# Series 200 Vacuum Degasser User's Manual

#### **Release History**

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Any comments about the documentation for this product should be addressed to:

User Assistance PerkinElmer 710 Bridgeport Avenue Shelton, CT 06484-4794

Or emailed to: info@perkinelmer.com

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# Safety and Regulatory Information **S**

# Electromagnetic Compatibility (EMC)

#### **United States**

This product is classified as a digital device used exclusively as industrial, commercial, or medical test equipment. It is exempt from the technical standards specified in Part 15 of the FCC Rules and Regulations, based on Section 15.103 (c).

#### Europe

All information concerning EMC standards is in the Declaration of Conformity and these statements will change as the European Union adds new requirements.

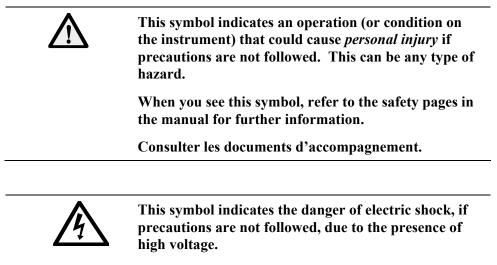
# Symbols and Conventions used in this Manual

In this manual the following graphic symbols and special text formats are used to set apart important safety information.

	A warning indicates an operation that can cause <i>personal injury</i> if precautions are not followed.	
	A caution indicates an operation that can cause <i>instrument damage</i> if precautions are not followed.	
Note	Notes emphasize significant information in a procedure or description.	

# Symbols used on the Instrument

There are two different types of warning symbols that appear on the instrument.



Attention. Risque de choc électrique.

# Warnings on the Instrument

The following warning labels are affixed to the side panel of the instrument for all models as shown in the figure below.

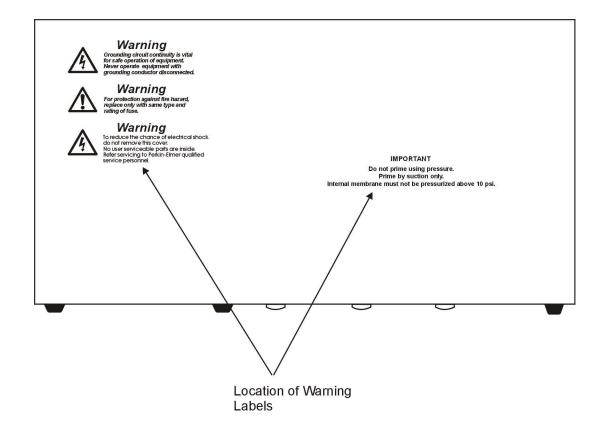
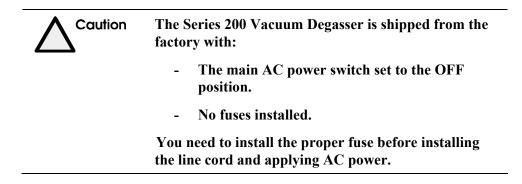


Figure S-1. Location of Warning Labels on the Series 200 Vacuum Degasser

Grounding circuit continuity is vital for safe operation of equipment. Never operate equipment with grounding conductor disconnected.
Débrancher le cordon d'alimentation avant d'ouvrir da continuité des masses est essentielle. Pour un fonctionnement sans danger. Ne jamais utiliser l'équipment si le fil de terre n'est pas raccordé.
For protection against fire hazard, replace only with same type and rating of fuse.
Afin d'assurer la protection contre les risques d'incendie, remplacer uniquement par un fusible de même type et de même courant nominal.
To reduce the chance of electric shock, do not remove this cover. No user-serviceable parts are inside. Refer servicing to PerkinElmer qualified service personnel.
Avertissement. Pour réduire le risque de chocs électriques, ne pas ouvrir les couvercles si un outil est nécessaire. Ne contient aucune pièce pouvant être réparée par l'utilisateur. Confier le dépannage au personnel qualifié de PerkinElmer.

IMPORTANT Do not prime using pressure. Prime by suction only. Internal membrane must not be pressurized above 10 psi. The following warning label covers the fuse receptacle located on the rear panel of the instrument:



Additional graphic symbols used on the instrument are the following:

	Indicates alternating current.
	Indicates the primary protective grounding terminal
$\bigcirc$	Indicates the <i>off</i> position of the main power switch.
	Indicates the <i>on</i> position of the main power switch.

# **Electrical Warnings**



Connect the instrument to an AC line power outlet that has a protective ground connection. To ensure satisfactory and safe operation of the instrument, it is essential that the protective ground conductor (the green/yellow lead) of the line power cord be connected to true electrical ground. Any interruption of the protective ground conductor, inside or outside the instrument, or disconnection of the protective ground terminal may impair the protection provided by the instrument.



Do not operate the instrument with any covers or parts removed.



Do not attempt to make adjustments, replacements or repairs to this instrument except as described in the accompanying User Documentation. Only a PerkinElmer service representative or similarly trained and authorized person should be permitted to service the instrument.



Use only fuses with the required current rating and of the specified type for replacement.

# **Quality Control/Good Laboratory Practices**

#### **Quality Control**

The user should develop appropriate quality control procedures for the LC Degasser (and the entire LC system) to ensure suitability for its intended use. These procedures typically consist of periodic performance verifications and routine inspections and suitability tests.

#### **Certificate of System Control**

Each Series 200 Vacuum Degasser is carefully built and tested in a controlled system in accordance with the requirements specified in its applicable PerkinElmer Final Assembly and Test Specification.

Each instrument is certified to meet its functional and performance specification upon release to shipment. The integrity of this quality system is routinely audited and is certified by a registrar.

#### Instrument Performance Verification (IPV)

To maintain functional performance, PerkinElmer recommends a yearly Instrument Performance Verification (IPV) of the Series 200 Vacuum Degasser by a PerkinElmer Service Engineer to ensure its operation within published specifications. These tests consist of measuring the most important Degasser characteristics such as retention time repeatability. Certification is available for regulatory compliance. Contact your local PerkinElmer Sales and Service office.

#### **Routine Inspection and Suitability Test**

The Series 200 Vacuum Degasser housing should also be inspected weekly through the ports for each Channel (A through D) and all 1/4-28 connectors for signs of liquid leaks. Prior to any sample analysis, a system suitability test, which closely resembles the intended assay, should be performed to ensure that the LC system is operating within established criteria (e.g., peak resolution, peak asymmetry, precision, retention time, column plate count, pressure limits, signal/noise ratio, etc.)

While the Universal Test Mix (UTM) Part No. 0089-0893, can be used for a system check, we recommend that you develop a separate system suitability test and acceptance criteria for each of your assays.

# **Hazardous Chemicals**

Before using mobile phase solvents, you should be thoroughly familiar with all hazards and safe handling practices. Observe the manufacturer's recommendations for use, storage and disposal. These recommendations are normally provided in the material safety data sheets (MSDS) supplied with the solvents.

Warning	Some chemicals used with this instrument may be hazardous or may become hazardous after completion of an analysis. The responsible body (e.g., Lab Manager) must take the necessary precautions to ensure that the surrounding workplace and that the instrument operators are not exposed to hazardous levels of toxic substances (chemical or biological) as defined in the applicable Material Safety Data Sheets (MSDS) or OSHA, ACGIH, or COSHH documents. Venting for fumes and disposal of waste must be in accordance with all national, state and local health and safety regulations and laws.
	The degassing membrane in the Series 200 Vacuum Degasser is manufactured from Teflon AF <sup>®</sup> . As with older membranes manufactured from PTFE, Teflon AF <sup>®</sup> is inert to all solvents normally used in HPLC. However, Teflon AF <sup>®</sup> is soluble in perfluorinated solvents such as Fluorinert <sup>®</sup> FC-75 and FC-40. Use of such solvents in the Series 200 vacuum degasser will result in the destruction of the membrane and degasser.

#### **Definitions in Warning for Hazardous Chemicals**

**Responsible body**. "Individual or group responsible for the use and maintenance of equipment, and for ensuring that operators are adequately trained." [per IEC 1010-1, Amendment 2].

**Operator**. "Person operating equipment for its intended purpose." [per IEC 1010-1, Amendment 2].

**OSHA:**Occupational Safety and Health Administration (United States)

ACGIH: American Conference of Governmental Industrial Hygienists

**COSHH:** Control of Substances Hazardous to Health (United Kingdom)

# **Temperature, Humidity and Environment**

	This instrument is designed for indoor use only.
	Do not operate in a Cold Room or a refrigerated area. The Series 200 Vacuum Degasser operates most efficiently under the following conditions:
	<ul> <li>Ambient temperature is 10 to 35 °C</li> </ul>
	<ul> <li>Ambient relative humidity is 20 to 80% noncondensing</li> </ul>
	• Altitude is in the range of 0 to 2000 m.
Warning	This instrument is not designed for operation in an

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This instrument is not designed for operation in an explosive environment.

#### **Installation Category**

This instrument is able to withstand transient overvoltage according to Installation Category II as defined in IEC 1010-1.

#### **Pollution Degree**

This equipment will operate safely in environments that contain nonconductive foreign matter up to Pollution Degree 2 in IEC 1010-1.

#### **Storage Conditions**

The Series 200 Vacuum Degasser may be stored under the following conditions:

- Ambient temperature is -20 to +60 °C,
- Ambient relative humidity is 20 to 80% noncondensing,
- Altitude is in the range 0 to 12,000 m.

# Introduction '

#### About the Series 200 Vacuum Degasser

The Vacuum Degasser is a high-efficiency in-line module that removes dissolved gasses from HPLC solvents. Its unique design assures reliable continuous operation and the highest level of continuous performance available without the need for helium degassing. Up to five solvent lines may be degassed simultaneously by one unit. The extremely low internal volume of each Teflon AF<sup>®</sup> channel offers very quick equilibration and very short startup times compared with PTFE degassing channels which have the same degassing efficiency.

Inside the unit, the solvent flows through a short length of Teflon AF<sup>®</sup> tubing which is located in a vacuum chamber. Within this chamber a partial vacuum is maintained by a constantly running, low RPM vacuum pump. Dissolved gasses migrate across the tubing wall under a concentration gradient produced by the vacuum as the solvent flows within the coil. Gasses removed are expelled, and the chamber is maintained at a constant, preset vacuum level by varying the vacuum pump speed as needed.

A special port in the vacuum pump continually flushes the pump head with a small "bleed" of air to remove any solvent vapors which may enter the pump from the vacuum chamber. This air bleed eliminates the need for any solenoid valves within the system. This patented\* design results in zero vacuum "hysteresis". Previous designs allowed the vacuum chamber pressure to fluctuate, with the pump cycling on and then off in response to the vacuum level.

<sup>\*</sup>This product is protected under U.S. patents 6,494,938; 6,248,157; 5,340,384 and 5,006,382. Other patents pending.

ZHCR® is a registered trademark of Systec, Inc., New Brighton, Minnesota.

Teflon AF<sup>®</sup> is a registered trademark of E.I. du Pont de Nemours and Company.

#### About the Controls and Indicators

#### **Front Panel Controls and Indicators**

A series of three LED's is located on the front of the instrument above the solvent inlets and outlets. These three LED's are used by the microprocessor to indicate the system status.

Immediately upon turning on the instrument, all 3 LED's will momentarily flash on then turn off. This confirms that the microprocessor has examined the vacuum sensor and found that it is within the expected range of 0 to 810 mm Hg absolute pressure.

Following the startup test, the microprocessor ramps the vacuum pump to high RPM, to quickly exhaust atmosphere from the vacuum chamber. The left-hand LED illuminates as the pump can be heard to run at high RPM. As vacuum is established in the chamber, the middle yellow LED illuminates, indicating the vacuum within the chamber has reached a value of less than 80 mm Hg absolute. As the vacuum pump continues to remove air within the vacuum chamber, the right-hand green LED will illuminate and the middle yellow LED extinguish, indicating that the vacuum chamber has reached a vacuum of 54 mm Hg absolute, where the performance of the degasser meets its specified level. When the vacuum level passes through 47 mm Hg absolute, the microprocessor ramps the pump speed to the low RPM, continuous run mode. At this low RPM, the vacuum will continue to fall to a constant value, wherein the fluctuation in the vacuum is less than 1mm Hg. This "zero hysteresis constant run" (ZHCR) mode is necessary, due to the extremely low mass, high response, Teflon AF<sup>®</sup> degassing channel. The effect of vacuum changes within the vacuum chamber can easily be seen in the UV absorbance of methanol (for example) at 215 nM. Only a ZHCR design ensures a baseline which is unaffected by the degasser.

An additional benefit of the constantly running pump is that a pressure rise in the vacuum chamber can be observed. This "smart leak detection" is a benefit of the patent-pending design of the degasser, and is best described as follows: Due to the fixed rate at which the pump runs, the gasses crossing the membrane pass from the chamber at a fixed rate as well. If a leak occurs within the chamber, the microprocessor will see this as a rise in pressure related to the vapor pressure of any leaking solvent. If the pressure within the chamber rises above 54 mm Hg absolute, the yellow LED will once again illuminate, indicating a possible leak condition within the chamber. If the pressure rises above 80 mm Hg, the system will shut down and go into a "safe" mode.

#### **Front Panel Connections**

Depending upon the model, there are from 1 to 5 degassing channels. Pairs of female ¼-28 connectors are located on the front of the Vacuum Degasser cabinet. These are the input and output ports for running up to 5 solvent lines through the Vacuum Degasser. Each channel has an input port and an output port on the same level, labeled on the front panel as "A" through "E". Flow direction is not critical. Plugs are provided to seal the ports of unused channels.

#### **Front Panel Indicators**

Three LEDs are located on the front of the instrument above the solvent inlets and outlets:

#### **POWER (Green)**

Indicates when power is applied to the Vacuum Degasser (plugged in and Power switch ON).

#### STATUS (Yellow)

Indicates when vacuum level is outside acceptable operating range. Normally it will come on at initial power-up and remain on during pump-down. It will go off in a few minutes when the vacuum level goes below 100 mm of Hg absolute. If an error condition occurs, this LED will flash in one of two modes:

- Flashing on and off in even 1-second intervals: pump was not able to reach vacuum set point, indicating a possible leak in the system.
- Flashing on for 1 second and off for 2 seconds indicates a vacuum signal error.

#### VACUUM (Green)

Indicates when vacuum level is within acceptable operating range. Normally it will come on after the initial pump-down, and remain on as long as the Vacuum Degasser is powered up and vacuum level is below 100 mm of Hg absolute.

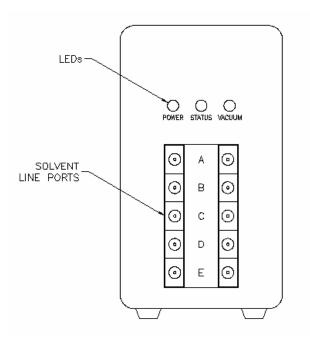


Figure 1. Front view of 5-Channel Vacuum Degasser

#### **Rear Panel Connections and Indicators**

#### **Exhaust Port**

The gas pumped out of the vacuum chamber leaves the unit through the exhaust port.

#### **Power Receptacle**

The power receptacle accepts the DC plug of the supplied AC Adapter. See Specifications section for further power requirement information.

#### **Power Switch**

This On/Off rocker type switch applies line power to the Vacuum Degasser. The rocker indicators are as follows: "O" = Off and "|" = On.

#### Validation Connector (Optional)

Depending on the model, there may be a 2-pin receptacle labeled "Validation" located next to the power switch. This receptacle and its mating screw-lock plug allow a validation signal from the Vacuum Degasser's control circuit to be sent to a computer or data system. This validation output indicates vacuum level (see Specifications section for details).

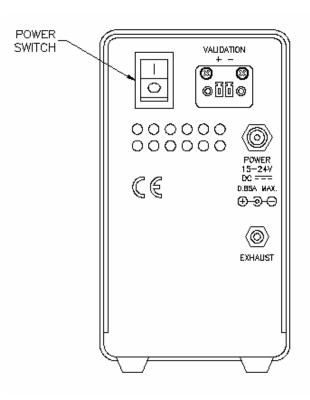


Figure 2. Rear view of Vacuum Degasser

# Scope of the Manual

This manual contains information about how to install, use, and troubleshoot the vacuum degasser. This manual is written for a person who has developed a working knowledge of liquid chromatography and understands:

• The function of each major component in a liquid chromatography system.

- How to select a column based on the analysis you wish to perform. •
- The principles of solvent selection and sample preparation. .

If you require an introduction to these concepts, the following book is an excellent reference: Practical Liquid Chromatography -- An Introduction by R. W. Yost, L. S. Ettre, and R. D. Conlon, published by The Perkin-Elmer Corporation (1980), Part No. 0993-9656.

## Warranty Exclusions and Limitations

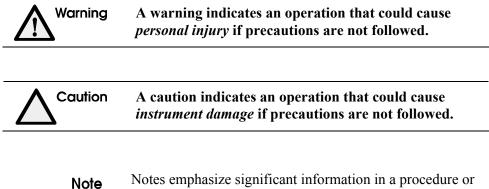
The Limited Warranty set forth on the PerkinElmer ORDERING INFORMATION form applies to the Series 200 Vacuum Degasser. Among those items excluded from the warranty under Part (B) "EXCLUSIONS AND LIMITATIONS" are:

- Fuses •
- Nuts •
- Ferrules

The items listed above are considered to be routinely replaced as part of the normal wear of the instrument.

## **Conventions**

The following conventions are used in this manual:



Notes emphasize significant information in a procedure or description.

# Warnings and Precautions

Read and understand the following warnings and precautions.

#### **Solvents**

Warning	Give careful attention to the hazards associated with the solvents you are using. Refer to the safety data sheets provided by the manufacturer. For example, Material Safety Data Sheets (MSDS) in the USA.
Warning	Do not use the Series 200 Vacuum Degasser to degas perfluorinated solvents. Teflon AF® is soluble in solvents such as the Fluorinert series from 3M company and PF series from Ausimont. Where there is a question about using a particular solvent that is fluorinated, contact a PerkinElmer representative

- Wear appropriate eye protection at all times when handling chemicals. Use safety glasses with side shields, goggles, or full-face shields, according to the types of chemicals you will be handling.
- Wear suitable protective clothing, including gloves that are specifically resistant to the chemicals being handled.
- Always use clean solvents. Solvents that have been distilled in glass (HPLC Grade) are highly recommended.
- Filter the solvents through a 0.5-micron medium as an additional precaution.
- Carefully use and store flammable solvents, which may form hazardous byproducts when the instrument is shut down, by following the recommended shutdown procedure.
- Check compatibility of solvent(s) with the type of column(s) being used.
- Know the relative polarity and miscibility of the solvents being used.

#### **Solvents with Low Boiling Points**

Do not use liquids that have a boiling point less than or equal to 30 °C (for example, pentane and  $CH_2CL_2$ ). When it is necessary to use these types of solvents, best results can be obtained using pressurization in a solvent chamber, which reduces the incidence of bubble formation in the pump inlet.

#### **Buffers**

Exercise care when using buffers in conjunction with organic solvents. NEVER LEAVE BUFFERS IN THE SYSTEM OVERNIGHT. Buffers left in the system can form salt crystals, which may cause premature pump seal failure, interfere with proper check valve operation, and plug the connecting tubing or the detector flowcell.

To remove buffers, flush the system first with water, then with methanol or isopropanol. Change the mobile phase from methanol or isopropanol to water before using buffers.

#### Corrosion

All parts that contact the mobile phase are made of PEEK, Kel-F<sup>®</sup>, Tefzel<sup>®</sup> or Teflon AF<sup>®</sup>. PEEK is sensitive to Sulfuric acid and certain solvents. If you have questions about your mobile phase, contact a PerkinElmer representative.

#### -1. Solvents Which May Corrode the Series 200 Vacuum Degasser

Aqua Regia (80% HCL, 20% HNO <sub>3</sub> )	Chloride salt solutions*
Chlorinated solvents	Halogenated solvents
Sulfuric Acid (Conc.)	

\* Titanium is more resistant to chloride salt solutions.

#### **Air Bubbles**

To prevent air from entering the system, and to ensure that pressure fluctuations do not occur, observe the following precautions:

- Ensure that the fittings are tightened on the degasser input and output ports.
- Ensure that the pump's solvent inlet filter is below the solvent level in the solvent reservoir.
- If the pump has not been used for an extended period of time, remove air bubbles by connecting a priming syringe to the drain valve on the pump, opening the drain valve, and fast-flushing the system to prime the pump. After priming the pump, close the drain valve completely.

#### **Pressure Buildup**

Over time you may notice a gradual increase in the system operating pressure. If you observe pressure readings greater than 3.45 MPa (500 psi) above the normal operating pressure of your analysis, check the following items:

- If you are injecting "clean" samples and there is no other apparent cause for the pressure buildup then remove, disassemble and clean the injector.
- If you are injecting "dirty" samples, the injector may be clogged or the column packing material may have retained contaminated particulates. Remove and clean the injector, and replace the column.
- The column end fitting or column frit may be plugged. Replace the end fitting or frit to relieve excess pressure. (Refer to the instructions supplied with the column.)
- In-line filters may be plugged. Replace the filter element.
- With the pump connected directly to the detector input, if you still experience excessive pressure, the problem could be a plugged flowcell. You can flush, reverse-flush, or rebuild the flowcell.

### **Specifications**

Specification	Description
Dimensions:	Height: 5.0 in. Width: 2.87 in. Depth: 9.81 in.
Weight	6 lb.
Channels	1-5 independent
Degassing Process	Gas permeation through a fluoropolymer membrane
Maximum Recommended Flow Rate <sup>1</sup>	3.0 mL/min.
Pressure Drop <sup>2</sup>	1.37 mm Hg/mL/min.
Degassing Capacity	~25% dissolved gases remaining in 60:40 MeOH/Water mixture at 1 mL/min.
Dead Volume	~480 microliters per channel for standard channel
Materials contacting solvents	PEEK, Glass-filled PTFE, Teflon AF <sup>®</sup>
Power:	
Power requirement if using supplied AC Adapter	100 to 240 VAC (±10%), 1A, 50 to 60 Hz (±3 Hz)
Power Requirement if <u>not</u> using supplied AC Adapter	15 to 24 VDC at 0.85 A maximum (0.5 A typical)
Wall Sockets	4 supplied with AC Adapter, interchangeable: North America/Japan, U.K., Continental Europe, Australia
Installation Over-Voltage Category	II
Validation Output:	
Signal	5 mVDC / 1 mm Hg absolute from 20 to 800 mm Hg (0.100 VDC at 20 mm Hg; 4.000 VDC at 800 mm Hg)
Accuracy	$\pm 1.0\%$ of reading $\pm 0.010$ VDC from 20 to 800 mm Hg
Operating Conditions:	
Ambient Temperature	10 to 35 °C
Ambient Relative Humidity (RH)	20 to 80 % RH (without condensation)
Altitude	0 to 2000 Meters
Indoor vs. Outdoor Use	Indoor
Pollution Degree	2
Storage Conditions:	
Ambient temperature	-20 to +60 °C
Ambient Relative Humidity	20 to 80% RH (without condensation)
Altitude	0 to 12000 M

#### Table 1-2. Specifications of the Series 200 Vacuum Degasser

1 Maximum recommended flow rate to prevent a 60:40 MeOH/Water mixture from outgassing. The estimate assumes low pressure mixing and low flow restriction prior to the HPLC pump. MeOH/Water mixing represents the worst outgassing case and maximum flow rate will likely increase with Acetonitrile/Water mixtures. High pressure mixing will also increase the maximum flow rate. Degassing is still recommended.

2 Calculated tubing pressure per unit change in flow assuming laminar flow with a viscosity of 1.0 cP. Inlet and outlet bulkheads may contribute to the overall pressure, but are not included in the estimate.

# Installation **2**

This chapter describes how to install your Series 200 Vacuum Degasser. The following installation topics are covered in this chapter:

- Preparing your laboratory
- Unpacking your vacuum Degasser
- Electrical requirements and settings
- Connecting the tubing
- System connections

# **Preparing Your Laboratory**

Before installing your Series 200 Vacuum Degasser, prepare your laboratory according to the following guidelines.

#### **Required Air Quality**

To minimize contamination problems in your laboratory, provide a relatively dust-free environment. Make sure that the following gases or vapors are not present at levels that exceed federal, state, and local ordinances for continuous human exposure:

- Flammable Caustic
- Explosive
- Corrosive
- Toxic

Make sure that your laboratory environment consists of the following temperature and humidity levels:

• Ambient temperature between 10 and 35 °C

• Constant relative humidity between 20 and 80%, without condensation Use care when working with hazardous solvents, or solvents that produce hazardous by-products.



Solvent vapor levels that are high enough to interfere with the detector performance should be considered hazardous to someone who is continuously exposed to the vapors.

#### Solvents/Mobile Phase

Use only HPLC grade solvents in all analyses. HPLC grade water and methanol (1 liter each) are required for performance verification.

The degassing membrane in the Series 200 Vacuum Degasser is manufactured from Teflon AF <sup>®</sup> . As with older membranes manufactured from PTFE, Teflon AF <sup>®</sup> is inert to all solvents normally used in HPLC. However, Teflon AF <sup>®</sup> is soluble in perfluorinated solvents such as Fluorinert <sup>®</sup> FC-75 and FC-40 and Fomblin perfluoro polyether solvents from Ausimont. Use of such solvents in
the Series 200 vacuum Degasser will result in the dissolution and hence destruction of the membrane.

#### Corrosion

All parts that contact the mobile phase are made of PEEK, Glass-filled PTFE or Teflon AF<sup>®</sup>. PEEK is sensitive to Sulfuric acid and certain solvents.

#### **Space Requirements**

The Series 200 Vacuum Degasser is designed to sit on a bench top, and is plumbed into the LC system between the solvent supply and pump. A space 5 <sup>1</sup>/<sub>4</sub>-in high and 3-in wide is sufficient. The case is 10-in deep (front to back), but additional space is required both in front, to accommodate the tubing connected to the unit, and behind to accommodate the power cord.

# **Unpacking Your Series 200 Vacuum Degasser**

Carefully unpack the Series 200 Vacuum Degasser and check for obvious signs of damage that may have occurred during shipment. Immediately report any damaged or missing items to the shipping carrier and PerkinElmer.

A Start-Up Kit is supplied with the Series 200 Vacuum Degasser. Use Table 2-1 to inventory the Start-Up Kit parts.

ltem	Quantity
AC Adapter (including cord)	1
Interchangeable Wall Plugs	4 (North America/Japan, U.K., Continental Europe, Australia)
Operator's Manual	1
Plug, <sup>1</sup> / <sub>4</sub> -28, Rheodyne 6118	2

Table 2-1. Start-Up Kit for the Series 200 Vacuum Degasser

# **Electrical Requirements**

The AC Adapter supplied with the Vacuum Degasser incorporates a universal AC input, switching regulator. This allows the instrument to operate at any AC line voltage from 100 to 240 VAC ( $\pm 10\%$ ) with a line frequency range of 47 to 63 Hz. The switching regulator senses the incoming line voltage and automatically adjusts its operation accordingly. The switching regulator assembly incorporates its own on-board line voltage fuse. This fuse is not user serviceable. In the event that this fuse blows, it will be necessary to replace the AC Adapter.

A set of four interchangeable wall plugs is included to allow the AC adapter to be plugged into the standard electrical sockets in North America, Japan, the U.K., most countries in continental Europe, and Australia.

It is recommended that the AC Adapter be used to supply power to the Vacuum Degasser. If another means is used to supply power to the rear panel power jack, the voltage must be within the range 15 to 24 VDC at 0.85 A maximum (0.5 A typical). The rear panel power jack will accept a 2.1 mm female plug. Correct polarity must be observed. The center connection of the plug must be positive, and the outside must be negative. See figure below.

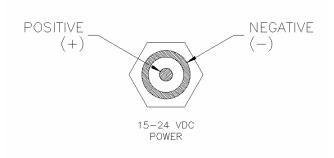


Figure 3. Rear Panel Power Connector Polarity

#### Installing the AC Adapter with Power Cord

Locate the AC Adapter with power cord and the set of four interchangeable wall plugs. Plug the round connector at the other end of the AC Adapter's cord into the Power jack on the Vacuum Degasser rear panel. From the set of four plugs, select the one appropriate for the local electrical socket and install it onto the AC adapter. With the power switch off, insert plug into the AC supply. The AC adapter should be positioned for easy disconnection.

# **Connecting the Tubing**

Solvent lines to be degassed are connected to the Vacuum Degasser's front panel ports, as detailed below. Unused ports must be plugged to enable the degasser to operate at its peak level of performance.

To make a tubing connection:

- 1. Run a line of 1/8" O.D. x 1/16" I.D. Teflon chromatography tubing from the solvent supply to the Vacuum Degasser.
- 2. Push the tubing through a PEEK 1/8" male <sup>1</sup>/<sub>4</sub>-28 fitting and slide a ferrule over the tubing end (see figure below). Cut the Teflon tubing so the end is flat.
- 3. Screw the <sup>1</sup>/<sub>4</sub>-28 fitting into one port on the front of the Vacuum Degasser (Channel A, for example). The direction of flow through the Vacuum Degasser is not critical. Plastic connectors should be tightened by hand. Overtightening them will damage the threads.
- 4. Repeat steps 1 through 3 to connect additional lines to be degassed.
- 5. Once all desired solvent lines have been connected to the Vacuum Degasser, any and all unused ports should be plugged. Use the plugs supplied. Press in by hand.

6. Prime each degassing membrane by pulling the solvent from the reservoir through the degassing system. This can be done by connecting a syringe to the tubing or LC pump priming port and drawing air and/or mobile phase into the syringe until no air remains in the tubing, approximately 5 milliliters.

DO NOT prime the membranes by pushing solvent through the degassing systems. This technique can generate several hundred pounds of pressure which might rupture the membrane, even though the Teflon AF <sup>®</sup> membrane is quite rugged. The maximum recommended pressure on the membrane is 1 mPa (100 psig, 7 Bar).

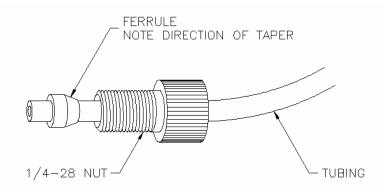


Figure 4. Configuration of 1/4-28 Nut, Ferrule and Tubing

### Connecting the Vacuum Degasser in a Typical System

The following illustration shows the tubing connections that are typically made between the Vacuum Degasser and other instruments in an LC system. The direction of flow through the Vacuum Degasser is not critical.

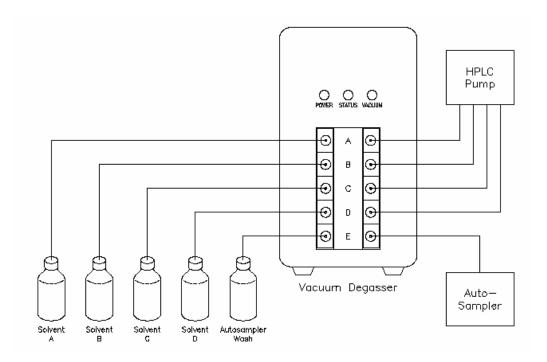


Figure 5. System Tubing Connections

#### Powering Up the Series 200 Vacuum Degasser

1. With the Vacuum Degasser plumbed into the system and the power cord installed, as described in the above section, flip on the rear panel power switch. The green Power LED should illuminate.

Immediately upon turning on the instrument, the microprocessor examines the vacuum sensor signal to confirm that it is within an expected range. Following the startup test, the microprocessor ramps the vacuum pump to high RPM, to quickly exhaust atmosphere from the vacuum chamber. As the vacuum level approaches the preset control value, the pump RPM will slowly ramp down to a low speed (typically 40 to 60 RPM) and will vary slightly as needed under the changing degassing load to maintain a virtually constant vacuum level.

During initial pump-down, the yellow Status LED will be lit. Once the vacuum has reached normal operating level, the yellow LED will extinguish and the green Vacuum LED will illuminate. If you want to confirm that the pump is running, beyond the front panel LEDs, the slight vibration caused by the microstepping of the motor driving the vacuum pump may be felt by placing your hand on the instrument.

# 2. Start solvent flow through the system and check for leaks around the 1/4-28 connectors.

If a leak occurs at the connection, tighten the fitting an additional 1/8 turn. If the leak persists, disconnect the leaking fitting and inspect it. If the nut and ferrule

appear to be in good condition, reconnect the fitting. If the leak persists, replace the nut and ferrule and repeat the procedure until you achieve leak-free operation.

The Vacuum Degasser maintains a constant vacuum pressure of 50 mm Hg absolute (nominal) by varying the speed of the vacuum pump as needed depending on the degassing load in the system. The pump is designed for at least 5 years of constant running and has integral in-pump venting, which eliminates the need for stop-start running (U.S. Patent 6,248,157). The vacuum level and pump speed is constantly monitored by the microprocessor for changes in operating conditions which might be attributed to chamber internal leaks. If a potential leak is detected, the pump will be shut down and the yellow Status LED will flash. The vacuum is maintained as long as the Vacuum Degasser is powered on. Solvent flowing through the Vacuum Degasser will continue to be degassed so long as the instrument is on and running.

- 3. Turn off the Vacuum Degasser when the LC to which it is connected is not in use. The vacuum chamber(s) will slowly return to atmospheric pressure when the unit is powered off. This is accomplished by a small, in-line vacuum bleed and reduces the possibility of solvent vapors condensing in the vacuum tubing or pump head.
- 4. When flushing a line of solvent, the single lumen coil inside the chamber contains a very small amount of solvent (approximately 480 microliters). When changing from one solvent to another where the final solvent is immiscible with the first, use an intermediate solvent miscible with both the initial and final solvent. Carryover from solvent to solvent is much less than previous PTFE designs. Once air bubbles have been cleared from the solvent line, any further bubbles observed will be coming from the solvent reservoir or from a leaking fitting.
  - Since there is virtually no solvent retained within the Note Vacuum Degasser (~480 microliters per channel), priming the system is relatively simple. Using the Prime mode on the LC system pump, allow the pump to draw each solvent to be used in the analysis at a flow rate of 2 mL/min. for 1-2 minutes. This ensures that the line from the degasser channel being primed through the proportioning valve on the pump has freshly degassed solvent. This dynamic priming method will allow an immediate startup of the analysis upon column equilibration. Contrary to previous PTFE-based degassers, the new Vacuum Degasser, which uses Teflon  $AF^{\mathbb{R}}$  membranes, fully degasses solvents within the time it takes for the volume to pass through the chamber, and yet degasses the solvents as thoroughly as, or better than, PTFE channels containing 40 times more solvent.

# Using the Series 200 Vacuum Degasser

This chapter describes how to use the Series 200 Vacuum Degasser.

Before beginning, make a final check of all tubing connections to and from the Series 200 Vacuum Degasser.

### **Principles of Operation**

The Series 200 Vacuum Degasser consists of a vacuum chamber, degassing tube, variable speed vacuum pump, microprocessor controller, sensor, and check valves. The solvent (mobile phase) flows into a degassing tube, which is inside a vacuum chamber. Decreased pressure in the chamber causes the outward movement of gas dissolved in the mobile phase across the tube wall, in accordance to Henry's Law, thus degassing the mobile phase. The pressure in the vacuum chamber is established by the vacuum pump and monitored by the microprocessor through an integrated absolute pressure sensor. Degassed mobile phase exits the vacuum degasser and enters the pump.

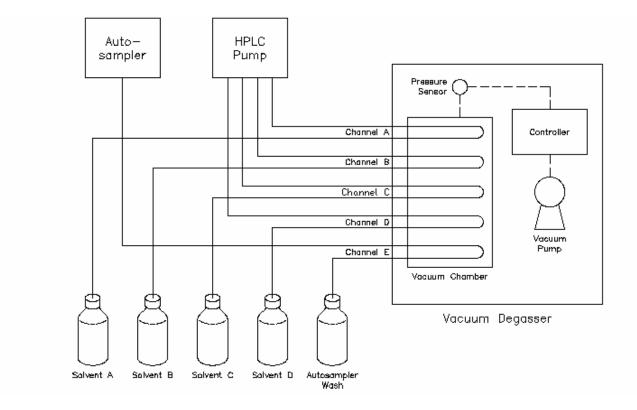


Figure 3-1. Schematic of the Series 200 Vacuum Degasser

#### How the Series 200 Vacuum Degasser Operates

Immediately upon turning on the instrument, the microprocessor examines the vacuum sensor signal to confirm that it is within an expected range. Following the startup test, the microprocessor ramps the vacuum pump to high RPM, to quickly exhaust atmosphere from the vacuum chamber. As the vacuum level approaches the preset control value, the pump RPM will slowly ramp down to a low speed (typically 40 to 60 RPM). Afterwards, the pump RPM will vary slightly, as needed under the changing degassing load, to maintain a virtually constant vacuum level ( $50 \pm 0.5 \text{ mm Hg}$ ). This "zero hysteresis, constant run" (ZHCR<sup>®</sup>) mode is necessary, due to the extremely low mass, high response, Teflon AF<sup>®</sup> degassing tubing. Only a ZHCR<sup>®</sup> design ensures a baseline which is unaffected by the degasser.

#### **Smart Leak Detection**

An additional benefit of maintaining a constant vacuum level is that a potential leak in the vacuum degassing system can be observed by monitoring the RPM of the pump. This "smart leak detection" is a benefit of the patented design of the degasser. If a leak occurs within the chamber, the microprocessor will increase the pump RPM in an attempt to maintain the vacuum level. If the pump cannot maintain the vacuum level (if it runs at an elevated RPM for more than 2 minutes), the yellow LED will flash, indicating a possible leak condition, and the system will shut down and go into a "safe" mode.

#### Principles of degassing using Teflon AF<sup>®</sup> membranes

This relatively recent addition to the field of degassing has properties not found in other fluoropolymers. The fully amorphous nature of this fluoropolymer and its molecular structure creates a molecular level porosity unlike the mechanically induced porosity in PTFE extruded tubing. In addition, unlike the process used in extruding PTFE, no extrusion agents are needed (like kerosene, etc.) which contaminate mobile phases until they are extracted by the mobile phase over time. Likewise, this molecular structure, combined with the very small surface areas required to degas the mobile phase, reduces the possibility of carryover from one solvent or mobile phase to another to virtually zero.

Teflon  $AF^{\text{(B)}}$  is so non-polar that it is both solvophobic and hydrophobic. This feature of Teflon  $AF^{\text{(B)}}$  reduces the possibility of cross-channel contamination from one channel to another, and when combined with the ultra-low internal volumes of Teflon  $AF^{\text{(B)}}$  channels needed for HPLC flow rates, all but eliminates this cross contamination concern by the chromatographer. Teflon  $AF^{\text{(B)}}$  has been used in certain optical systems associated with HPLC for a few years without concern for normal HPLC solvents. However, Teflon

 $AF^{\mathbb{R}}$  is soluble in certain solvents (see cautionary statements) and must not be used to degas these types of solvents.

Teflon AF<sup>®</sup> is permeable to some degree to water vapor whereas PTFE is not. While the vacuum pump in the Vacuum Degasser contains internal provisions for sweeping water or solvent vapor from the pump continuously, it is possible that over time, high concentration buffers may form crystals within the channel due to the loss of water within the channel. The same precautions should be taken to prevent crystallization within these channels as are taken for the HPLC pump. See the "Short-term Shutdown" procedures.

# **Operating Summary**

The following steps summarize how to use the vacuum degasser:

- 1. Select and fill each solvent reservoir with the mobile phase for your analysis.
- 2. Verify that the Series 200 Vacuum Degasser is properly installed as described in Chapter 2, "Installation."



#### Never connect the Series 200 Vacuum Degasser to the output side of the pump. The high pressure may cause permanent damage to the degassing membrane.

- 3. Verify that the tubing to your injector, column, and detector is properly connected. Also verify that plugs are installed in the unused ports.
- 4. Disconnect and remove the tubing that is connected to the output port, connect the priming syringe to this port, and pull the solvent through the degasser until bubbles no longer appear. Then reconnect the tubing to the output port.
- 5. Switch on the degasser and start the pump at 1.0 mL/min. Allow the system to equilibrate for 5-10 minutes. The small volumes contained in the Series 200 Vacuum Degasser should only be considered in chromatograph equilibration time when flow rates less than 1 mL/min are used.

**Note** Use of this product outside the scope of this manual may present a hazard.

#### Extending the degassing flow rate range

Certain organic solvents used in reversed phase chromatography outgas upon mixing with water, if not properly degassed. These solvents are generally alcohols (e.g. methanol), acetonitrile and tetrahydrofuran. Passing water and methanol through a single channel is generally sufficient to degas these solvents so outgassing does not occur upon mixing when a 60:40 methanol/water mixture is generated by your Vacuum Degasser or pump at a flow rate of 3 mL/min. If outgassing does occur, or if a flow rate higher than 3 mL/min. is required, it is a general rule that only the organic portion of the mobile phase needs to be passed through a second degassing channel to ensure outgassing does not occur. This is due to the ability of all organic solvents (e.g. methanol) to hold at least 10 times more dissolved atmosphere than water can.

To more thoroughly degas a mobile phase, connect the outlet of the organic channel to the inlet a second channel and the outlet of the second channel to the pump. This places the two channels in series and doubles the degassing capacity for the organic portion of the mobile phase.

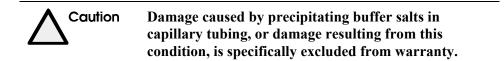
# Shutdown

There are two types of shutdown procedures: long-term and short-term.

#### Short-Term Shutdown (Overnight and Weekends)

Observe all precautions pertaining to hazardous solvents and/or those solvents that form harmful deposits or by-products.

- 1. Remove harmful mobile phases from the vacuum degasser and other instruments in the system.
- 2. Flush the column according to the instructions supplied with the column. Flush buffer salts from the system with water. Evaporation leaves salt crystals that may form harmful deposits. Remove chloroform or solvents that can decompose to form hydrochloric acid from the system.



- 3. After removing harmful mobile phases, prepare the detector for most mobile phases by flushing it with isopropanol. To avoid contaminating the system, refilter or discard solvents (including water) that were exposed to the environment for more than 24 hours before use.
- 4. For weekend storage we recommend flushing 60/40% MeOH/Water through the vacuum degasser, pump, column, and flowcell (provided your column is compatible with MeOH/Water). Then turn off the vacuum degasser, pump, and detector.

#### Long-Term Shutdown

- 1. Follow Short-Term Shutdown procedure Steps 1 and 2.
- 2. Remove the column and direct the pump output tubing to a beaker. Flush the vacuum degasser first with water and then with isopropanol.
- 3. Turn off the vacuum degasser. Then disconnect the tubing between the vacuum degasser and solvent reservoirs and the vacuum degasser and pump. Plug all of the ports on the vacuum degasser.
- 4. Store the vacuum degasser in a clean, dry location.
- 5. Before using the vacuum degasser, completely purge it with the correct solvent for the column before reconnecting the column and restarting the system.

# Maintenance 4

You can experience long and trouble-free performance from your Series 200 Vacuum Degasser by performing both routine and preventive maintenance procedures.

# **Preventive Maintenance**

Perform preventive maintenance to ensure that your Series 200 Vacuum Degasser will perform consistently at an optimal level. To maintain the Degasser in the best condition, the following measures are recommended:

- 1. Adhere to standard laboratory cleanliness practices.
  - Use only high-purity solvents (preferably HPLC Grade) for mobile phases. (Water should be bottled HPLC grade, or filtered and deionized tap water.)
  - Filter all solvents to prevent particulate contamination and tubing blockages.
- 2. Use only high-purity gases when drying contact areas.
  - Ensure that all new tubing (stainless steel) is passivated and thoroughly flushed before making pump connections. (The tubing available from PerkinElmer is passivated.)
  - Follow the short- and long-term shutdown procedures that are described in Chapter 3 of this manual.

# **Routine Maintenance**

Routine maintenance is defined as replacing the normal wear items when you notice degradation in performance.

If you have a problem, you can save time and money by referring to the Troubleshooting Guide (on the next page) before calling your local PerkinElmer Service Engineer. Your problem may be minor and you may be able to correct it yourself by using the Troubleshooting Guide to pinpoint the cause.



Never remove the Series 200 Vacuum Degasser cover. There is nothing inside the detector that requires customer maintenance. If you encounter a problem, call your local PerkinElmer Service Engineer for assistance.

# **Troubleshooting Guide**

Problem	Probable Cause	Solution
1) Power switch is ON but all 3 LED's are off, indicating no power to the degasser.	(a) Series 200 Vacuum Degasser AC adapter is not plugged into the AC outlet.	(a) Plug the AC adapter into the AC outlet.
	(b) Blown fuse.	(b) Replace the fuse.
2) Yellow Status LED is on steadily, pump is running and RPM seems high.	2) Pump is in initial pull-down phase or system's degassing demand has increased.	2) Typically normal operation, although if pump speed continues to rise for an extended period of time (as heard by the pitch of the stepper motor) it could indicate a potential fault condition.
3) Yellow Status LED is flashing approximately 1 second off, 1 second on. Vacuum pump is not running.	3) Possible system leak.	3) Contact your Service Representative.
4) Yellow Status LED is flashing approximately 2 seconds off, 1 second on. Vacuum pump is not running.	4) Possible sensor or Control Board fault.	4) Contact your Service Representative.
5) Is there a way to check whether the system is operating correctly when Power and Vacuum green LEDs are illuminated, but pump can't be heard running?	5) Due to the design of the pump and degasser, the pump is virtually silent at low RPM, even though vacuum is good and degassing is normal.	5a) Place a hand on the top of the unit. A slight vibration can be felt indicating the pump is operating at low RPM.
		5b) Monitor the UV absorbance of non-degassed methanol at 215 nM versus degassed methanol coming through the degasser. Proper performance of the degasser should decrease the UV absorbance of the methanol significantly.
6) Bubbles appear through the output tubing.	6) Loose fitting(s).	6) Tighten the input and output fittings.
7) No solvent flow.	7a) Air in the HPLC pump head.	7a) Prime/purge the pump head.
	7b) If a buffer solvent was left in the degasser for some time after use, it may plug the degasser elements.	7b) Use a different channel, or connect the channel to a beaker of the solvent without the buffer. Draw the solvent through the channel to dissolve the buffer. Do not push the solvent through the channel. If this flushing action does not work, contact your Service Representative.

# Series 200 Vacuum Degasser Performance

The following graph illustrates the performance of the Series 200 Vacuum Degasser.

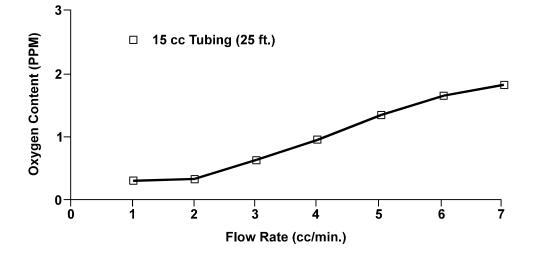


Figure 4-1. Graphical representation of the performance of the Series 200 Vacuum Degasser



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