

# Degassing in HPLC and Related Technologies

## Operator's Manual



Notices:

This system is covered by a limited warranty. A copy of the warranty is included with this manual. The analyst is required to perform routine maintenance as described herein on a periodic basis to keep the warranty in effect.

All information in this manual is subject to change without notice and does not represent a commitment on the part of the supplier. The supplier believes that the information in its user manuals is accurate as of the date of publication. In the event of a technical or typographical error, the supplier may revise its user manuals without notice. Please consult your supplier if you suspect that a user manual contains serious errors.

This manual may not be reproduced or transmitted in any form, electronic or mechanical, including photocopying, recording, storing in an information retrieval system, or translating, in whole or in part, without the prior written consent of the supplier.

ZHCR<sup>®</sup> is a registered trademark of Systec, Inc.

Teflon<sup>®</sup> and Teflon AF<sup>®</sup> are registered trademarks of E.I. du Pont de Nemours and Company

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

© 2007. All rights reserved.

Printed in Sweden



## Warnings and Safety Precautions

The Vacuum Degasser is designed to remove gases from a liquid such as the mobile phase used in HPLC. The following precautions should be followed to minimize the possibility of personal injury and/or damage to property while using the system.

**1) Maintain a well-ventilated laboratory.**

The mobile phase typically contains a volatile organic solvent. Ensure that the laboratory is well ventilated so that a build-up of vaporized solvent cannot occur.

**2) Avoid open flames and sparks.**

Do not use an open flame in the laboratory and do not install any equipment that can cause sparks in the same room as the instrument.

**3) The instrument must be plugged into a grounded power line.**

Ensure that all parts of the system are properly grounded. It is strongly recommended that all parts of the system are connected to a common ground.

**4) Treat the mobile phase as if it is capable of containing hazardous substances.**

The mobile phase may contain compounds which may present a health hazard. If you are analyzing biological/clinical samples, treat them in accordance with the infectious disease control program of your institution.

**5) DO NOT prime the system by pushing solvent through it.**

Priming the system by pushing solvent through it can generate several hundred pounds of pressure which might rupture the tubing, even though the Teflon AF<sup>®</sup> tubing is quite rugged. The maximum recommended pressure on the tubing is 1 mPa (100 psig, 7 Bar). Do not connect the vacuum degasser to the output side of the HPLC pump as the high pressure can cause permanent damage to the degasser.

# Table of Contents

<b>Warnings and Safety Precautions .....</b>	<b>3</b>
<b>1 Introduction .....</b>	<b>6</b>
1.1 Overview .....	6
1.2 Principle of Operation .....	6
1.3 The Benefits of Teflon AF <sup>®</sup> .....	7
1.4 Principle of Operation .....	8
1.5 External Components of the Degasser .....	9
1.5.1 The Front Panel .....	9
1.5.2 Rear Panel .....	10
<b>2 Installation .....</b>	<b>11</b>
2.1 Unpacking the Vacuum Degasser .....	11
2.2 Space Requirements .....	11
2.3 Electrical Power Requirements .....	12
2.4 Setup .....	13
2.4.1 Installing the AC Adapter .....	13
2.4.2 Connecting the Tubing .....	13
2.4.3 Extending the Degassing Flow Rate Range .....	14
2.5 Connecting the Vacuum Degasser in a Typical System .....	15
<b>3 Operation .....</b>	<b>16</b>
3.1 Operating Summary .....	16
3.2 Powering up the Vacuum Degasser .....	17
3.3 Using the Degasser .....	17
3.3.1 Operational Considerations .....	17
3.3.2 Priming the Degasser .....	18
3.3.3 Changing the Solvent .....	19
3.4 Shutdown .....	19
3.4.1 Short-term Shutdown (Overnight and Weekends) .....	19
3.4.2 Long-term Shutdown .....	20
<b>4 Maintenance and Troubleshooting .....</b>	<b>21</b>
4.1 Maintenance .....	21
4.1.1 Preventative Maintenance .....	21
4.1.2 Routine Maintenance .....	21
4.2 Troubleshooting Guide .....	22

<b>Appendix A Specifications of the Vacuum Degasser</b> .....	<b>23</b>
A.1 General .....	23
A.2 Power.....	23
A.3 Validation Output .....	23
A.4 Operating Conditions.....	24
A.5 Storage Conditions .....	24
A.6 Physical Characteristics.....	24
<b>Appendix B Regulatory Considerations</b> .....	<b>25</b>
B.1 CE Declaration of Conformity .....	25
B.2 Federal Communications Commissions (FCC) Notice (US only).....	26
B.3 Disposal of Electrical and Electronic Equipment.....	27
<b>Index</b> .....	<b>1</b>

# 1 Introduction

## 1.1 Overview

The Vacuum Degasser is a high-efficiency in-line system that is designed to remove dissolved gases from a solvent. The system is commonly used to degas the mobile phase for high performance liquid chromatography (HPLC) systems and can be employed in other applications where gases that are dissolved in the operating liquid may interfere with the use of the system (e.g. an autotitrator).

The degasser is easy to use, provides reliable continuous operation, and eliminates the need for helium sparging to remove gases. The extremely low internal volume of the Teflon AF<sup>®</sup> tubing used in the degasser provides for quick equilibration and very short startup times, compared to the use of a degasser which uses PTFE<sup>®</sup> degassing channels that has the same degassing efficiency.

## 1.2 Principle of Operation

The critical component of the vacuum degasser is a short length of Teflon AF<sup>®</sup> tubing through which the solvent flows. This tubing is located in a chamber in which a partial vacuum is maintained by a vacuum pump which is constantly running at a low speed. Dissolved gasses migrate across the tubing wall under a concentration gradient produced by the vacuum as the solvent flows within the tubing in accordance with Henry's law. The gases are expelled from the system 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".

### 1.3 The Benefits of Teflon AF<sup>®</sup>

The use of Teflon AF<sup>®</sup> for vacuum degassing of solvents is a relatively recent application of the polymer. The polymer has properties that are not found in other fluoropolymers which are especially well suited for solvent degassing, including:

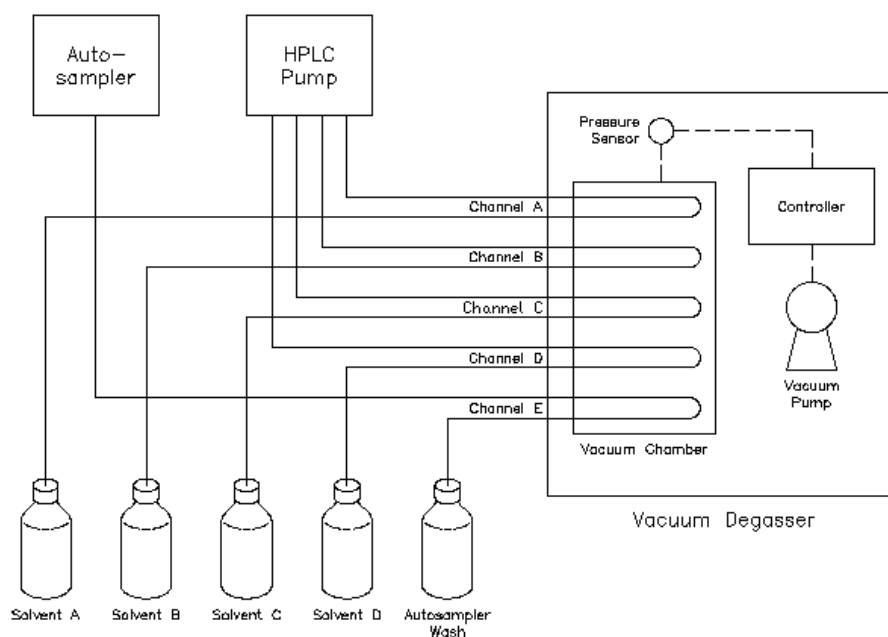
- The fully amorphous nature of this fluoropolymer and its molecular structure creates a molecular level porosity unlike the mechanically induced porosity in PTFE extruded tubing.
- The process used for extruding the polymer does not require an extrusion agent. In contrast, the extrusion of PTFE uses kerosene which will contaminate the mobile phase until it is totally extracted by the mobile phase over time.
- The molecular structure of the polymer, combined with the very small surface area required to degas the mobile phase, reduces the possibility of carryover from one solvent or mobile phase to another to virtually zero.
- Teflon AF<sup>®</sup> is so non-polar that it is both solvophobic and hydrophobic. This feature of Teflon AF<sup>®</sup> reduces the possibility of cross-channel contamination from one channel to another, and when combined with the ultra-low internal volumes of Teflon AF<sup>®</sup> channels needed for HPLC flow rates, all but eliminates the concern of cross contamination by the chromatographer.

**Note: Teflon AF<sup>®</sup> is soluble in certain perfluorinated solvents such as Fluorinert<sup>®</sup> FC-75 and FC-40 and Fomblin perfluoro polyether solvents from Ausimont. In addition, Freon<sup>®</sup> solvents will adversely affect Teflon AF<sup>®</sup> and must not be used to degas these types of solvents.**

The operator should note that Teflon AF<sup>®</sup> is somewhat permeable to water vapor while PTFE is not. Although the vacuum pump in the Vacuum Degasser contains internal provisions for sweeping water or solvent vapor from the pump on a continuous basis, it is possible that over time, high concentration buffers may lead to the deposition of crystals within the channel due to the loss of water within the channel. To avoid the possibility that buffer salt crystals form in the degasser, use the same precautions that are used to prevent crystallization with an HPLC pump (see Section 4.3.1, Short Term Shutdown Procedures for details).

## 1.4 Principle of Operation

When the degasser is used in conjunction with an HPLC system, the mobile phase is withdrawn from the solvent reservoir and the degassed liquid is delivered to the pump and autosampler (if applicable) as shown in Figure 1-1 for a low pressure gradient system. If the HPLC is a high pressure system with a pump for each mobile phase, a degassed solvent line would be provided as input to each pump.



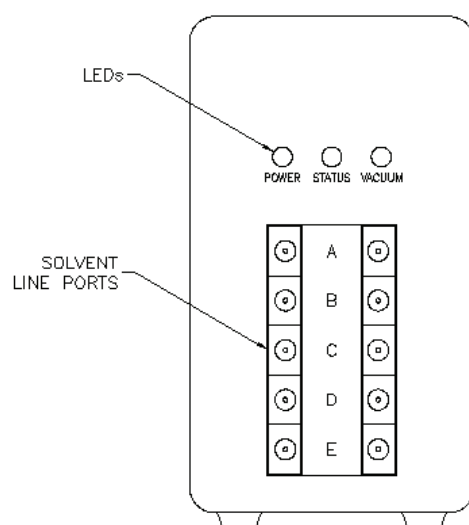
**Figure 1-1. Block Diagram of Vacuum Degasser**



## 1.5 External Components of the Degasser

### 1.5.1 The Front Panel

The front panel (Figure 1-2) includes three LED'S and a series of degassing ports (the number of ports is dependent on the system and can range from 1-5). Each channel has an input port and an output port on the same level, the flow direction is not critical. If your system has less than 5 channels, unused channels are sealed by plugs.



**Figure 1-2. Front View of 5-Channel Vacuum Degasser**

The three LED's that are located on the front of the instrument above the solvent inlets and outlets provide the following:

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

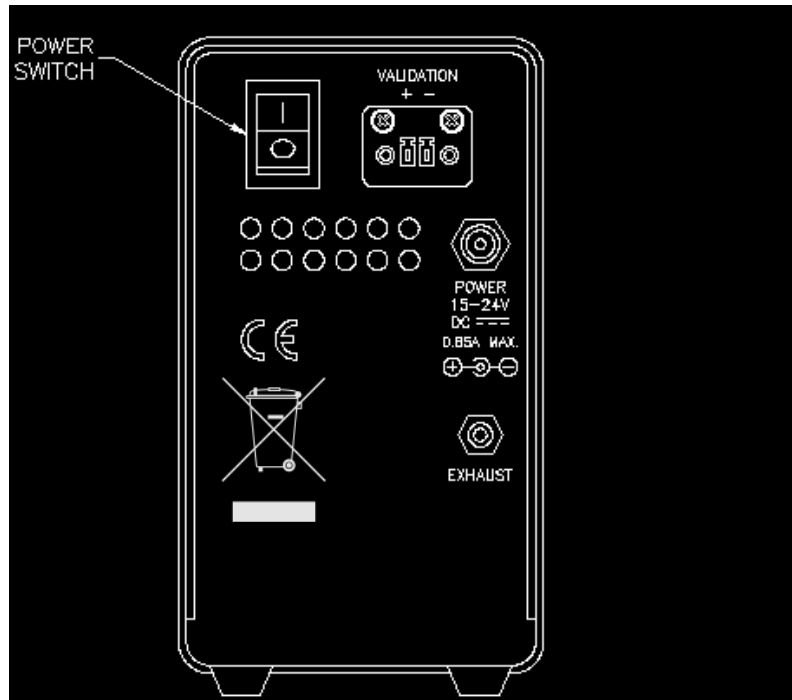
**STATUS** (Yellow) - Indicates when vacuum level is outside the acceptable operating range. Normally it will come on at initial power-up and remain on during pump-down. It will go off in a few minutes when the vacuum level goes below 100 mm of Hg absolute. If an error condition occurs, this LED will 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 the LED will illuminate after the initial pump-down, and remain on as long as the vacuum degasser is powered up and the vacuum level is below 100 mm of Hg absolute.

## 1.5.2 Rear Panel

The rear panel connections and controls are shown in Figure 1-3 (the RoHS mark is present on all units sold within the European community).



**Figure 1-3. Rear View of Vacuum Degasser**

### Exhaust Port

The gas that is evacuated from the vacuum chamber leaves the unit through the exhaust port.

### Power Receptacle

The power receptacle accepts the DC plug of the supplied AC Adapter.

### Power Switch

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

### Validation Connector (Optional)

Some models include a 2-pin receptacle labeled “Validation”, which is 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 the vacuum level and its characteristics are described in Appendix A (Specifications).

## 2 Installation

### 2.1 Unpacking the Vacuum Degasser

Carefully unpack the vacuum degasser and check for obvious signs of damage that may have occurred during shipment. The items that are shipped with the vacuum degasser are listed in Table 2-1. If any items are damaged or are missing, please contact your service representative and the shipper immediately.

**Table 2-1. Items supplied with the Vacuum Degasser**

Item	Quantity
AC Adapter (including cord)	1
Interchangeable Wall Plugs	4 (North America/Japan, U.K., Continental Europe, Australia)
Operator's Manual	1
Plug, ¼-28, Rheodyne 6118	2

**Note: If the vacuum degasser or AC adapter appears to be damaged in shipping, do not plug the system into the mains. Please call your service representative for advice.**

### 2.2 Space Requirements

The vacuum degasser is designed to sit on a bench top and is plumbed into the LC system between the solvent supply and the pump. The footprint of the degasser is approximately 8 cm (3") wide and 25 cm (10") deep). In addition, area should be provided in front of the degasser to accommodate the tubing and behind the degasser should be provided to accommodate the power cord.

## 2.3 Electrical Power Requirements

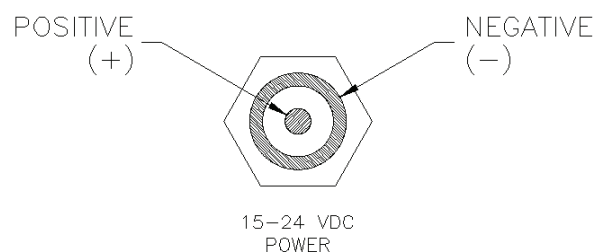
The AC adapter supplied with the vacuum degasser incorporates a universal AC input, switching regulator. This allows the instrument to operate at any AC line voltage from 100 to 240 VAC ( $\pm 10\%$ ) with a line frequency range of 47 to 63 Hz. The switching regulator senses the incoming line voltage and automatically adjusts its operation accordingly.

The switching regulator assembly in the power supply 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.

The AC Adapter that is provided with the system is normally used to supply power to the vacuum degasser. 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.

If another means is used to supply power to the rear panel power jack, the voltage must be within the range from 15 to 24 VDC at 0.85 A maximum (0.5 A typical) and should be provided using a 2.1 mm female plug.

If an alternative power supply is employed, verify that the correct polarity is observed. The center connection of the plug must be positive, and the outside must be negative as shown in Figure 2-1.



**Figure 2-1. Rear Panel Power Connector Polarity**

## 2.4 Setup

### 2.4.1 Installing the AC Adapter

To Install the AC Adapter

- a) Plug the round connector at the end of the AC adapter's cord into the power jack on the rear panel of the vacuum degasser.
- b) Select the appropriate wall plug to the mains provided in your facility and place it on the mains cable on the AC adapter.
- c) Check that the power switch is OFF and insert the mains plug into the mains.

### 2.4.2 Connecting the Tubing

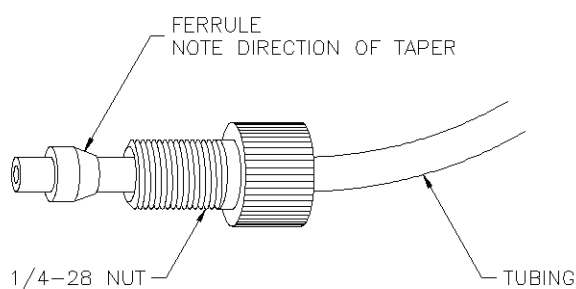
The solvent lines to be degassed and the lines to the HPLC pump are connected to the front panel ports. Each channel is defined by an input port and output port at the same horizontal level (A-E). The flow direction through the ports is not important (i.e. the input can be on the left and the output on the right or vice versa).

**Note: Unused ports must be plugged to enable the degasser to operate at its peak level of performance.**

To make a tubing connection:

- a) Connect a piece of 1/8" O.D. x 1/16" I.D. FEP<sup>®</sup> chromatography tubing to the solvent supply (do not use Teflon<sup>®</sup> tubing as air can permeate through it).
- b) Push the tubing through a PEEK 1/8" male 1/4-28 fitting and slide a ferrule over the tubing end as shown in Figure 2-2.

**Note: Make certain that gastight tubings and fittings are employed that are capable of handling solvents that are commonly used in HPLC.**



**Figure 2-2. Configuration of 1/4-28 Nut, Ferrule and Tubing**

- c) Cut the Teflon tubing so the end is flat.
- d) Screw the ¼-28 fitting into a port on the front of the vacuum degasser (Channel A, for example). The direction of flow through the vacuum degasser is not critical.

**Note: The plastic connectors should be tightened by hand. Do not overtighten the fittings as that will damage the threads.**

- e) Repeat steps a through d to connect additional lines to be degassed.
- f) Once all desired solvent lines have been connected to the vacuum degasser, plugs should be inserted into any unused ports.
- g) Prime each degassing channel 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 mL. As an alternative, you can use the priming function of the HPLC pump.



**Caution: DO NOT prime the system by pushing solvent through it. This technique can generate several hundred pounds of pressure which might rupture the tubing, even though the Teflon AF® tubing is quite rugged. The maximum recommended pressure on the tubing is 1 mPa (100 psig, 7 Bar). Do not connect the vacuum degasser to the output side of the HPLC pump as the high pressure can cause permanent damage to the degasser.**

### 2.4.3 Extending the Degassing Flow Rate Range

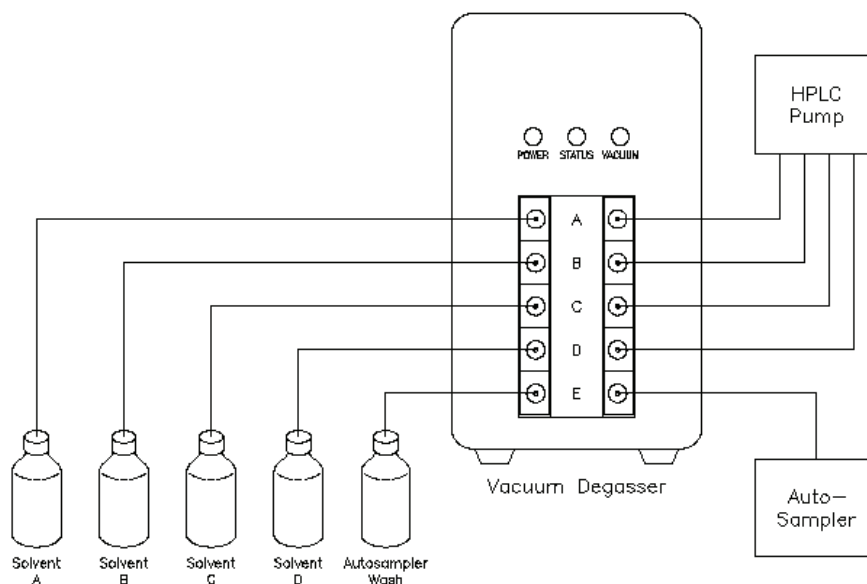
Certain organic solvents used in reversed phase chromatography including alcohols (e.g. methanol), acetonitrile and tetrahydrofuran may outgas upon mixing with water if they are not properly degassed. Degassing water and methanol through a single channel is generally sufficient to degas these solvents so outgassing does not occur upon mixing when a 75% methanol: 25% water mixture is generated by your Vacuum Degasser or pump at flow rates below 2 mL/min.

If outgassing does occur, or if a flow rate higher than 2 mL/min is required, the organic portion of the mobile phase should be passed through a second degassing channel to ensure that outgassing does not occur. This additional degassing is due to the ability of all organic solvents (e.g. methanol) to hold at least 10 times more dissolved gas than water can.

To more thoroughly degas a mobile phase, connect the outlet of the organic channel to the inlet of 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.

## 2.5 Connecting the Vacuum Degasser in a Typical System

Figure 2-3 shows the tubing connections that are made between the Vacuum Degasser and other instruments in a typical LC system. The direction of flow through the Vacuum Degasser is not critical.



**Figure 2-3. System Tubing Connections**



**Caution:** Do not connect the vacuum degasser to the output side of the HPLC pump as the high pressure can cause permanent damage to the degasser.

## 3 Operation

### 3.1 Operating Summary

**Note: This section provides a short step by step description about the use of the degasser. The operator should review the detailed information this chapter before using the system.**

When the vacuum degasser is used to degas the mobile phase for HPLC, the following steps should be performed.

- a) Select and fill each solvent reservoir with the mobile phase for your analysis.
- b) Verify that the Vacuum Degasser is properly installed as described in “Setup.”



**Caution: Never connect the Vacuum Degasser to the output side of the HPLC pump. The high pressure may cause permanent damage to the degassing membrane.**

- c) Verify that the tubing to your injector, column and detector is properly connected and check that plugs are installed in the unused ports.
- d) Disconnect the tubing that is connected to the output port and connect the priming syringe to this port. Pull the solvent through the degasser until bubbles no longer appear and then reconnect the tubing to the output port (see Section 3.3.2 for a detailed discussing about priming).
- e) Switch on the degasser (see Section 3.2) and set the flow rate of the HPLC pump to 1.0 mL/min. Allow the system to equilibrate for 5-10 minutes. The small volume contained in the 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.**



## 3.2 Powering up the Vacuum Degasser

Check to see that there is sufficient liquid in the solvent bottles before turning on the power switch on the rear panel to power up the system. When the system is powered up, the green Power LED on the front panel will be illuminated.

When the unit is initially powered up, the microprocessor examines the vacuum sensor signal to confirm that it is within the expected range. If the vacuum test is successful, the pump will be ramped to a high speed to quickly exhaust atmosphere from the vacuum chamber. As the vacuum level approaches the preset control value, the pump speed will slowly ramp down to a lower 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 be turned off and the green Vacuum LED will be lit. If you want additional confirmation that the pump is running, the slight vibration caused by the microstepping of the motor driving the vacuum pump may be felt by placing your hand on the degasser.

## 3.3 Using the Degasser

### 3.3.1 Operational Considerations



**Caution:** The degassing tubing in the Vacuum Degasser is manufactured from Teflon AF<sup>®</sup>. Teflon AF<sup>®</sup> is inert to all solvents normally used in HPLC, however, it is soluble in perfluorinated solvents such as Fluorinert<sup>®</sup> FC-75 and FC-40 and Fomblin perfluoro polyether solvents from Ausimont. In addition, Freon<sup>®</sup> solvents will adversely affect Teflon AF<sup>®</sup>. Use of such solvents in the Vacuum Degasser will result in the dissolution and hence destruction of the tubing.

**Note:** 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.

When the degasser is used, check for leaks around the ¼-28 connectors. If a leak occurs at a connector, 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.

**Note:** The plastic connectors should be tightened by hand. Do not overtighten the fittings as that will damage the threads.

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.

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 (if the speed of the pump is greater than > 300 RPM for 30 minutes), the pump will be shut down and the yellow Status LED will flash.

Maintaining a constant vacuum level allows the system to detect 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 30 minutes), the yellow LED will flash, indicating a possible leak condition, and the system will shut down and go into a “safe” mode.

### 3.3.2 Priming the Degasser

Since there is virtually no solvent retained within the Vacuum Degasser (~480 µL per channel), priming the system is relatively simple. To prime the system, use the Prime mode on the LC system pump and 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 and allows for an immediate startup of the analysis upon column equilibration.

**Note: The use of Teflon AF<sup>®</sup> membranes fully degasses solvents within the time it takes for the volume to pass through the chamber and degasses the solvents as thoroughly as, or better than the use of PTFE channels which contain approximately 40 times more solvent.**

Once air bubbles have been cleared from the solvent line, any further bubbles that are observed will be due to the lack of solvent in the solvent reservoir or from a leaking fitting.

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 system is on and running.

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.

### 3.3.3 Changing the Solvent

The single lumen coil inside the chamber contains a very small amount of solvent (approximately 480  $\mu$ L).

- If you want to change from one solvent to another where the final solvent is miscible with the first, allow the new solvent to flow through the degasser for 1-2 min before using the solvent.
- If you want to change from one solvent to another where the final solvent is not miscible with the first, use an intermediate solvent miscible with both the initial and final solvent.

**Note: Carryover from solvent to solvent is much less than previous degasser systems due to the use of Teflon AF<sup>®</sup>.**

## 3.4 Shutdown

### 3.4.1 Short-term Shutdown (Overnight and Weekends)

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

To shutdown the degasser:

- a) Remove harmful mobile phases from the vacuum degasser and other instruments in the system.
- b) Flush the column according to the instructions supplied with the column.
- c) Flush the degasser

If the solvent contains buffer salts, flush with water. If the mobile phase is allowed to evaporate, salt crystals that may form harmful deposits.



**Caution: Damage caused by precipitating buffer salts in capillary tubing, or damage resulting from this condition, is specifically excluded from warranty.**

If the mobile phase contains chloroform (or other solvents that can decompose to form hydrochloric acid) remove all traces of the mobile from the system using a suitable solvent

- d) After removing any potentially harmful mobile phases, prepare the detector 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.
- e) For weekend storage we recommend flushing 60/40% MeOH/Water through the Vacuum Degasser, pump, column, flow cell (provided your column is compatible with MeOH/Water) for a few minutes before you turn off the Vacuum Degasser, pump and detector.

### 3.4.2 Long-term Shutdown

To shut down the system for a long period of time:

- a) Follow steps (a) and (b) in the short-term shutdown procedure (Section 3.4.1).
- b) Remove the column and direct the pump output tubing to a beaker. Flush the Vacuum Degasser, first with water and then with isopropanol.
- c) Turn off the vacuum degasser. 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.
- d) Store the vacuum degasser in a clean, dry location.

**Note: Before using the vacuum degasser, completely purge it with the correct solvent for the column before reconnecting the column and restarting the system.**

## 4 Maintenance and Troubleshooting

### 4.1 Maintenance

#### 4.1.1 Preventative Maintenance

The Vacuum Degasser is designed to provide long and trouble-free performance. The procedures described below should be performed on a routine basis to minimize problems:

- a) 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.
- b) Use only high-purity gases when drying any area that may be in contact with the mobile phase.
  - Ensure that all new tubing is employed. If stainless steel tubing is used, passivate it and thoroughly flush it before making pump connections.
- c) Follow the short- and long-term shutdown procedures that are described above.
- d) Routine cleaning of the external surfaces of the instrument can be done using a clean, damp cloth. Immediately clean any spills which occur on or near the instrument using methods appropriate for the type of spill. Some solvents can damage the appearance and function of the instrument.

#### 4.1.2 Routine Maintenance

On a routine basis:

- Check the conditions of the filters in the system and replace if necessary. Do not allow particulate matter to enter the degasser.
- Check that the tubing connecting the solvent bottle and the degasser, as well as the tubing between the degasser and the pump. Replace the tubing if you see discoloration or cracks.

## 4.2 Troubleshooting Guide

Problem	Probable Cause	Solution
1) Power switch is on, but all 3 LEDs are off, indicating no power to the degasser.	1a) The AC Adapter is not plugged into the AC outlet. 1b) Blown fuse.	1a) Plug the AC Adapter into the AC outlet. 1b) Contact your Service Representative.
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) This is normal start up operation. If pump speed continues at a high speed 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) The Power and Vacuum green LEDs are illuminated, but the pump cannot 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) Outgassing of the mobile phase after the degasser	7) Organic solvent is mixed with water	7) Use a second degassing channel (see Section 2.4.3)
7) No solvent flow.	7a) Air in the HPLC pump head. 7b) If a buffer solvent was left in the degasser for some time after use, it may plug the degasser elements.	7a) Prime/purge the pump head. 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.

# Appendix A Specifications of the Vacuum Degasser

## A.1 General

Channels	1-5 independent
Mode of Degassing	Gas permeation through a fluoropolymer tube
Maximum Flow Rate	10 mL/min.
Degassing Capacity	~2 ppm at 1 mL/min.
Dead Volume	~480 $\mu$ L per channel for standard channel
Materials contacting solvents	PEEK, Glass-filled PTFE, Teflon AF <sup>®</sup>

## A.2 Power

Power requirement if using supplied AC Adapter	100 to 240 VAC ( $\pm$ 10%), 1A, 50 to 60 Hz ( $\pm$ 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

## A.3 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

#### A.4 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

#### A.5 Storage Conditions

Ambient temperature	-20 to +60 °C
Ambient Relative Humidity	20 to 80% RH (without condensation)
Altitude	0 to 12000 M

#### A.6 Physical Characteristics

Dimensions:	Height: 127 mm (5.0" in.) Width: 73 mm (2.8" in.) Depth: 250 mm (9.8" in.)
Weight	2.7 kg (6 lb).



# Appendix B Regulatory Considerations

## B.1 CE Declaration of Conformity

### SUPPLIER DETAILS

#### Manufacturer

Biotech AB

#### Address

Box 133  
S-43923 Onsala  
Sweden

### PRODUCT DETAILS

#### Product Description - name and number

Name: Vacuum Degasser

Model

Description: Vacuum Degasser

#### Directive(s) and Standard(s) to which conformity is declared

EMC directive 89/336/EEC as amended by 92/31/EEC and 93/68/EEC

Low Voltage Directive 73/23/EEC as amended by 93/68/EEC

EN 61326-1: 1997 Incorporating Amendments A1: 1998

EN 61010-1:2001 (2<sup>nd</sup> Edition)

### DECLARATIONS

I hereby declare that the product specified conforms with the provisions of Council Directive(s) and standards listed above

Signed

Date: 2007-03-01

Print Name: Ralf Jutvik

## B.2 Federal Communications Commissions (FCC) Notice (US only)

This product conforms to FCC regulations relating to Radio Frequency Devices. These have been satisfied by testing the product against, and being found to be compliant with

*FCC CFR 47 Part 15: March 2003 Class A digital device*

The 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.

**Note: This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communication. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.**

**Note: Changes or modifications not expressly approved by the supplier could void the user's authority to operate the equipment.**

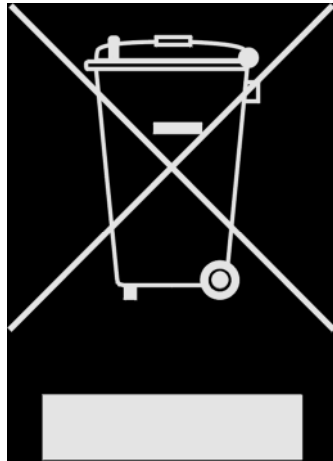
### **B.3 Disposal of Electrical and Electronic Equipment**

This regulation is applicable in the European Union and other European countries with separate collection systems.

The symbol in Figure B-1 is attached to the product to indicate that when the last user wants to discard this product, it must not be treated as general waste. It should be given to the appropriate facility for the recovery and recycling of electrical and electronic equipment.

By not discarding this product along with other household-type waste, the volume of waste sent to incinerators or landfills will be reduced and natural resources will be conserved.

For more details about recycling of this product, please contact your local refuse department, your waste disposal service or the supplier of the product.



**Figure B-1. Disposal Mark**

# Index

## **C**

Changing the Solvent, 19  
Connecting the Tubing, 13

## **D**

Declaration of Conformity, 25  
Disposal of Electrical and Electronic Equipment, 27

## **E**

Exhaust Port, 10  
Extending the Degassing Flow Rate Range, 14  
External Components, 9

## **F**

Federal Communications Commissions (FCC) Notice, 26  
Front Panel, 9

## **I**

Installation, 11  
Installing the AC Adapter, 13  
Introduction, 6

## **L**

LED's, 9  
Long-term Shutdown, 20

## **M**

Maintenance, 21

## **O**

Operation, 16  
Overview, 6

## **P**

POWER LED, 9  
Power Connector Polarity, 12  
Power Receptacle, 10  
Power Requirements, 12  
Power Switch, 10  
Powering up the Vacuum Degasser, 17  
Preventative Maintenance, 21

Priming the Degasser, 18  
Principle of Operation, 6, 8

## **R**

Rear Panel, 10  
Regulatory Considerations, 25  
Routine Maintenance, 21

## **S**

Setup, 13  
Shipping List, 11  
Short-term Shutdown, 19  
Shutdown, 19  
Space Requirements, 11  
Specifications, 23  
STATUS LED, 9  
System Tubing Connections, 15

## **T**

Teflon AF, 7  
Troubleshooting, 22

## **U**

Unpacking, 11  
Using the Degasser, 17

## **V**

VACUUM LED, 9  
Validation Connector, 10

## **Z**

Zero Vacuum “Hysteresis”, 6

---

Notes

A series of horizontal dashed lines for writing notes.

---

Notes

-----

-----

-----

-----

-----

-----

-----

-----



