flow range.

After changing the liquid end type on the display, press [ENTER]. A message (Figure 5.6) will prompt you to write down the old values in the Maintenance Log which correspond to the liquid ends you presumably just removed. It is important to keep records for each set of liquid ends you use. If you reinstall the "old" liquid ends, you will need to reenter the dates and statistics for the "old" ends into the Maintenance Log table. You are also reminded to enter new values into the log's VOL field, which correspond to the newly installed liquid ends. Normally the VOL field is not edited, but when new liquid ends are installed, ensure that the VOL fields for the appropriate maintenance items are reset to 0.

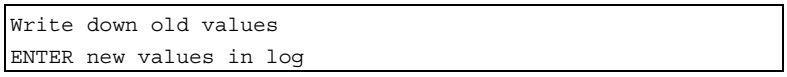


Figure 5.6 Reminder to keep proper records when liquid ends are changed

Flow Correction

An additional menu appears whenever you press an arrow key, [ENTER], or [+]/ [-] from the display shown in Figure 5.7. This menu allows you to set a Flow Correction, if desired. OLD and NEW values are displayed. For no Flow Correction, enter 100.00%.

NOTE: The Flow Correction menu is the same as the menu displayed when a flow calibration (/TESTS/, /Calibration/, /Flow Calibration/) has been initiated, except that it does not include the Use, Save, or Scrap option.

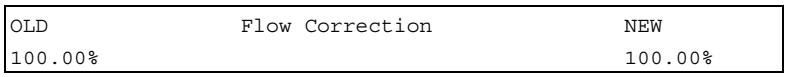


Figure 5.7 Flow Correction Menu

Flow correction is a value, in percent, which adjusts the actual volume that the pump delivers. As you use the pump, you may feel that although the pump is set at a specific flow rate, for example 2 mL/min, the pump actually delivers slightly more or less than this volume per minute. This can be due to a variety of maintenance- or LC-related reasons (seals, valves, and so on).

If desired, manually enter a flow correction value. This value can be entered automatically, based on the result of the flow calibration test, initiated from /TESTS/, /Calibration/, /Flow Calibration/. This test [which requires you to enter an accurately measured operation value (time, volume or flow rate)] is fully explained in Chapter 4.

If no correction to the flow is desired, enter a value of 100% in this field. Values from 90% to 110% are valid. For example, if you pump for one minute at 1 mL/min and collect 0.95 mL, then the pump is actually delivering 5% less solvent than expected. To compensate, enter 105.26% in the Flow Correction field. The pump's Status Screen will still display a flow rate of 1.0 mL/min, but the pump will actually deliver 105.26% of what it normally delivers at 1.0 mL/min.

Press [ENTER] to save the value, or simply leave the menu by pressing an arrow key.

Extending the Maintenance Period

As mentioned earlier, the volume of mobile phase you can expect to pump before maintenance is due is very much dependent on the way that the pump is being used. Following these guidelines helps you extend the life and improve the performance of your pump.

- Use high quality, spectro-grade or HPLC-grade solvents. These solvents do not usually need to be filtered before use.
- Filter water and prepared solvents through at least a 0.45-micron filter before placing them in the solvent reservoirs to remove particulate matter and organic contamination.
- Avoid pH extremes.
- Verify that the solvents used are miscible in all proportions. This is very important for a buffered mobile phase. Precipitation of salts quickly damages maintenance parts.
- Never leave the pump filled with buffered solvent when not pumping. Either lower the flow to 0.1 mL/min. or thoroughly flush the pump. Flush with at least 25 mL of pure filtered water.
- The pump should be filled with methanol if it is to be left idle for more than two days. This avoids the possible growth of organisms in aqueous solvent systems.
- Never use hydrochloric acids solutions.

- Avoid metal ions that can cause corrosion due to electrochemical processes. Typical metal ions to avoid: manganese, chromium, nickel, copper, iron, molybdenum.

Maintenance Procedures

SAFETY PRECAUTIONS

Observe the following safety precautions whenever performing periodic maintenance.

Caution!
A caution alerts you to situations that could result in personal injury. It also tells you how to avoid them.

High Voltage!
This icon alerts you to the presence of high voltage and to the potential injury that could occur from electrical shock were you to come in contact with a specific instrument area or component. It also tells you how to avoid contact with the high-voltage areas in your instrument.

Hot Surface!
This icon alerts you to potential injury that could occur from coming in contact with a heated surface or area on or in an instrument. It also tells you how to avoid contact with the heated surfaces in your instrument.

This section includes procedures for:

- Complete liquid end maintenance (includes disassembly and assembly)
- Check valve replacement
- Passivating stainless steel parts

Also included are maintenance tips for pump parts that are not involved with pump flow.

TOOLS

The following tools are useful to have on-hand as you perform maintenance procedures.

- Tweezers
- Open-end wrenches (1/4-inch, 5/16-inch, 1/2-inch)
- Loupe or magnifying glass
- Allen wrench (Hex head) 9/64-inch

PREPARATION

Prepare the pump for maintenance before performing any maintenance procedure.

To prepare the pump for maintenance, flush the pump with 25 mL of methanol. If an incompatible solvent is resident in the pump, flush with appropriate intermediate solvents before flushing with methanol. For example, if chloroform is being used as the mobile phase solvent, an intermediate flush of 25 mL methylene chloride would be appropriate before flushing with methanol.

LIQUID END MAINTENANCE

Complete liquid end maintenance includes procedures for seal and piston maintenance:

- Removal
- Disassembly
  — inspection for contamination
  — cleaning
  — piston inspection
  — cleaning/replacing parts if necessary
- Assembly
- Installation

For thorough cleaning, piston replacement, or total liquid end reconditioning, the liquid ends must be removed.

Having a second set of reconditioned liquid ends on hand for quick replacement will save additional time and allow maintenance to be performed at your convenience. Contact your local Thermo Fisher Scientific representative if you are interested in obtaining spare components. Part numbers are included in Appendix C.

NOTE: Keep the liquid end components as clean as possible. Contamination decreases seal life significantly.

Preparation

1. Flush your pump with 25 mL prior to disassembling your liquid ends. If methanol is not compatible with the mobile phase in your pump, flush the system with 25 mL of an intermediate solvent before flushing with methanol.
2. Remove the front cover, exposing the liquid ends (Figure 5.8).

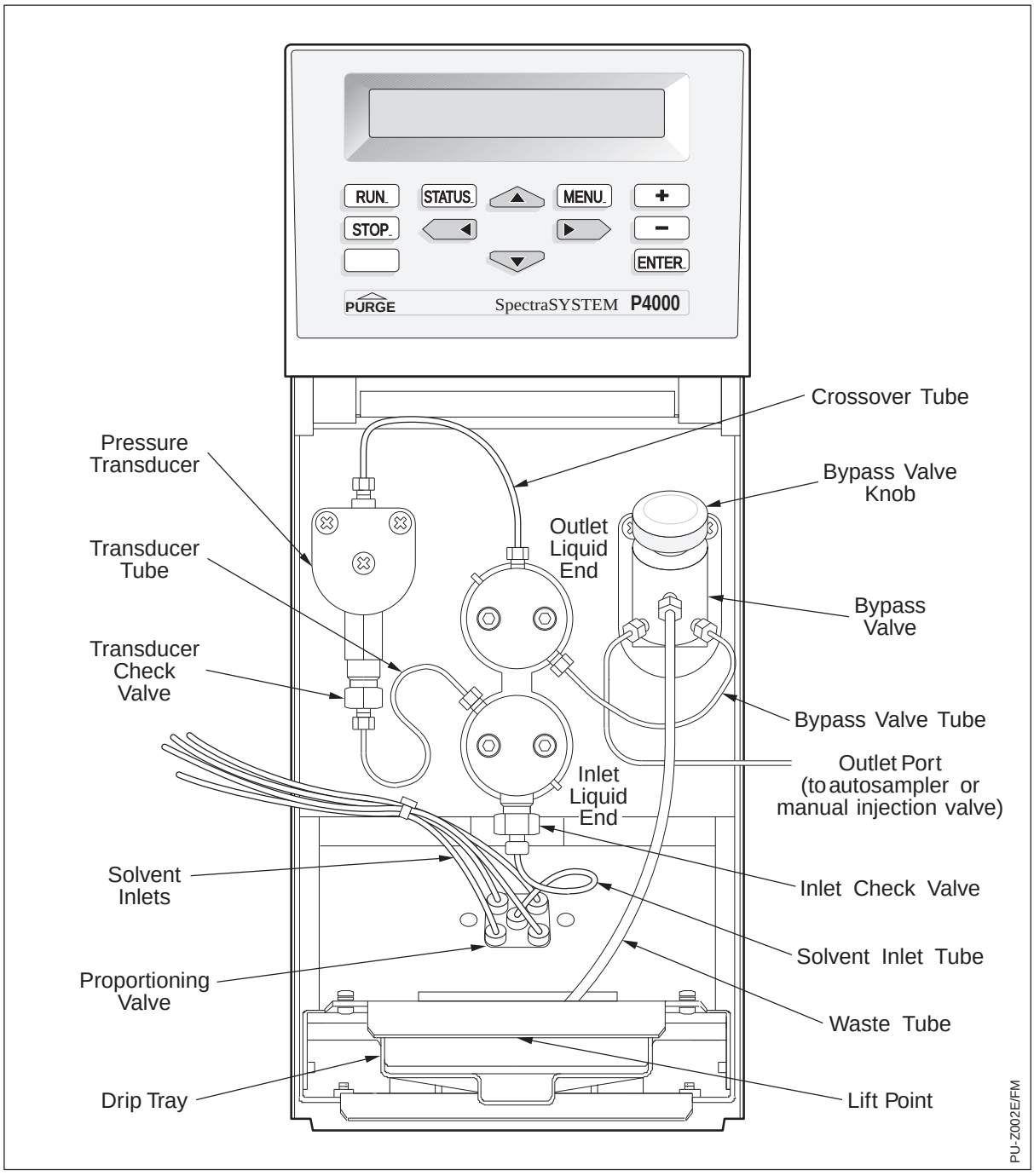


Figure 5.8 Pump with front cover removed

Liquid End Removal

To remove the liquid ends from the pump:

1. Remove all tubing attached to the pump heads. Turn the nuts counter-clockwise to remove (Figure 5.9).

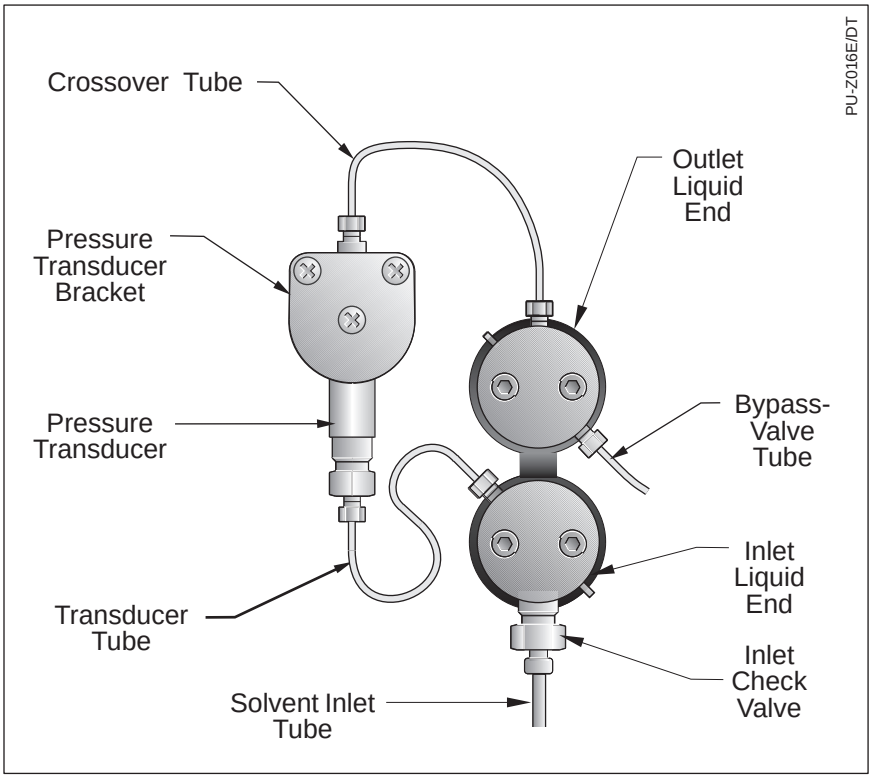


Figure 5.9 Liquid ends and tubing

2. Position the pump cam to enable the liquid ends to be removed. To do this, press [MENU], and select /TESTS/, /Maintenance Log/. Then move the cursor to /Maintenance Position/ and press [ENTER]. The display shown in Figure 5.10 appears.

```
To install or remove
liquid ends, press ENTER
```

Figure 5.10 Maintenance position message

Press [ENTER] to continue. The pump motor rotates for a few seconds and then is electrically locked into position. While the motor rotates the display appears as in Figure 5.11. When the cam is in the maintenance position, the display in Figure 5.12 appears.

```
Install or remove liquid
ends when motor stops
```

Figure 5.11 The display when the motor moves cam to maintenance position

```
X-ducer       (date)       (due)       (vol)
· Maintenance Position
```

Figure 5.12 The display after the motor moves the cam to the maintenance position

The pump is now in its maintenance position. It will hold this position (if power is maintained) until a file is initialized or a purge is started.

3. Remove the inlet check valve (Figure 5.13) from the inlet pump head.

NOTE: It is not necessary to remove the check valve to replace a piston, however, it is easier to remove at this time if total liquid end reconditioning is to be performed.

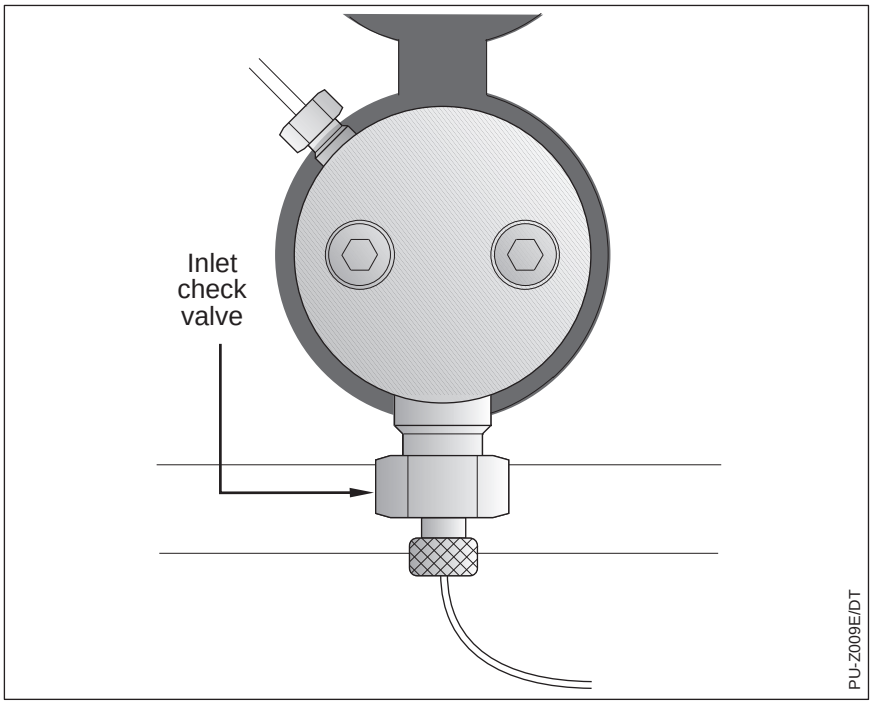


Figure 5.13 Inlet check valve

4. Push in the outlet (upper) liquid end and rotate it (90 degrees counter clockwise) until it releases from the pump module. Remove the liquid end and set it aside.
5. Push in the inlet (lower) liquid end and rotate it (90 degrees clockwise) until it releases from the pump module. Remove it and set it aside.

Liquid End Disassembly

To disassemble the liquid ends:

1. Separate the pump head from the piston holder housing by removing the two 9/64-inch hex cap screws. Turn the screws counter-clockwise.
2. Examine the Kel-F® seal in the cylinder bore. If the seal is damaged (scratched, warped or torn) it must be removed. Use tweezers to remove it by pulling gently on the seal's inner circumference. (Be careful not to scratch the cylinder surface!)
3. Examine the pump head for contamination. Flush the pump head with methanol or place it into an ultrasonic bath.
4. Remove the seal holder from the piston holder housing by grasping both ends of the exposed tube, and pulling gently (Figure 5.14).

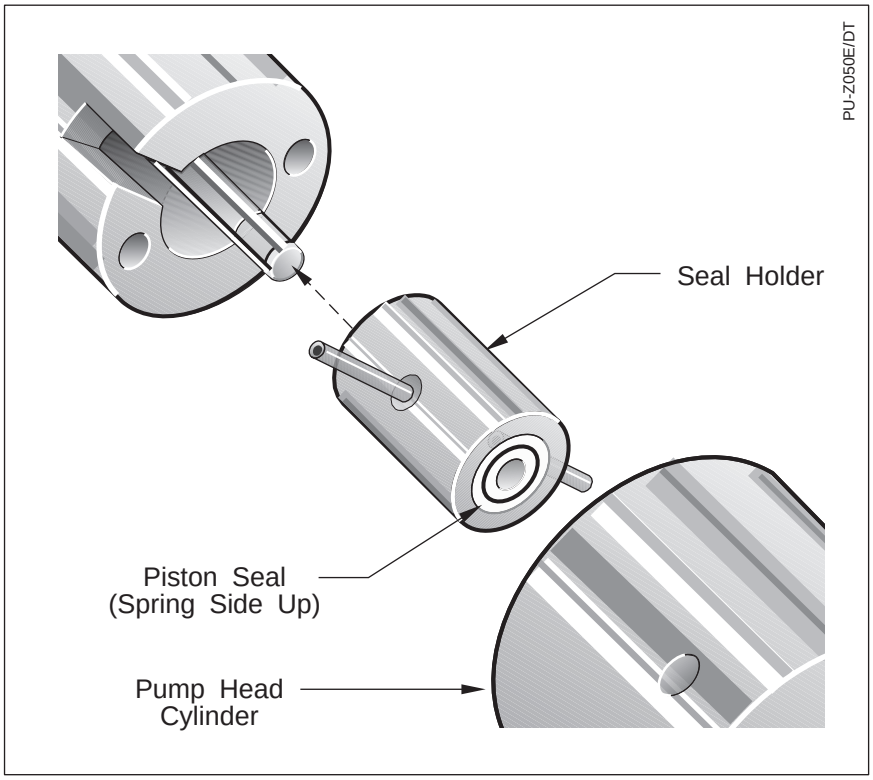


Figure 5.14 Removing the seal holder from the piston holder housing

5. Carefully remove the piston seals from the seal holders using the seal removal tool supplied in the accessory kit. Insert the tool and wiggle it in a circular manner to remove the seal. (See Figure 5.15). Flush the holders with methanol if contamination is present.

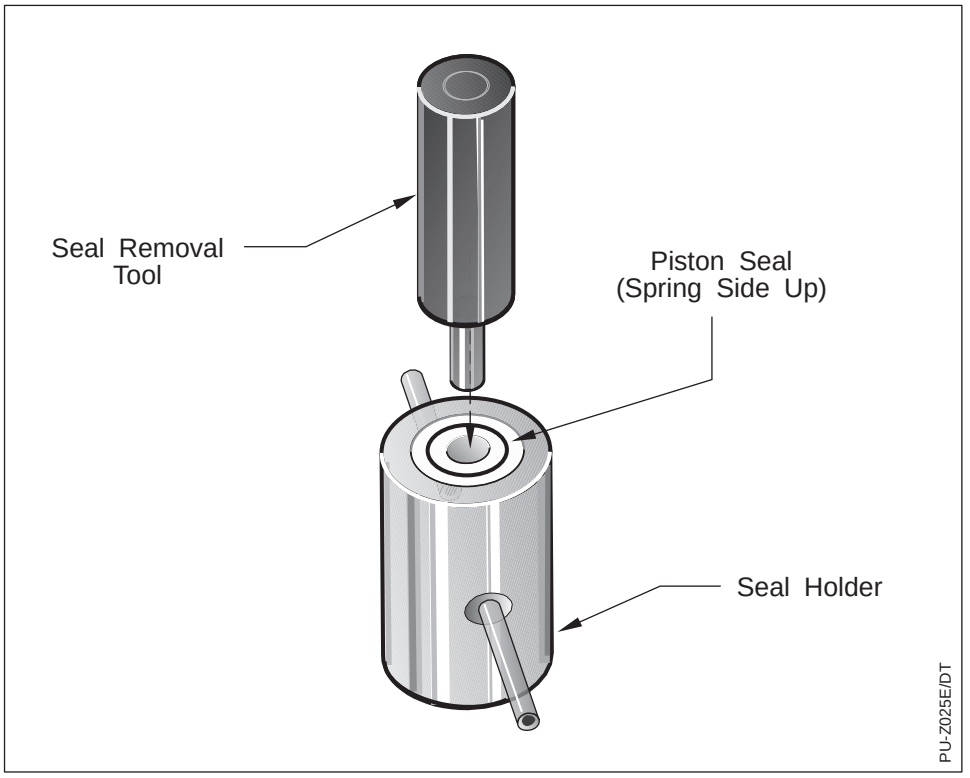


Figure 5.15 Using the seal removal tool

6. While retaining the piston holder, remove the 9/64-inch retaining cap screw (Figure 5.15). This allows the piston holder to be removed from the piston holder housing. Separate the holder, piston, spring and housing.

CAUTION! Wear safety glasses! The piston components are spring loaded and may shoot out! (Figure 5.16).

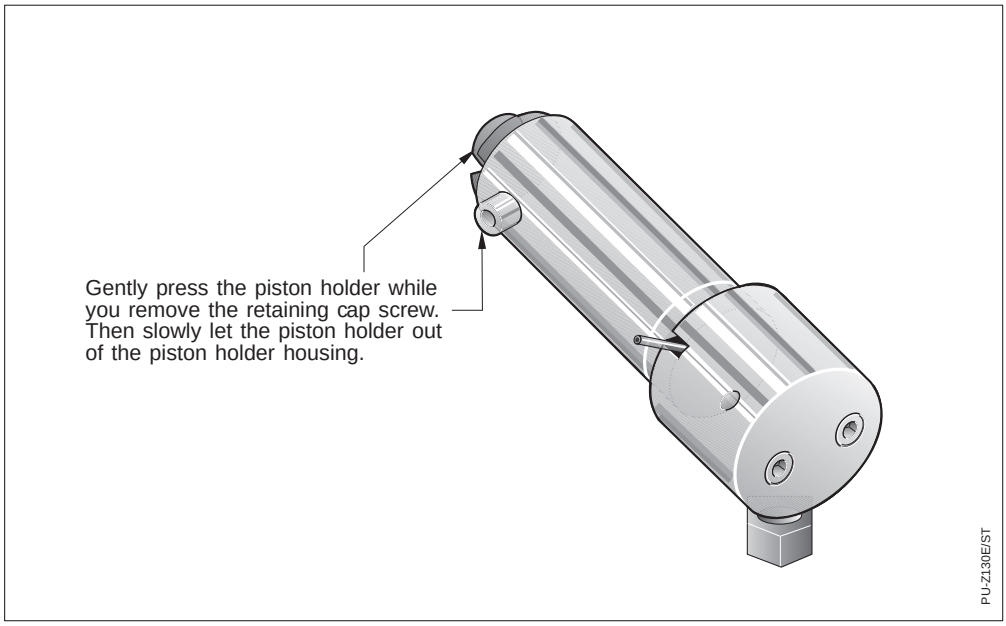


Figure 5.16 Retaining the piston holder

7. Examine all parts for wear, corrosion or contamination. Clean all deposits. Look for wear marks on the stainless steel shaft of the piston and corresponding wear marks on the inner aspects of the spring which indicate bending or bowing. If these wear marks are present, replace the spring (Figure 5.17).

NOTE: It is normal for the piston holder to produce a small amount of wear particles.

8. Examine the piston carefully under a low-power microscope or magnifying glass for fine scratches, ridges, or scoring which can reduce seal life (Figure 5.18). Some apparent scratches are actually deposits that can be cleaned. The piston can be cleaned by wiping it gently with a laboratory towlette or cotton swab that has been immersed in methanol. Replace the piston if scratched or pitted. The new piston should also be cleaned prior to installation.
9. Thoroughly flush all components with methanol.

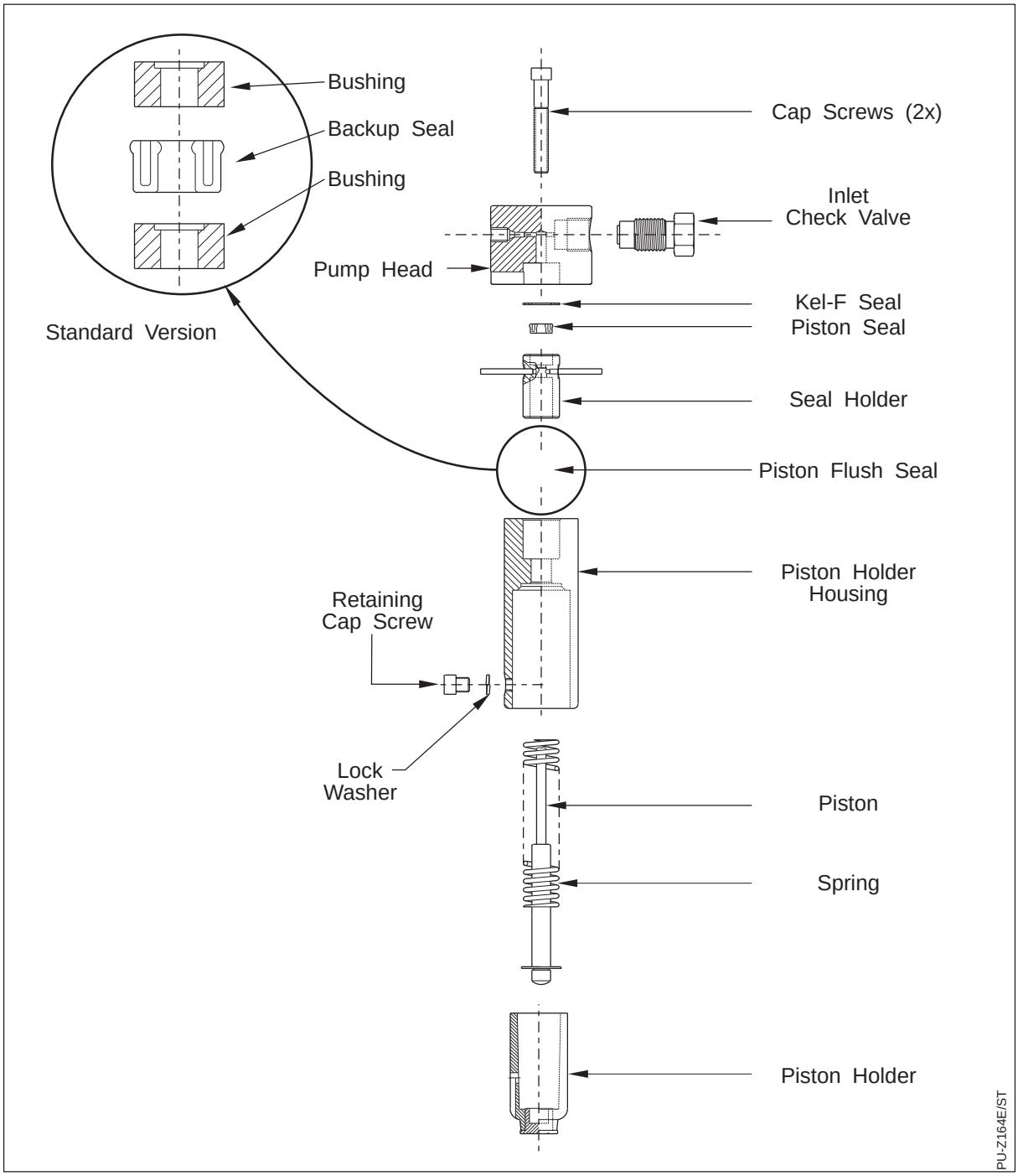


Figure 5.17 Liquid end components

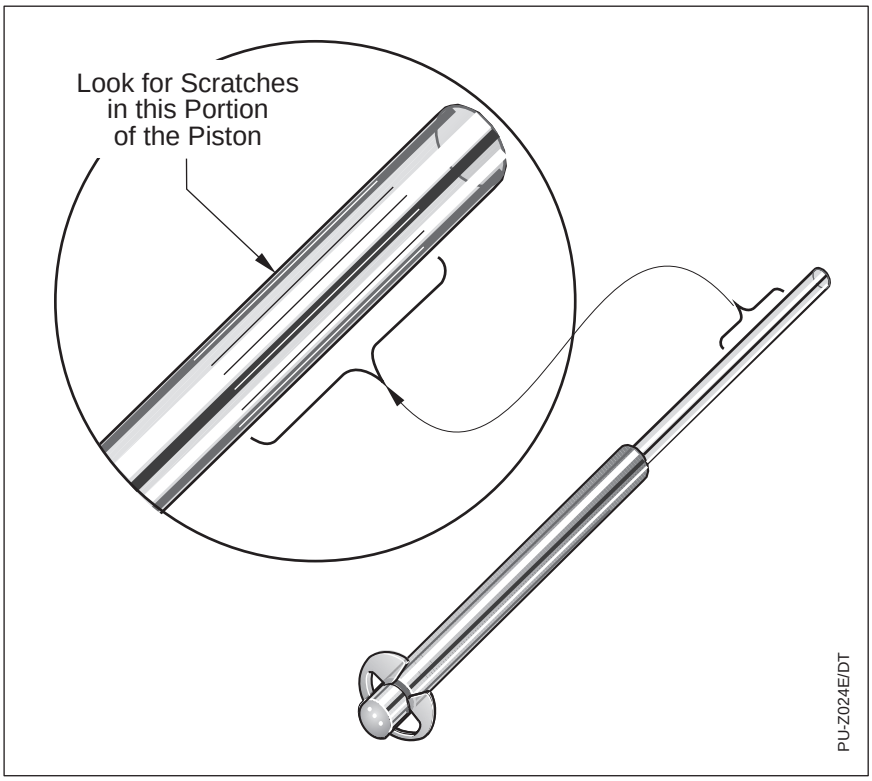


Figure 5.18 Piston scratches (under magnification)

Liquid End Assembly

To replace the piston seal and reassemble the liquid end:

1. Place the seal holder on end on a clean, flat surface. Place the seal holder into the pump head with the seal down (spring should face the inside of the pump head.) Install new piston seals by setting them in position on the seal holder (spring side up) and gently pressing them into place with the pump head (Figure 5.19).

NOTE: It is possible to install the seal in the wrong end of the seal holder. If installed in the wrong end, the seal will not be flush with the top of the holder. The opposite end of the seal holder is deeper, to accommodate the piston flush seal. Install the piston seal only in the end closest to the tubes.

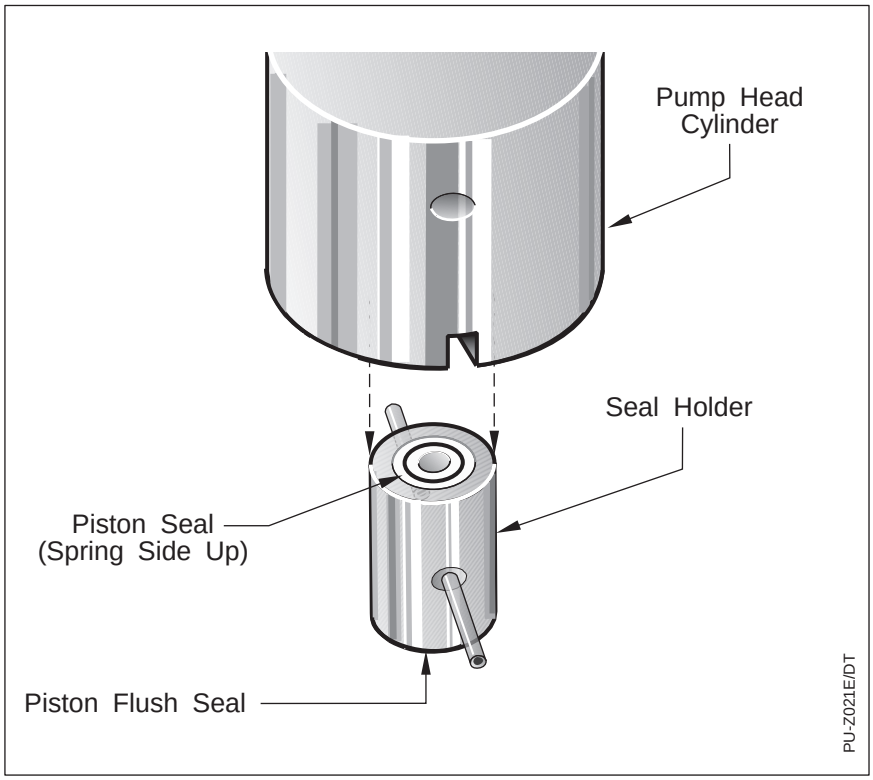


Figure 5.19 Seal installation

2. A piston flush seal (part of a Piston Flush Seal Kit and normally used with buffers) may be located at the opposite end of the seal holder. If you use a piston flush seal it should be replaced once a year. The piston flush seal is not subject to the higher pressures seen by the piston seal, so maintenance of this part is only occasionally necessary.

NOTE: The piston guide bushings do not need replacement. Retain them for new seal replacement.

To replace the piston flush seal:

a. Use the seal removal tool to remove the piston flush seal.

b. Insert a new piston flush seal into the seal holder (spring side down). The piston flush seal is thicker than a piston seal. The seal holder's cavity on the piston flush seal side is deeper to accommodate the larger size.

c. Ensure that the piston flush seal is flush with the edge of the seal holder. Use the large end of the seal removal tool to push the seal into the holder.

3. Place the seal holder into the piston holder housing spring side up (Figure 5.20).

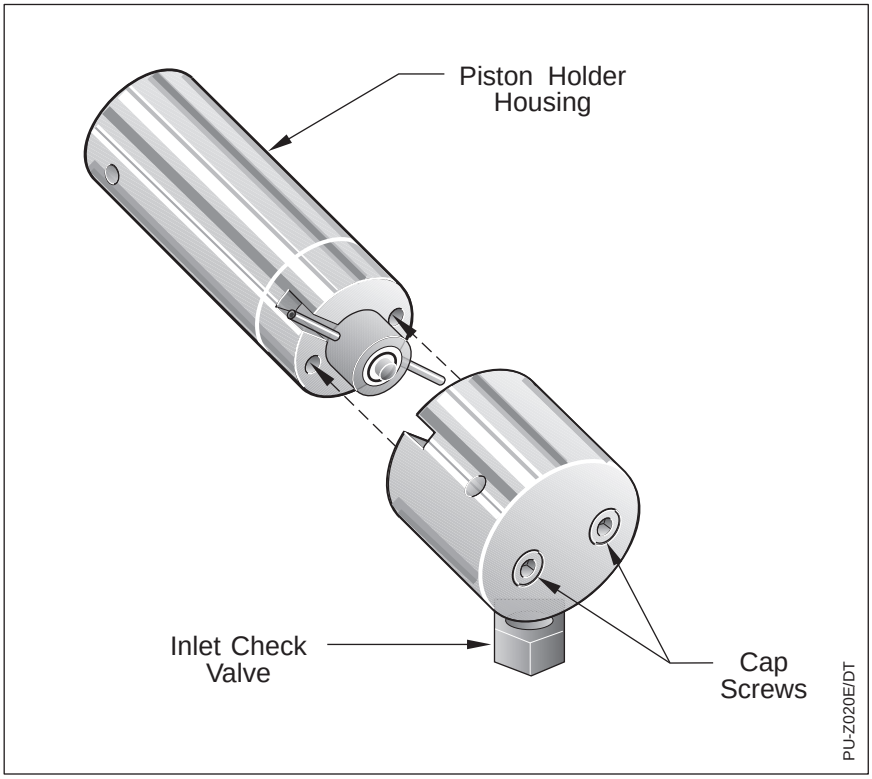


Figure 5.20 Seal holder alignment

4. If the Kel-F seal is being replaced, put the new seal in the pump head cavity now.
5. Install the pump head onto the housing using the two 9/64-inch Allen head screws.
   a) For the inlet liquid end, the pump head must be connected to the piston holder housing as shown in Figure 5.21 (Inlet check valve down, retaining cap screw to the left).
   b) For the outlet liquid end, the pump head must be oriented as shown in Figure 5.22.

   Evenly tighten the screws to forty inch-pounds (tight).

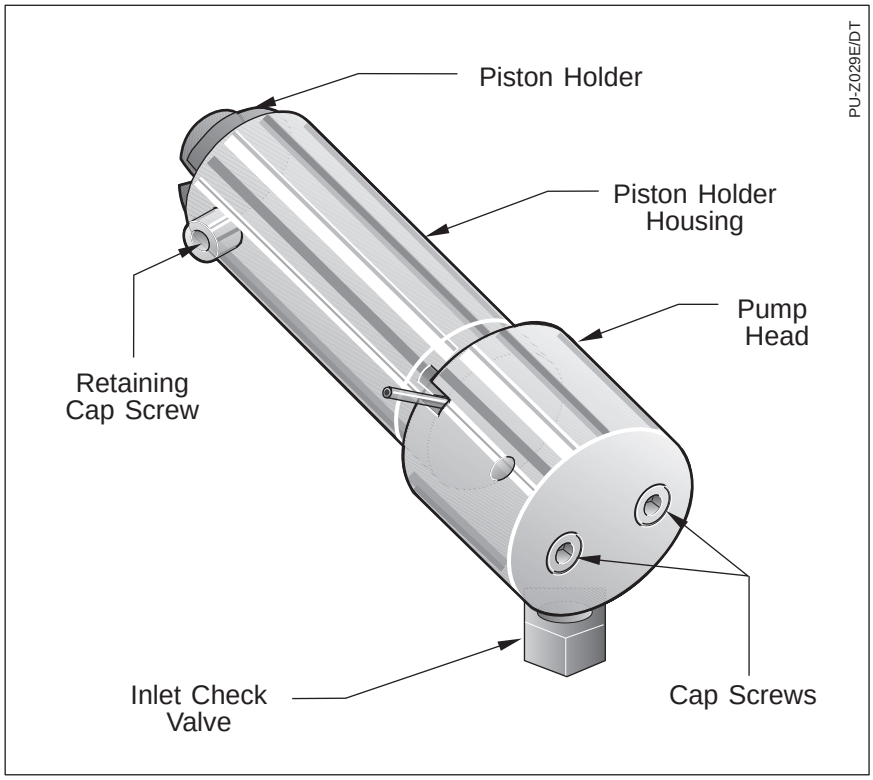


Figure 5.21 Installing the retaining cap screw (inlet liquid end)

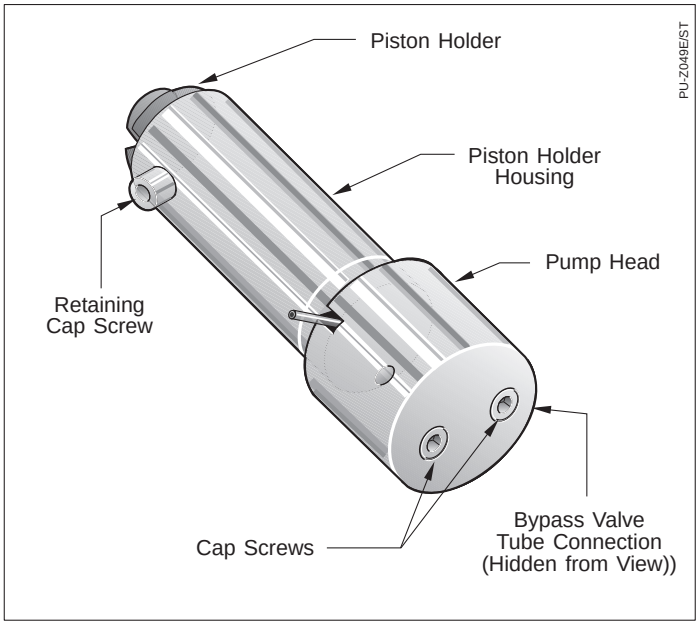


Figure 5.22 Installing the retaining cap screw (outlet liquid end)

6. Install the piston into the piston spring and then place them both into the piston holder housing (Figure 5.23). Do not press the piston through the seal at this time.
7. Compress the piston holder into the holder housing and install the retaining cap screw with its washer as shown in Figure 5.21 and Figure 5.22, so that the screw enters the slot in the piston holder. This action pushes the piston through the seal. Tighten the screw until snug.

NOTE: Make sure the retaining cap screw is oriented on the left-hand side of the piston holder housing for both the inlet and outlet liquid ends.

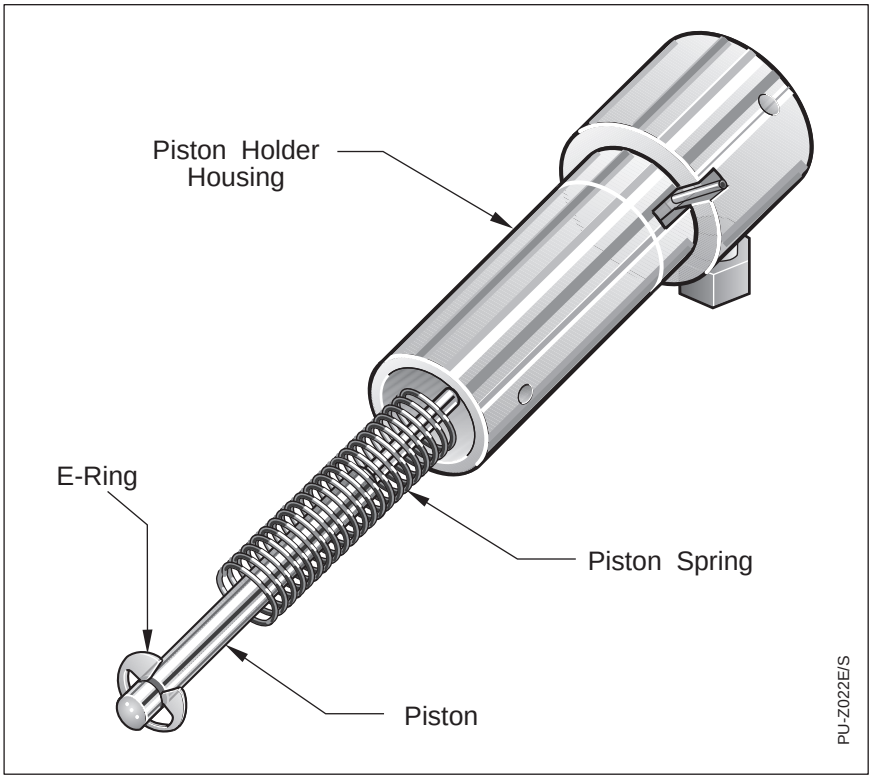


Figure 5.23 Piston installation

Liquid End Installation

To install the liquid end assemblies into the pump, the pump must be in the maintenance position. If the pump has not been turned off since the liquid ends were removed, the pump motor should still be in its maintenance position. If not, press [MENU], /TESTS/, /Maintenance Log/ then move the cursor to /Maintenance Position/ and press [ENTER]. The display shows:

```
To install or remove
liquid ends, press ENTER
```

Figure 5.24 Maintenance position message

Press [ENTER] to continue. The pump motor rotates for a few seconds and then is electrically locked into position. While the motor rotates the display shows:

```
Install or remove liquid
ends when motor stops
```

Figure 5.25 Pump rotating cam to maintenance position

The pump is now in its maintenance position. The display will appear as shown in Figure 5.26. The liquid ends can be installed.

```
X-ducer      (date)      (due)      (vol)
· Maintenance Position
```

Figure 5.26 The display after the motor moves cam to the maintenance position

1. Replace the inlet liquid end first. This liquid end contains tapped holes for the inlet check valve and the transducer tube. Install it by pressing in and turning it approximately 90 degrees counter-clockwise, until it locks into position. Be sure that the check valve (or the check valve hole, if the valve is not installed) is pointed down, and the transducer tube hole is pointed to the upper left-hand side (10 o'clock) (Figure 5.27).

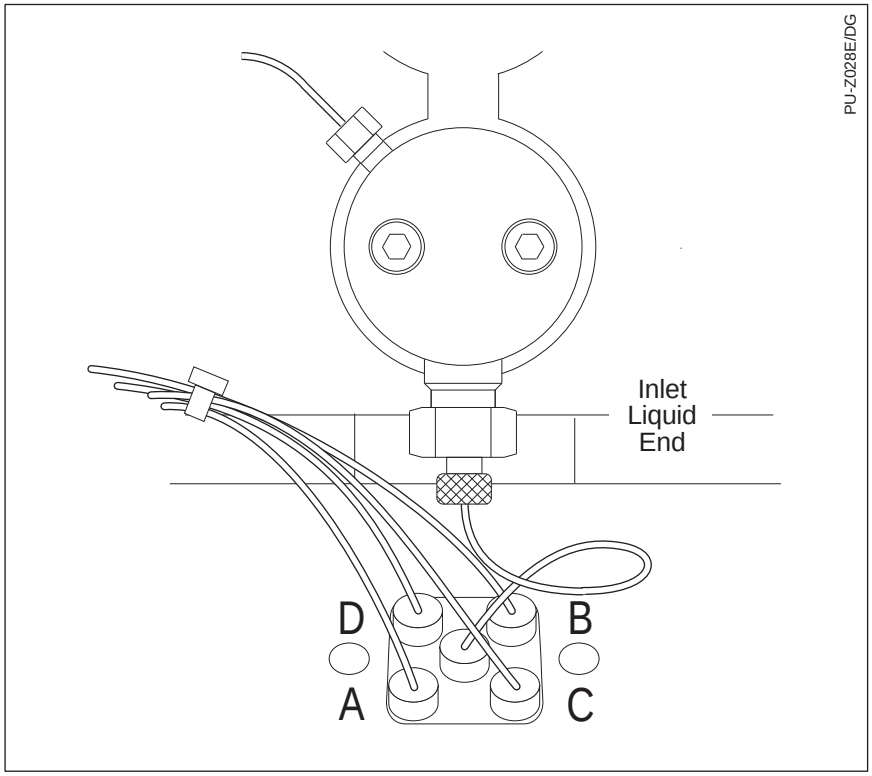


Figure 5.27 Inlet liquid end

2. Replace the outlet (upper) liquid end. This liquid end contains tapped holes for the crossover tube and the bypass valve tube. This liquid end is installed by pressing in and turning it approximately 90 degrees clockwise, until it locks into position. Be sure that the crossover tube hole is pointed up, and the bypass valve tube is pointed to the lower right-hand side (4 o'clock).
3. Replace the check valve and tubing. (Do not over-tighten fittings.) Generally, a 1/16-turn beyond finger-tight is sufficient to make a leak-free connection.

HINT: If the transducer check valve has been removed you can distinguish the two check valves: the inlet check valve has a wider fitting opening than the transducer check valve (connected to the pressure transducer).

4. After replacing the liquid ends, reset the VOL values in the Maintenance Log for all replaced components to zero (0).

POST-INSTALLATION SEAL CONDITIONING

Follow the procedure below each time you replace liquid end seals.

1. Open the column bypass valve and purge with methanol to clear air from the solvent lines and liquid ends.
2. Purge the pump through an old column or flow restrictor at 4000 psi for a minimum of 20 minutes. Reduce the flow to 1 mL/min and continue pumping for 15 minutes.
3. Check for solvent leaks. Do not return the pump to every-day service unless you are sure that no leaks are present.

CHECK VALVE MAINTENANCE

If the pump has notified you that it is time to replace check valves or if check valve replacement was recommended in Appendix A, *Troubleshooting*, then follow these steps. Check valve maintenance consists of:

- Inlet check valve removal and installation
- Transducer check valve removal and installation

NOTE: The factory-supplied replacement check valves are manufactured in a clean-room environment and capped to protect them from contamination. It is very important to maintain a clean environment when installing them.

Inlet Check Valve (bottom position)

To remove the existing inlet check valve and install a new one:

1. Remove the solvent inlet tube (Figure 5.27) from the check valve.
2. Remove the defective check valve by rotating the valve counter-clockwise with a 1/2-inch open-end wrench.
3. Install the new check valve by rotating clockwise until the valve is snug against the liquid end cylinder. Reconnect the inlet tubing. Tighten to finger-tight.

Transducer Check Valve

To remove the existing transducer check valve (connected to the Pressure Transducer) and install a new one:

1. Remove the transducer tubing from the transducer check valve and the inlet pump head (Figure 5.28).

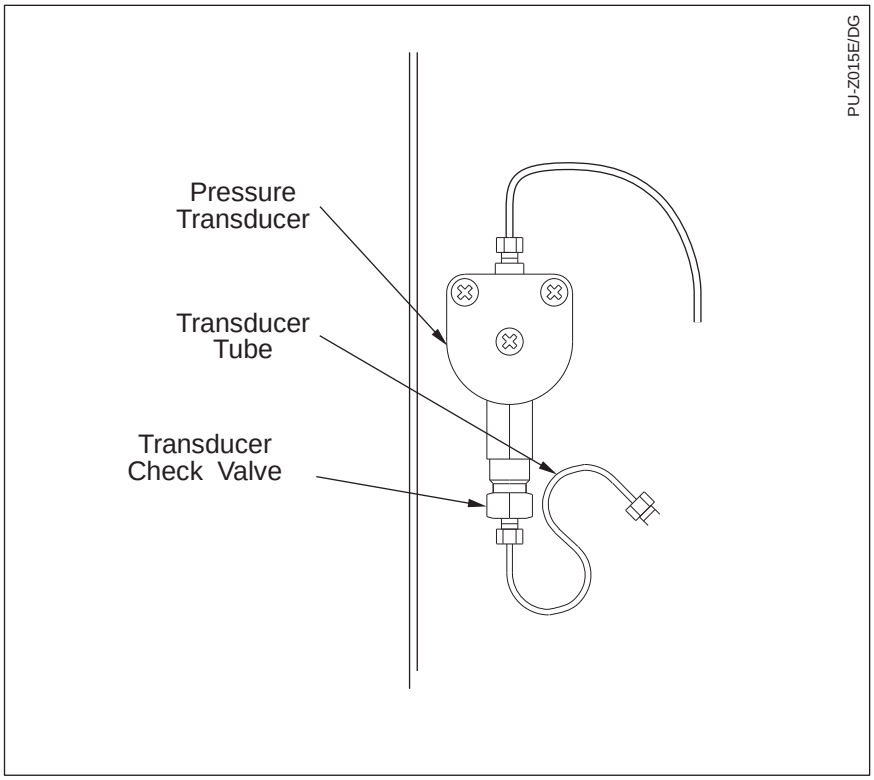


Figure 5.28 Transducer check valve

2. Remove the defective check valve by rotating it counter clockwise with a 1/2-inch open-end wrench.

3. Install the new check valve by rotating it clockwise until snug, and tighten with a 1/2-inch open-end wrench. Replace the connecting tubing. Tighten fittings only enough to stop leaks. Generally, this is 1/16-turn beyond finger-tight.

Maintenance Tips

This section contains useful maintenance tips for pump parts not directly related to solvent flow.

DRIP TRAY

A removable, white plastic solvent drip tray is located underneath the inlet bracket of your pump.

To remove the tray squeeze the top, front-edge of both sides of the tray together and carefully pull the tray out. You may need to wiggle the tray as you pull. If you see solvents in the tray, be particularly careful not to spill them as the tray is removed.

HINT: It may be easiest to use the index finger of each hand to push the sides together.

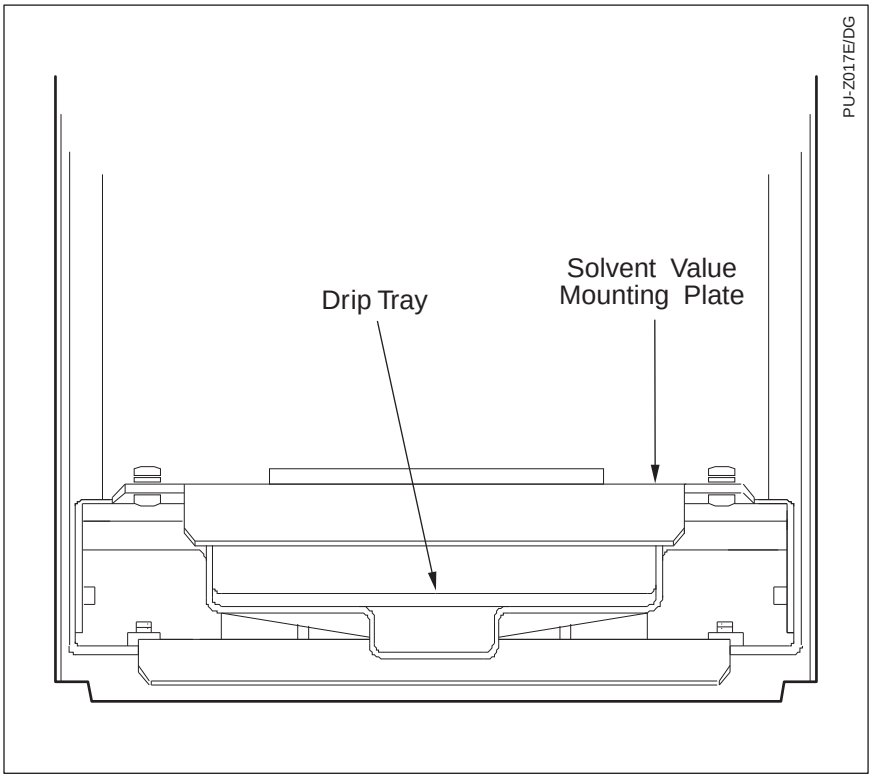


Figure 5.29 Drip tray installed

PASSIVATION OF STAINLESS STEEL COMPONENTS

All the major type 316 stainless steel components used in the SpectraSYSTEM pumps are passivated prior to assembly to ensure the removal of porous particles from the surface and to coat the surface with a layer of chromium oxide, which is highly resistant to corrosion. All stainless steel replacement parts purchased from Thermo Fisher Scientific are also passivated.

However, stainless steel components are subject to corrosion from strong acid solutions (in particular, materials containing halides), organic acids, and sometimes even water. Resistance to corrosion of the stainless steel components can be enhanced by using the following procedures.

CAUTION—Chemical Hazard! Take care when passivating with strong acids. Wear protective eye covering and protective clothing.

NOTE: Before installing any new parts not supplied from the Factory such as stainless steel tubing, the parts should first be passivated using the methods below.

NOTE: DO NOT expose a column to the passivation mixture. Remove the column before pumping if it is necessary to pump passivation solvents through the pump. It is preferable, however, to remove the components from the pump and then passivate them apart from the system.

1. When the surface area to be passivated is thoroughly clean, it is passivated by wetting the surface with a 20% nitric acid solution in deionized water for about 10 minutes at room temperature.
2. After passivation, thoroughly clean the parts to remove any residual nitric acid. Wash with deionized water until the system is neutral to pH paper. Follow up with another wash using 50-50 water/methanol followed by methanol. When thoroughly clean, blow dry using nitrogen. (Do not use the laboratory air system or air from a compressor that may contain an oily residue.)

Repair Instructions

If troubleshooting has pointed to a blown fuse in the power entry module, or to the need to replace the solvent switching valve (P4000), use the procedures below to make repairs.

POWER ENTRY MODULE FUSE REPLACEMENT

Instrument power is supplied by two 4.0-amp fuses housed in the fuse compartment of the power entry module, above the power cord receptacle.

To replace the fuses:

1. Ensure that the power cord is *not* connected to the pump.
2. Use a small, flat blade screwdriver to pry open the power selector/fuse cover. You will probably hear the top edge of the cover snap as it is pried open.
3. Pull out the fuse holder and discard the bad fuse. Place the new fuse into the holder with the metal end visible.
4. Snap the fuse holder back into place.

NOTE: If the power selector barrel accidentally comes out, be sure to replace it so that the correct voltage for your area shows through the voltage window.

5. Firmly snap the housing cover back in place. Be sure that the correct voltage is visible in the voltage window.

HINT: Use two thumbs to push up on the top half of the cover as you push in.

SOLVENT SWITCHING VALVE REPLACEMENT (P4000 ONLY)

To remove the solvent switching valve:

1. Turn off power.
2. Remove front cover.
3. Remove the solvent inlet tube fitting from the inlet check valve.
4. Loosen the two knurled screws on the front-left and front-right sides of the inlet bracket.
5. Lift and pull the bracket forward 3 - 4 inches.
6. Disconnect the inlet tubes from the solvent switching valves at locations A and B.
7. Remove the solvent inlet line from the center port of the valve.
8. Disconnect the solvent switching valve cable at the rear of the solvent valve cavity, and remove the solvent valve/bracket fully from the pump.

To install a new solvent switching valve:

1. Connect the replacement valve's cable to the connector at the rear of the solvent valve cavity.
2. Connect the solvent inlet line to the center port of the replacement valve. Tighten snugly but only finger-tight.
3. Re-connect the inlet tubes to the valve ports, making sure the identification of each inlet tube matches the identification of each valve port location.
4. Install the valve and bracket into the solvent valve cavity. Tighten the knurled screws.
5. Attach the solvent inlet line to the inlet check valve. Tighten snugly but only finger-tight.
6. Prime the pump using the technique described in Chapter 1.

A Troubleshooting

Introduction

Your SpectraSYSTEM pump is designed to operate trouble-free for many years when properly maintained. Most pump problems can be avoided by simple, periodic maintenance, as described in Chapter 5. However, in the event that an error message is displayed or if a mechanical or electrical failure is suspected, the problem can be easily diagnosed. Further, if the diagnosis indicates that a problem exists with non user-serviceable parts inside the pump, a qualified Thermo Fisher Scientific service representative can quickly and easily replace most malfunctioning parts.

This appendix contains information on:

- Theory of operation
- General LC system troubleshooting
- Pump-specific Troubleshooting
- Error Messages
- Display Messages

A quick-reference hardware troubleshooting guide is included at the end of this appendix that includes tips for diagnosing and remedying hardware problems. This guide can save you time in diagnosing problems when the symptoms are known.

Theory of Operation

A gradient pump works by first pulling a solvent into a proportioning valve. The P4000 pump has four proportioning valves. Solvents are measured by percentages, as specified by the user, and mixed inside the pump. The solvents then travel to the pump head, where a piston regulates the flow of the mixture to an outlet tube. The mixed solvent is routed through the pressure transducer, into a second pump head, then through a bypass valve (closed during normal operation), and finally out to the LC system. The pump's outlet tubing is then generally connected to an automatic injector, such as an autosampler.

Troubleshooting Your Pump

SAFETY PRECAUTIONS

Observe the following safety precautions whenever troubleshooting hardware difficulties.

Caution!
A caution alerts you to situations that could result in personal injury. It also tells you how to avoid them.

High Voltage!
This icon alerts you to the presence of high voltage and to the potential injury that could occur from electrical shock were you to come in contact with a specific instrument area or component. It also tells you how to avoid contact with the high-voltage areas in your instrument.

Hot Surface!
This icon alerts you to potential injury that could occur from coming in contact with a heated surface or area on or in an instrument. It also tells you how to avoid contact with the heated surfaces in your instrument.

TROUBLESHOOTING TIPS

Eliminate all Other possible sources of trouble
Before you spend any time trying to diagnose a suspected pump problem you should verify that the pump is the only source of difficulty. Systematically eliminate all other instruments in your LC system as the source of trouble. If you are not sure which component of your chromatography system is responsible for poor system performance the *General LC System Troubleshooting* section beginning on page 159 of this appendix provides useful suggestions.

When You're Sure Its the Pump
Once you have isolated the pump as the only remaining source of difficulty, a variety of self-tests are built into the pump to help you determine if your pump is operating correctly.

The next section describes the tests found in the /TESTS/ menu item. Instructions for the built-in hardware and electronics diagnostics are contained within the description of the Tests Menu.

Table A.1 General Troubleshooting Table

Symptom	Cause/Remedy
1. No flow.	a) Check mobile phase connections. b) Check for leaks. c) Check pump troubleshooting guide.
2. High back pressure.	a) Check flow rate and system/column specifications. b) Check for tubing or column blockage. c) Check pump troubleshooting guide.
3. Unstable baseline or drift.	a) System/column not equilibrated; allow more time. b) Check detector troubleshooting guide. c) Check pump troubleshooting guide.
4. Baseline noise.	a) Check for air bubbles in system, degas solvents. b) Check for system/solvent contamination. c) Check pump troubleshooting guide.
5. No peaks.	a) Check detector and data system connections. b) Check autosampler troubleshooting guide. c) Check sample retention with chromatographic conditions.
6. Contaminating/ghost peaks.	a) Clean system and column. b) Check autosampler troubleshooting guide. c) Check pump troubleshooting guide.
7. Poor peak shape.	a) Check system for leaks. b) Check fittings and tubing lengths. c) Check column performance. d) Check autosampler troubleshooting guide. e) Check pump troubleshooting guide. f) Check detector troubleshooting guide.

Table A.1 General Troubleshooting Table, *continued*

Symptom	Cause/Remedy
8. Poor retention time reproducibility.	a) Check system for leaks and bubbles. b) System/column not equilibrated, allow more time. c) Check column performance. d) Check pump troubleshooting guide. e) Check autosampler troubleshooting guide. f) Check data system troubleshooting guide.
9. Poor peak area reproducibility.	a) Check column performance. b) Check autosampler troubleshooting guide. c) Check data system troubleshooting guide.
10. Non-integrated or too many peaks.	a) Check integrator or data system troubleshooting guide.
11. No instrument of device control.	a) Check cable connections. b) Check system configuration. c) Check individual instrument troubleshooting guide. d) Check integrator or data system troubleshooting guide.

For more detailed chromatographic troubleshooting, refer to any HPLC troubleshooting reference book or call your local sales or service representative.

Table A.2 Pump-Specific Hardware Problems

Symptom	Possible Cause	Diagnostic/Remedy
1. No response when power is switched on.	a) Power cord not firmly installed.	a) Re-seat cord.
	b) Power cord defective.	b) None. Replace cord.
	c) Power Entry Module Fuse blown.	c) None. Refer to page 154 for fuse replacement.
2. No display. Fan and pump run OK.	a) Display contrast needs adjustment.	a) Press [STATUS] then press and hold the right-arrow key. Press the [+] or [-] key to adjust the display contrast. Press [STATUS] again.
	b) Internal fuse blown.	b) None. Contact Thermo Fisher Scientific representative for service.
3. No display.	a) System PCB defective.	a) None. Contact Thermo Fisher Scientific representative for service.
4. Only fan runs with power on.	a) Internal fuse blown.	a) None. Contact Thermo Fisher Scientific representative for service.
	b) Display cable loose/unplugged.	b) None. Contact Thermo Fisher Scientific representative for service.
5. No response to keypad entry. Display OK.	a) Keypad defective or System PCB defective.	a) None. Contact Thermo Fisher Scientific representative for service.
6. Random display.	a) Display defective.	a) Display Test (page 120). Contact Thermo Fisher Scientific representative for service.
7. Pump motor will not run.	a) Max pressure or flow rate set to zero.	a) Press [STATUS]. Set Maximum Pressure to a value > 100 psi.
	b) Motor defective.	b) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	c) Motor cable unplugged.	c) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.

Table A.2 Pump-Specific Hardware Problems, *continued*

Symptom	Possible Cause	Diagnostic/Remedy
	d) Drive circuit.	d) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	e) Internal fuse blown.	e) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	f) Target pressure low.	f) None. Press [PURGE] and then reinitialize file.
8. Oil found on inlet bracket.	a) Main bearing over-lubricated.	a) None. Small amount of oil is normal.
9. Improper composition.	a) Proportioning valve cable loose.	a) Hardware Series Test (page 121). Reconnect cable.
	b) Proportioning valve defective.	b) Hardware Series Test (page 121). Replace solvent proportioning; switching valve.
	c) Proportioning valve drive defective.	c) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	d) Cam marker failure.	d) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
10. Proportioning valve does not click open.	a) Internal fuse defective.	a) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	b) Proportioning valve drive failure.	b) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.

Table A.2 Pump-Specific Hardware Problems, *continued*

Symptom	Possible Cause	Diagnostic/Remedy
11. Pump will not go READY (possible unstable flow)	a) Check valve failure.	a) Flow Stability Test, page 34. Check Valve Test page 112. Replace check valve - see Appendix C.
	b) Immiscible solvents.	b) Flow Stability Test, page 34. Check Valve Test, page 112. Change solvent system.
	c) Unstable load (column).	c) Flow Stability Test, page 34. Check Valve Test, page 112. Pump must see stable, non-compressible load.
	d) Insufficient degas.	d) Flow Stability Test, page 34. Check Valve Test, page 112. Increase helium rate (if helium degas) or decrease flow rate (if vacuum degas). Use vent line and good bottle cap seal.
	e) Circuitry failure.	e) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
	f) Partially clogged frit or filter on high pressure side of pump.	f) Hardware Series Test (page 121). Replace filter or frit.
12. Sudden shift in pressure display with no flow.	a) Pressure transducer failure.	a) None. Contact Thermo Fisher Scientific representative for service.
	b) Circuitry failure.	b) Hardware Series Test (page 121). Contact Thermo Fisher Scientific representative for service.
13. Pump goes from RUN immediately to READY (will not maintain RUN state).	a) Run file has only one time line.	a) View run file. Add time line and reload file.
14. Pump will not start or stop remotely.	a) Incorrect wiring.	a) External Inputs Test 120. Correct wiring.

Table A.3 Pump Operation Error Messages

BELOW MINIMUM PRESSURE;	The column pressure has fallen below the file's Minimum Pressure setting. Check for mobile phase leaks.
CHECKSUM ERROR BAD PROGRAM;	The program memory may have been corrupted. Make a note of the circumstances which preceded the message and contact Thermo Fisher Scientific.
CODE ERROR PROGRAM LOST;	The program code has errors. Make a note of the software version and the circumstances which preceded the message. Contact Thermo Fisher Scientific.
CODE ERROR STACK UNDERFLOW;	The program code has errors. Make a note of the software version and the circumstances which preceded the message. Contact Thermo Fisher Scientific.
CODE ERROR STACK OVERFLOW;	The program code has errors. Make a note of the software version and the circumstances which preceded the message. Contact Thermo Fisher Scientific.
CODE ERROR FALSE POWER FAIL;	You may have experience low line voltage ("brown-out") or there may be hardware problems. Note the circumstances which preceded the message and contact Thermo Fisher Scientific.
EXCEEDS FLOW RANGE	A flow rate was entered in the pump file which exceeded the flow rate capabilities of the pump. The maximum flow rate for standard SpectraSYSTEM pumps is 10 mL/min. If higher flow rates are needed, inert/biocompatible liquid ends are available to extend the flow rate to 30 mL/min. Contact your local sales representative for information.
MAX PRESSURE EXCEEDED;	The column pressure of the system has exceeded the MaxP (maximum pressure) value entered into the pump file. The file's Maximum Pressure value may need to be increased. The default value is 3000 psi for 10 mL/min liquid ends (the maximum is 6000 psi). If your operating column pressure is increasing, check for column plugging.
MOTOR STALLED	The motor is unable to maintain the combination of requested flow rate and needed pressure for operation. Reduce flow rate or check for flow restriction or plugged column frit.
OVER MAXIMUM TEMPERATURE;	The pump has overheated. This may be due to blocked ventilation slots or to a hardware malfunction. If necessary, clean the air filter.
POWER FAILURE CONTINUE;	A power failure has occurred or the pump was switched off with the motor running. The pump has automatically resumed operation. ("Continue" was selected in /OPTIONS/, /Error Recovery/, AC Power Fail.)
POWER FAILURE STOP;	A power failure has occurred or the pump was switched off with the motor running. The pump has automatically stopped. ("Stop" was selected in /OPTIONS/, /Error Recovery/, AC Power Fail.)
POWER FAILURE SHUTDOWN;	A power failure has occurred or the pump was switched off with the motor running. The pump has automatically loaded and run the shutdown file. ("Shutdown" was selected in /OPTIONS/, /Error Recovery/, AC Power Fail.)
ZERO FLOW RATE	A time line (other than Time = 0.0 min) with a zero flow rate was encountered. To remedy, enter a valid flow rate in the first line of the pump file. Rates between .01 and 10 mL/min are valid for standard SpectraSYSTEM Pumps. The optional inert/biocompatible liquid ends extend the maximum flow rate to 30 mL/min.

Table A.4 Display Messages

Cannot Load File:	(P4000 only) The five messages below indicate that Develop File parameters are inconsistent.
Solvent1 = Fixed Solvent Solv 1 = Solv not used Start%1 + Fixed% > 100 End % 1 + Fixed % > 100 Solvent 1 = Solvent 2	
#Runs > 999	Too many runs are set up. Edit the develop file parameters to reduce the total number of runs.
Maintenance Due See Log	A volume milestone has been reached. Consult the Maintenance Log for component by pressing the [MENU] key and selecting /TESTS/, /Maintenance Log/. For more information, refer to Chapter 5.
Memory Full File Not Copied	There is not enough memory available to copy the parameters of one file into another. Free memory by deleting an old or unused file, or by reducing the number of time lines in a file. Try to copy the file again.
Memory Nearly Full Data May Not Be Saved	There may not be enough memory available. Double-check the file to ensure that no parameters or settings were lost. Free memory by deleting an old or unused file, or by reducing the number of time lines in a file. Try to save the file again.
No Queue Available	You cannot load a queue if none has been set up first.
Not Enough Room File Not Saved	The run file changes cannot be saved to the file. Free memory by deleting an old or unused file, or by reducing the number of time lines in a file. Try to save the run file once more from the Status Menu.
Protected File Cannot Be Copied To	You cannot modify a protected file.
Protected File Cannot Be Deleted	You cannot modify a protected file.
Protected File Cannot Be Edited	You cannot modify a protected file.
Queue Loaded Cannot Load File	When a queue is loaded you cannot load any other file without first pausing the queue.
Run In Progress No Testing Allowed	The test cannot be initiated because the pump is in RUN or is in HOLD.

Table A.5 Solvent Compatibility for Pump Wetted Surfaces

Reagents	Stainless Steel (316)	Polyetheretherketone (PEEK)	Piston Seal Gold	Piston Seal Black	PTFE	Check Valve Ceramic
Acids						
Acetic, 10%	Excellent	Excellent	Excellent	Limited	Excellent	Excellent
Acetic, 50%	Limited	Excellent	Excellent	Limited	Excellent	Excellent
Hydrochloric, 10%	Unsatisfactory	Excellent	Excellent	Limited	Excellent	Limited
Hydrochloric, 50%	Unsatisfactory	Excellent	Excellent	Limited	Excellent	Limited
Nitric, 10%	Excellent	Excellent	Excellent	Limited	Excellent	Excellent
Nitric, 50%	Limited	Excellent	Limited	Limited	Excellent	Excellent
Phospohoric, 10%	Excellent	Excellent	Limited	Limited	Excellent	Excellent
Phosphoric, 50%	Limited	Excellent	Limited	Limited	Excellent	Excellent
Sulfuric, 10%	Limited	Excellent	Excellent	Limited	Excellent	Excellent
Sulfuric, 50%	Unsatisfactory	Excellent	Excellent	Limited	Excellent	Excellent
Water, 100%	Excellent	Excellent	Excellent	Unsatisfactory	Excellent	Excellent
Bases						
Ammonium Hydroxide, 10%	Excellent	Excellent	Excellent	Limited	Excellent	Excellent
Sodium Hydroxide, 10%	Limited	Excellent	Excellent	Limited	Excellent	Excellent
Sodium Hydroxide, 50%	Unsatisfactory	Excellent	Excellent	Limited	Excellent	Excellent
Organics						
Acetone, 100%	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent
Acetonitrile, 100%	Excellent	Unsatisfactory	Unsatisfactory	Excellent	Excellent	Excellent
Chloroform, 100%	Excellent	Unsatisfactory	Unsatisfactory	Unsatisfactory	Limited	Excellent
Dimethyl Sulfoxide, 10%	Excellent	Limited	Limited	Limited	Excellent	Excellent
Dimethyl Sulfoxide, 80%	Excellent	Unsatisfactory	Unsatisfactory	Unsatisfactory	No Data	No Data
Isopropanol, 100%	Excellent	Limited	Excellent	Excellent	Excellent	Excellent
Methanol, 100%	Excellent	Limited	Excellent	Excellent	Excellent	Excellent
Methylene Chloride, 100%	Excellent	Unsatisfactory	Unsatisfactory	No Data	Excellent	Excellent
Tetrahydrofuran, 10%	Excellent	Limited	Excellent	Excellent	Excellent	Excellent
Tetrahydrofuran, 80%	Excellent	Unsatisfactory	Limited	Limited	Excellent	Excellent
Toluene, 100%	Excellent	Unsatisfactory	Unsatisfactory	No Data	Limited	Excellent
Urea, 100%	Excellent	Excellent	Excellent	No Data	Excellent	Limited

Excellent = Standard or better lifetime with no appreciable chemical attack
Limited = Variable lifetime given actual mobile phase composition, pressure, and temperature
Unsatisfactory = Significant chemical attack and decrease in lifetime

B

Glossary

A

autosampler — an instrument designed to automatically inject samples into the sample flow path with a high degree of precision and reproducibility; sometimes called an injector

B

baseline — the reference line at the bottom of a chromatogram from which measurements are made; a baseline represents the chromatogram that would be drawn if only the mobile phase (with no sample) were run through the column

binary — capable of mixing or switching between two solvents

biocompatible — describes components that are inert when used with biological samples; biocompatible components are usually made from titanium, PEEK, Teflon, quartz, or sapphire

blend — a purge option in the P4000 pump allowing all solvents to be purged simultaneously

buffer — a substance that can neutralize both acids and bases

C

channel — the path along which something (solvent or information) flows

chromatogram — a plot depicting the separated components in a sample (absorbance units versus time); each component is shown as a separate peak whose concentration can be determined by studying the area under the peak

chromatograph — the basic set of instruments needed to perform chromatography: a pump, injector (manual or automatic), a column, and a detector; various recording and data handling instruments are common additions

chromatography — a means of separating and analyzing mixtures of chemical substances

column — the packed tube through which a sample is passed for separation; the sample separates according to the way in which it adheres to the column's packing material

component — an "ingredient" in a chemical mixture, also the individual parts of a liquid end assembly

conditioning the process of preparing the surface of the column wall and introducing the buffer pH conditions into the column before a run

configuration the way instruments are interconnected to form a system

cursor a moving or blinking symbol on the display which indicates where information is entered

D

default a value or choice built into a system; if no specific choice is made, instruments will run (or data analyzed) using the default settings

degassing removal of dissolved gas (i.e. oxygen) from the solvent to prevent bubbles from forming in the pump; degassing can be done by vacuum or by sparging

detector the instrument used to detect the presence of a chemical compound

develop file in the P4000 pump a file which allows a comprehensive set of parameters to be changed, automatically and sequentially

diagnostics ways of detecting and isolating instrument or software problems

digit an editable space within a field

display the backlit LCD screen on all SpectraSYSTEM™ instruments

E

elution time the length of time needed to pass a particular sample through a packed LC column

equilibration the process used to bring a system (solvent, column, and so on) to a point of equilibrium, where all thermal and chemical reactions occur at equal rates; a stable baseline is a good sign of a well-equilibrated system

error message a printed or displayed message that notifies the user of an error condition

error recovery user selectable responses to error conditions detected by the instrument, such as a power interruption or over-pressure

external event an action performed by an external device that is under the control of the current instrument (see also timed event)

F

field — an area in a display, screen, or menu where an entry is required or a choice must be made

file protect — a setting which allows files to be edited when "off" and protects files from being changed by editing when "on"

flow parameters — flow rate, run time and solvent composition

flow rate — the rate at which solvent flows through a system

G

gradient — changes the percentage composition of two or more solvents over time; changes may be continuous or in steps

gradient curve — pre-programmed gradient time line accessed in the Develop file of the P4000 pump. Also refers to the shape of the solvent composition curve between two time lines.

ground terminal — a terminal used to connect the ground or earth lead of a signal or contact closure cable; generally green and/or black

H

helium manifold — a pneumatic assembly containing valves and switches for regulating helium sparging

helium sparging — see sparging

I

inert — see biocompatible

injection — the manual or automatic introduction of a sample into a chromatography system

integrator — the instrument used to analyze data and produce a chromatogram

isocratic — constant flow and solvent composition

K

Kel-F seal — the translucent seal, made of Kel-F material, inside the pump head which faces the piston seal

keypad — all of the keys by which you can communicate with an instrument or computer

L

LC — Liquid Chromatography

linear — a gradient curve that follows a straight line

liquid end — the inlet or outlet assemblies of the pump consisting of the head, piston, seals, and sometimes a check valve

M

maintenance log — a place to record dates, service, and cumulative solvent volume pumped

menu — a list of choices

method — the set of parameters that define how one or more analyses will be accomplished

method development — the process of specifying the parameters under which an instrument will perform a particular function

N

NOVRAM — Non-volatile RAM (random access memory). Computer memory into which the user can enter information and instructions and from which the user can recall information. Data in NOVRAM are saved even when the instrument is switched off

P

parameter — a value or set of values used to define the characteristics or behavior of an instrument or system

PEEK — polyetheretherketone; a material frequently used in fabricating inert/biocompatible components

piston — the short cylinder piece which moves inside the sealed cylindrical opening and used to pressurize fluid

piston holder housing	the shaft into which the piston and its holder are housed
piston flush seal	the low-pressure spring seal inside the liquid ends, facing the piston assembly
piston seal	also called pump seal, a high-pressure spring seal located inside the pump head
plot	the presentation of analytical data in a graphical manner; typical plots include chromatogram traces and calibration curves
prime	to flush the solvents contained in a new pump in order to prepare the pump for solvents chosen by the user
proportioning	the process of opening and closing solenoid valves in sequence to create a desired solvent composition; usually makes a clicking sound as the solvent composition is generated
pump	the instrument used to push a liquid solvent through a chromatography system
purge	to flush the system with fresh, degassed solvent

Q

quaternary	capable of mixing or switching between as many as four solvents
queue	a set of files in a prearranged order

R

RAM	Random Access Memory (computer)
real-time	the current, actual time
reproducibility	the precision with which a piece of data can be repeated; a good measure of a system's overall performance
retaining screw	also retaining cap screw; the screw which holds the piston assembly into the piston holder housing
run	a complete analytical operation cycle of the chromatographic system
run file	the file that has been loaded and that the pump is currently operating by
run time	the duration of a sample run, from injection to separation

S

sample — a known or unknown substance in a small quantity

seal holder — a metal part used for pumps fitted with standard parts which contains two seals, (piston seal and piston flush seal) and allows the pump head and piston holder housing to be joined

shutdown file — a special file used by the pump after the pump has been in a READY state for a period of time set by the user

solvent — a substance that can completely dissolve another; the mobile phase of an LC system

solvent filter — a small cylindrical attachment for inlet tubing used to filter a solvent prior to the solvent entering a pump

solvent program — a set of time lines indicating pump flow and solvent composition at specific times during a run

solvent-strength gradient — a gradient in which the secondary solvents' composition ratios remain constant during the run

sparging — a degassing technique in which solvent gases are replaced with an inert gas such as helium or nitrogen

status — the current condition

status lock — a feature used to prevent a run file from being changed from the Status Menu

step gradient — a gradient created in step-wise fashion using two solvents, pumped through a binary, isocratic, solvent delivery system

stroke — one complete revolution of the pump's cam which displaces both pistons

system — a set of chromatography instruments that operate together in a concerted manner to produce an analytical result

T

timed event — an instrument action triggered to occur at a specific, preset time during a run or analysis

trace — a chromatogram

transducer check valve — the valve which attached to the inlet of the pressure transducer

V

vacuum degassing	the technique of removing dissolved gasses from solvents by passing the solvent through tubing made of gas-permeable membrane, and creating a vacuum around the tubing, thus allowing gasses to be evacuated out of the solvent and into the surrounding chamber
viscosity	the degree to which a fluid resists flow

C

Kits and Parts Lists

Introduction

This chapter contains unpacking lists and information for several kits and accessories available from Thermo Fisher Scientific for use with your SpectraSYSTEM pump. Described in this chapter are:

- Accessory Kit p/n A4070-010
- Standard Maintenance Kit p/n A4050-010
- Standard LC Fittings Kit p/n A4051-010
- Piston Flush Seal Kit (10 mL) p/n A4114-010
- Piston Seal Kit (30 mL) p/n A4084-010
- Solvent Inlet Tube Kit p/n A4074-010
- Solvent Tube Extension Kit p/n A4117-010
- Manual Injection Valve Bracket Kit (also included with A4052-010, the Rheodyne 7125 Standard Bracket Kit, and A4053-010) p/n A4054-010
- Narrow-bore Upgrade Kit p/n A5190-060

Accessory Kit

Your kit consists of:

2	4 amp, 250V fuse
1	12-inch piece stainless steel tubing (0.06 OD x 0.02 ID) (27.5 cm length)
2	nuts, 0.06 OD
2	ferrules
1	seal removal tool
2	piston seals
1	hex/ball wrench
1	4-connection cable
1	external function connector
1	20 mL (cc) priming syringe with Luer LOK® tip
1	Luer adapter
1	waste tube kit:
1	48-inch Teflon tubing, 0.031 ID (123 cm)
1	washer and finger-tight fitting
1	solvent bottle label
3	stainless steel tubing, 0.06 OD x 0.007 ID

Standard Maintenance Kit

The Standard Maintenance Kit contains the following:

4	inlet filter cartridges
1	inlet check valve
1	transducer check valve
2	sapphire pistons (0.125 inch-diameter)
1	barbed fitting (nylon)
1	piece piston flush tube (Tygon®), 0.0655 ID (approx. 76 cm long)
1	syringe (20 cc)
6	piston seals
2	piston flush seals
6	Kel-F seals
1	seal removal tool
1	seal insertion tool

Standard LC Fittings Kit

Your kit consists of:

1	storage/carrying case
1	120-inch piece Teflon tubing, 0.063-inch ID
2	adapter fittings (10-32M)
4	Rheodyne nuts/ferrules
4	Parker type nuts/ferrules
3	wrenches (sizes: 1/4" - 5/16", 3/8" - 7/16", 1/2" - 9/16")
1	1/8-inch tube adapter for gas regulator assorted tubing: stainless steel 0.020-inch ID stainless steel 0.010-inch ID

Fittings kits contain tubing and fittings commonly needed for LC systems. These fittings can be used for several different instruments. If you change your LC configuration or damage connections, the fittings provided in this kit should allow you to make changes or replacements quickly and easily. All fittings can be kept in the compartmentalized storage box.

Piston Flush Seal Kit (10 mL)

Your kit consists of:

1	piece tubing, 30-inch, Tygon (76 cm length)
1	barbed fitting (nylon)
2	piston flush seals
1	seal removal tool
1	seal insertion tool
1	syringe, 20 cc

Piston Seal Kit (30 mL)

Your kit consists of:

1	piston (with small O-ring, high pressure)
3	PEEK wash rings
1	large O-ring
1	Piston flush seal (low-pressure)

Solvent Inlet Tube Kit

Your kit consists of:

1	bottle cap
1	tubing, Teflon, 0.063 ID

Solvent Tube Extension Kit

Your kit consists of:

4	60-inch extension tubing, with washer and union (152 cm length)
1	tubing Teflon, 0.063 ID

Manual Injection Valve Bracket Kit

Your kit contains:

1	11-3/16 inch steel mounting rod
2	rod brackets
2	short column brackets
2	long column brackets
1	manual injector valve mount
4	short set screws (6-32 x 1/4-inch)
2	flat-head screws (8-32 x 3/8-inch)
2	screws (6-32 x 7/16-inch)
2	long set screws (6-32 x 5/8-inch)
1	Allen wrench (1/16-inch)
1	12 inches stainless steel tubing, 0.06 OD x 0.01 ID, 12 inches

If you purchased a Rheodyne valve, you also received the valve, accompanied by Rheodyne's documentation.

Installing the Holder onto the Valve

Refer to page 187 to install the valve onto the bracket:

1. Move the injector valve handle to the "LOAD" position. Using the Allen wrench supplied with your Rheodyne valve, loosen the two set screws and remove the injection valve handle.
2. Hold the valve mounting bracket so that the two set screw holes are on the left. Place the injector valve into the bracket from the rear. If your injector valve has a remote start cable attached to it, place the cable into the cut-out on the left side of the bracket. When aligned correctly the "V" made by the two flats of the valve shaft will point to the upper left-hand mounting hole.
3. Fasten the valve securely with the two flat-head (Phillips) screws.
4. Attach the handle to the valve by tightening the two set screws, making sure that each screw is positioned over a flat edge of the valve shaft.
5. Install a long (5/8-inch) set screw into each of the valve bracket's mounting holes.
6. Install the rod through the valve bracket so that the notched portion of the rod faces you, as you look at the front of the injection valve.

7. Slide the valve bracket so that it is within the top quarter of the rod. Tighten the two set screws, securing the valve bracket to the rod.
8. Brackets for long (22 cm - 25 cm) and short (3 cm - 10 cm) columns are provided. Choose the bracket size that matches the type of column you use. Both brackets can be mounted, if desired.
9. Install a short (1/4-inch) set screw into each column bracket you wish to use. Slide the brackets onto the rod for positioning. Temporarily tighten the set screws to hold the brackets in place (Figure C.1).
10. Remove the two top cover screws from the right side of your SpectraSYSTEM pump or detector. Install a rod bracket into the lower hole, using a 7/16-inch Phillips-head screw.
11. Rest the rod in the lower rod bracket (with the injector valve facing toward the front) and place the upper rod bracket on top of the rod. Attach the upper rod bracket to the pump or detector, using the other 7/16-inch screw.
12. Re-position the column brackets or manual injector valve as needed, and tighten the set screws.

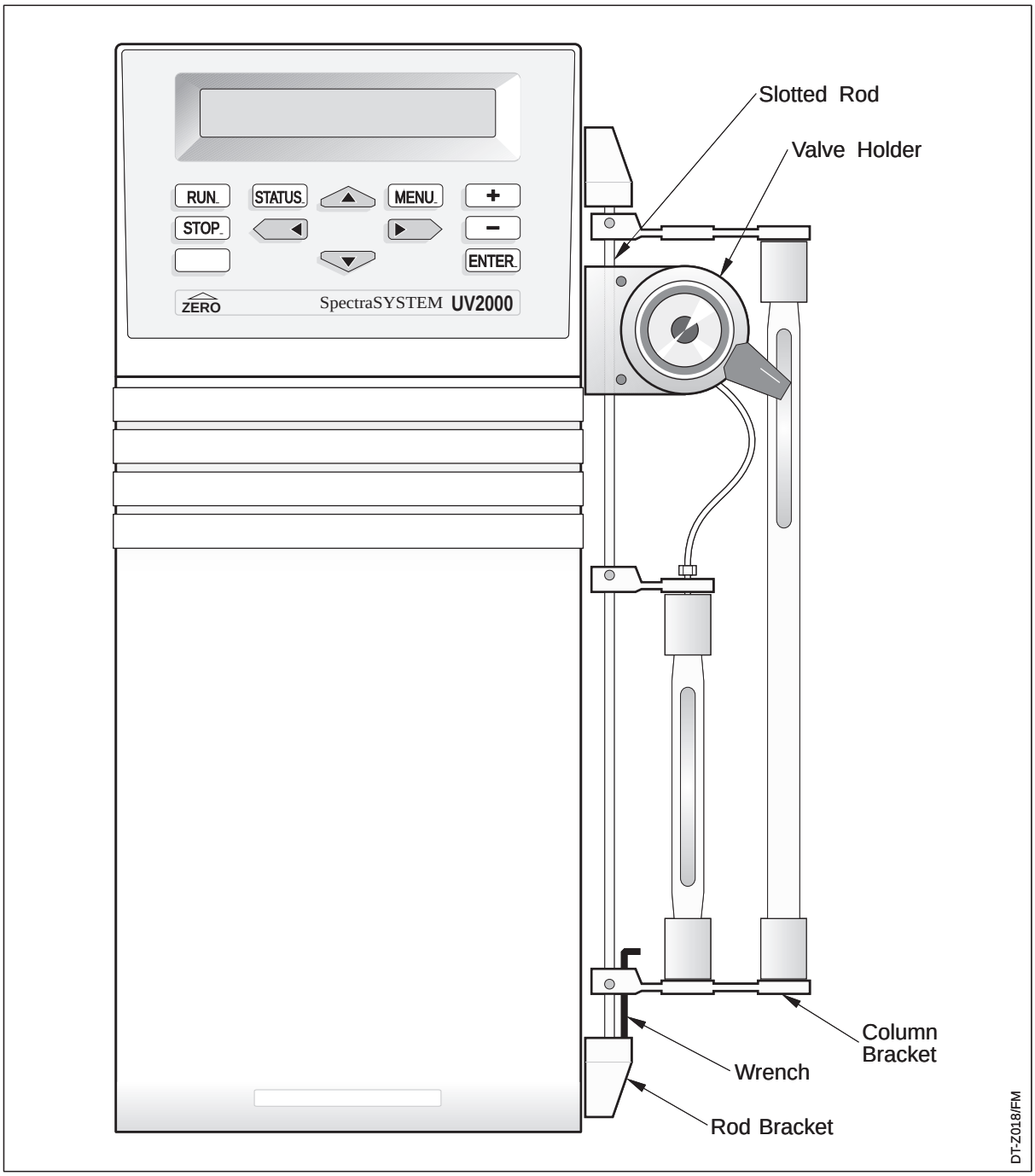


Figure C.1 Manual Injection Valve/Column Bracket mounted to a SpectraSYSTEM instrument

Narrow-bore Upgrade Kit

Your kit consists of:

- 0.01” ID bypass, crossover, and transducer stainless steel tubes
- 0.03” ID Teflon solvent inlet tube
- Low-volume outlet liquid-end assembly
- 3 µL static mixer
- 0.007” stainless steel tubing
- Nuts and ferrules for all tubes
- Instruction sheet

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