



Discovery Normal Phase SPE Products

Base Silica: irregular shaped, acid washed; 50µm particle size, 70Å pore diameter, 480m²/g specific surface area, 0.9cm³/g pore volume

Discovery normal phase SPE products are specifically developed, tested and quality controlled for normal phase pharmaceutical applications and other modified flash techniques. The Discovery normal phase product line enables you to quickly and effectively extract, isolate, purify, and concentrate polar compounds from non-polar solutions. Its highly selective properties allows the user to separate or remove structurally similar molecules through successive wash/elutions with increasingly polar solutions.

- Excellent capacity for the **baseline clean-up** of solution phase combinatorial chemistry reactions (removing target molecules from reaction by-products and excess reagents)
- Available in Büchner Funnels for easier scalability, and 96-well plates for the high throughput purification of CombiChem libraries
- Improve extraction selectivity through Discovery's narrower pore size distribution
- Acid washed to reduced metal chelating activity

A comprehensive line of normal-phase chemistries (four different SPE chemistries) and hardware configurations to meet your diverse and most demanding sample prep needs.

DSC-Si -Si-OH	<ul style="list-style-type: none"> - Unbonded acid washed silica sorbent ideal for normal phase SPE and other modified flash techniques - Considered the most polar normal phase sorbent available - Excellent capacity for purifying solution phase CombiChem reactions when removing target molecules from reaction by-products and excess reagents - Available in Büchner Funnel configurations for easy scalability
DSC-Diol $\begin{array}{c} \\ \text{---Si---}(\text{CH}_2)_3\text{CH}_2\text{CH---CH}_2 \\ \qquad \qquad \qquad \qquad \qquad \qquad \\ \qquad \qquad \qquad \text{OH} \qquad \qquad \qquad \text{OH} \end{array}$ <small>G001627</small>	<ul style="list-style-type: none"> - Polymerically bonded, 2,3-Dihydroxypropoxypropyl (7% C) - Polar sorbent most commonly used for normal phase applications (polar extractions from non-polar matrices) - The sorbent's dihydroxy groups facilitates strong hydrogen bonding - Excellent selectivity when extracting structurally similar molecules
DSC-CN $\begin{array}{c} \\ \text{---Si---}(\text{CH}_2)_3\text{CN} \\ \end{array}$ <small>G001626</small>	<ul style="list-style-type: none"> - Monomerically bonded, Cyanopropyl (7% C), endcapped - Can behave as either reversed phase or normal phase - Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-1B - Less retentive than DSC-Si or DSC-Diol when used as normal phase (organic matrices such as hexane or oils) - Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents
DSC-NH₂ $\begin{array}{c} \\ \text{---Si---}(\text{CH}_2)_3\text{NH}_2 \\ \end{array}$ <small>G001631</small>	<ul style="list-style-type: none"> - Polymerically bonded, aminopropyl phase that is very polar in nature (hydrogen bonding) allowing for both normal phase and ion exchange applications - A weak anion exchanger with a pKa of 9.8. At pH 7.8 or below, the functional groups are positively charged - Allows the rapid release of very strong anions such as sulfonic acids that may be retained irreversibly on SAX (a quaternary amine sorbent that is always positively charged) - Can be used in some reversed phase applications (due to ethyl spacer); however, it is predominately used as an ion-exchanger or normal phase sorbent due to its polar nature

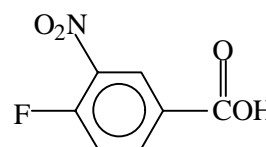
Here's the Proof:

Combinatorial Chemistry Application

Many Combinatorial Chemistry Labs of today are synthesizing and characterizing extensive compound libraries. Chemists are therefore employing modified flash chromatography techniques in a 96-well SPE format for the purposes of sample clean-up, solid baseline impurity removal. In such cases, impurities are selectively retained on the stationary phase, and the compounds of interest are collected during the sample load and/or subsequent wash steps. In many combinatorial chemistry labs, capacity is a primary concern for such applications.

In our studies, we have determined the binding capacity of 4-Fluoro-3-Nitrobenzoic acid when loaded on to DSC-SI SPE-96 (100 mg/well). Our results show that 112.5mg of the load compound can be loaded on to 100mg DSC-SI before breakthrough occurs. Breakthrough (determined) was analyzed via HPLC analysis. See **Table 1**.

Table 1. Binding Capacity of 4-Fluoro-3-Nitrobenzoic acid on DSC-SI (100mg/well)



4-Fluoro-3-Nitrobenzoic acid

G001555

Load Amount (Same as Matrix = 200µL Methylene Chloride)	Breakthrough Amount
2.5mg	No Breakthrough
5.0mg	No Breakthrough
10.0mg	No Breakthrough
12.5mg	No Breakthrough
15.0mg	0.10% Breakthrough Occurred

n = 3 for each load amount.

Ordering Information:

Discovery Normal Phase SPE Products

Product	Qty./Pk	DSC-CN	DSC-Si	DSC-Diol	DSC-NH ₂
Discovery SPE Tubes					
50mg/1mL	100 qty/pk	52693-U	52652-U	52747-U	52635-U
100mg/1mL	100 qty/pk	52694-U	52653-U	52748-U	52636-U
500mg/5mL	54 qty/pk	52695-U	52654-U	52751-U	52637-U
500mg/6mL	30 qty/pk	52696-U	52655-U	52752-U	52638-U
1g/6mL	20 qty/pk	52697-U	52656-U	52753-U	52640-U
2g/12mL	30 qty/pk	52698-U	52657-U	Custom	52641-U
5g/20mL	20 qty/pk	52699-U	52658-U	Custom	52642-U
10g/60mL	20 qty/pk	52700-U	52659-U	Custom	52644-U
Bulk packing	100g	52222-U	52651-U	52229-U	52212-U
Discovery SPE-96 Well Plates					
100mg/well	1ea	575624-U	575609-U	575646-U	575615-U
50mg/well	1ea	575625-U	575608-U	575647-U	575616-U
25mg/well	1ea	575626-U	575607-U	575648-U	575617-U
Discovery Büchner Funnels					
55mm ID x 30mm H, 25g	6 qty/pk	Custom	52591-U	Custom	Custom
75mm ID x 40mm H, 25g	6 qty/pk	Custom	52592-U	Custom	Custom
90mm ID x 42mm H, 50g	6 qty/pk	Custom	52593-U	Custom	Custom
110mm ID x 66mm H, 100g	6 qty/pk	Custom	52594-U	Custom	Custom

Trademark

Discovery is a trademark of Sigma-Aldrich Co.

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This Data Sheet Contains Important Information About The Product.

VersaFlashSM

High Throughput Flash Purification Station
(Patent pending)
Cat. No. 97732-U



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SUPELCO

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VersaFlash™



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This system is covered by a one year limited warranty.

A copy of the warranty is included in this manual.

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Printed in the United States.

VersaFlash™

Station

Cartridges

Pumps

Kits



Accessories

Stacking
Connection

Replacement
Parts

Warnings and Safety Information

1. Install the VersaFlash station in a fume hood or a vented enclosure capable of handling solvent fumes associated with VersaFlash operation in accordance with OSHA and internal health, safety and environmental guidelines.
2. Store VersaPak™ cartridges in the original packaging in a cool, dry location.
3. Before disposing of spent VersaPak cartridges, expel solvent by applying an air flow or by vacuum using the VersaVac™ manifold.
4. Dispose of all VersaPak cartridges in accordance with health, safety and environmental guidelines.
5. Install the VersaFlash station in a location free of temperature fluctuations greater than +/- 3°C (+/- 5°F). For best results maintain a temperature of approximately 24°C.
6. Keep all solvent bottles covered and stored in a flameproof solvent cabinet.
7. For best pump performance, filter all solvents prior to use with the VersaFlash station. The Supelco 10µm stainless steel inlet filter (Cat. No. 59277) is recommended.
8. Thoroughly clean and rinse solvent reservoir glassware prior to use with the VersaFlash station.
9. Do not attach clamps or ringstand apparatus to the uprights of the VersaFlash station as this may damage the smooth operation of the system.
10. Do not force the handle of the VersaFlash station when securing a VersaPak cartridge into the OPERATE position.
11. Do not attempt to open VersaPak cartridges. Supelco will not be responsible for VersaPak cartridges that have been opened or repacked.
12. Use of any repacked or non Supelco approved cartridges may void the warranty of the VersaFlash station.
13. Immediately report any leaks from the VersaFlash station to Supelco Technical Service.
14. When changing solvents, flush the VersaFlash station using the solvent flush connector. This is required after step-wise or continuous gradient purification.
15. Follow all safety precautions indicated in the manual provided with the pump.
16. Attaching accessories such as a UV detector or fraction collector downstream of the VersaFlash station can create additional backpressure on the station and the cartridges. Care should be taken not to exceed the maximum pressure ratings of the cartridges.
17. The crimped ends of the VersaPak cartridge are designed only to hold the end-fittings, frits and packing material in place during shipment. They are not designed to withstand normal operating pressures for flash chromatography. In order for the cartridges to work properly without leakage, they must be securely held in place between the Upper and Lower Platens (refer to VersaFlash Station Operating Instructions).
18. The spring loaded PEEK seals are designed to leak before the cartridge o-rings leak. If leakage is coming from the PEEK seal, then the operating pressure is exceeding the specified maximum. To correct, simply decrease the pump flow rate, and the PEEK seal should automatically seal.

◀ Figure 1-1:
The Supelco VersaFlash
High Throughput Flash
Purification System

VersaFlashTM

High Throughput Flash Purification Station

Chapter 1: Introduction

The Supelco VersaFlash System is a high throughput flash purification system (Figure 1-1) that is used for rapid separation of substances from a sample so that they can be used for further evaluation.

The complete system consists of three components (also sold separately):

- A VersaFlash Station which holds the VersaPak cartridge in place
- A pump which is used to deliver the mobile phase to perform the separation.
- A VersaVac sample loading station for loading samples onto cartridges prior to purification.

A variety of cartridges designed to be used with the unit are available from Supelco. A listing of available cartridges is provided on page 13.

VersaPak cartridges are pre-compressed, disposable cartridges containing an adsorbent or silica based bonded phase sorbent, quality-controlled to meet your stringent separation requirements. Cartridge compression reduces particle interstitial spacing and wall effects to provide even mass transfer and balanced chemical interaction. The

spherical geometry of the particles greatly reduces the possibility of particulate fines due to fracture within the cartridge bed. The result is faster separations, greater band definition and lower back-pressure.

The unique, patent pending symmetrical cartridge design leads to a variety of features that will allow you the versatility to easily complete flash separations. The versatile design means that the cartridge is bi-directional, leading to the ability to let the solvent travel through the cartridge in either direction and even reverse the flow during the separation, should you encounter a slow moving, or tightly retained compound that you would like to elute quickly and with less solvent (Rev-Elution).

The symmetrical cartridge design also allows the cartridges to be stacked one on top of the other, leading to the ability to expand length and/or combine phase chemistries for multiple, simultaneous separations.

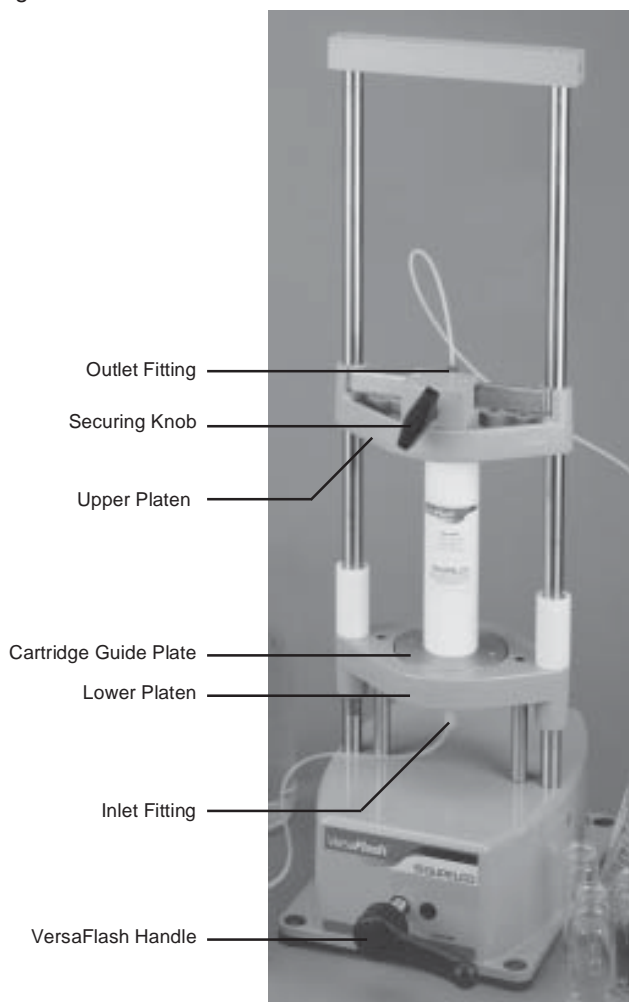
An additional advantage of the VersaPak cartridges is the way in which they seal in the VersaFlash High Throughput Flash Purification (HTFP) station. This allows users to employ all four sizes of cartridges in the same system, eliminating the need to purchase and change-out additional expensive compression barrels.

Chapter 2: Installation

2.1: Overview

This chapter describes how the system is to be installed in the laboratory and includes a short protocol to verify that it is operating properly. Once you have completed the installation procedure, the system is ready to operate. Figure 2-1 shows the key components critical to the VersaFlash Station operation.

Figure 2-1: The VersaFlash Station



2.2: Unpacking

The complete VersaFlash system is comprised of the following components (sold separately).

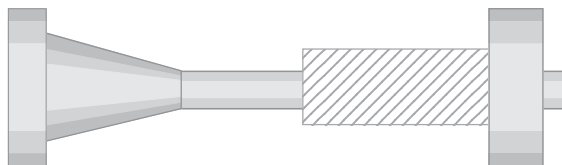
- A VersaFlash Station which holds the VersaPak cartridge in place
- A pump which is used to deliver the mobile phase to perform the separation.
- A VersaVac sample loading station for loading samples onto cartridges.

2.3: Locating the VersaFlash Station

Place the VersaFlash Station in a fume hood or a vented enclosure in a position with easy access to the handle and the Upper Platen.

Place the pump next to the VersaFlash Station so that the outlet of the pump faces the inlet tubing of the VersaFlash Station.

- a) Cut a length of tubing to be connected to the outlet of the pump. The length should be sufficient to reach the inlet of the VersaFlash Station. Use a safety razor or other sharp instrument to cut the plastic tubing. The cut end must be smooth and square.
- b) Slide the knurled Delrin nut onto the tubing.
- c) Slide the Tefzel ferrule onto the tubing with the beveled edge facing the nut. Align the outer surface of the ferrule flush with the end of the tubing.



- d) Insert the fitting into the outlet fitting of the pump. It should be finger tight so that no leaks occur.

Note: The ferrule and the nut can be reused multiple times, so long as the ferrule is not scored or scratched or the threads on the nut are not stripped. Over-tightening can result in leaks.

2.3: Locating the VersaFlash Station (cont.)

Note: If the ferrule is not placed in the correct direction as shown in c), the fitting will leak.

- e) Cut another length of tubing to be used to deliver solvent to the inlet of the pump. The length should allow for immersion to the bottom of a large flask or solvent bottle.
- f) Follow the same procedure (steps b and c) to place a fitting on one end of the tubing.
- g) Insert the fitting into the inlet fitting of the pump and tighten.
- h) Place a 10 μ m stainless steel filter (Supelco Cat. No. 59277) on the open end of the inlet tubing and place the tube with the filter into a flask containing distilled water.
- i) Place a Solvent Flush Connector (from the starter kit) into the VersaFlash Station following the procedure in section 3.2.
- j) Cut another length of tubing for the outlet of the VersaFlash Station. There should be ample length to reach the point of collection of the effluent from the VersaFlash Station.
- k) Follow the aforementioned procedure to put a fitting on the end of the tubing and connect the fitting to the outlet of the Solvent Station.
- l) Connect the loose end of the pump outlet tubing to the inlet fitting of the VersaFlash Station.

Leak Test Procedure

- m) Place the end of the outlet tubing from the VersaFlash Station into a collection flask.
- n) Turn on the pump (see pump manual). Set pump speed to 2.5 on the dial.
- o) Watch the flow path and check for leaks at all fitting connections. Tighten fittings as needed.

Note: If leaks occur at the inlet or outlet of the VersaFlash System after tightening of the fittings, re-tighten leaking fittings.

- p) When the system is free of leaks, it is ready for use.

Caution: Follow OSHA and internal safety guidelines as applied to flammable and hazardous solvents.

Chapter 3: General Operating Procedure

3.1: Overview

This chapter describes the general operating protocol for the system and includes all the information required for operating the system on a routine basis.

An overview of the system is presented in Figure 3-1.

3.2: Cartridge Loading

To load a cartridge:

Note: Be sure that the cartridge loading procedure is done with the pump off.

- a) Place the handle of the VersaFlash station in the **Idle** position and pull it out as shown in Figure 3-2, then place the VersaPak cartridge on the lower platen so that the inlet hole aligns with the PEEK seal (shown in Figure 3-3) of the lower platen.
- b) Push the handle inward causing the Lower Platen to rise. The handle is now in the **Idle** position as shown in Figure 3-4.

Figure 3-1: The VersaFlash High Throughput Flash System



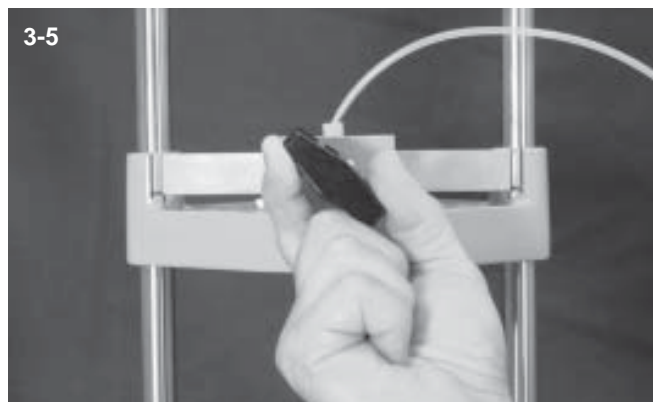
- c) Lower the Upper Platen by turning the Securing Knob (Figure 3-5) counterclockwise and guiding it downward until it securely meets with the outlet end fitting of the cartridge and the PEEK seal is aligned with the outlet hole of the cartridge. Continue to lower the Upper Platen until it rests flush with the cartridge end fitting.
- d) Turn the Securing Knob clockwise until the Upper Platen is secure and the cartridge is held tightly in place.
- e) Turn the handle clockwise until it reaches the **Operate** position and the cartridge is tightly held between the Upper and Lower platens.

Note: Do not overtighten. It is not critical that the handle turns all the way to the operate position. The patent pending design allows the Lower Platen to lock into position at any point along the clockwise rotation. How far the handle turns is dependent on the pressure applied to the Upper Platen when securing it on top of the cartridge. The more pressure applied, the less likely you will be able to turn the handle all the way to the operate position.

- f) Ensure that the bottom cartridge guide plate cannot be depressed. By using your thumb and forefingers, try to depress it on the Lower Platen as shown in Figure 3-8.

Note: It is critical that the station is set up so that the Upper and Lower Platens securely hold the cartridge end-fittings in place. The Bottom Guide Plate should be fully depressed.

- g) Turn on the pump and adjust the flow rate as necessary.



3.3: Sample Loading

There are at least five ways to load a sample onto a VersaPak Cartridge. They are:

1. Direct syringe injection onto the cartridge (off-line) – Section 3.3.1.
2. Vacuum aspiration onto the cartridge (off-line) – Section 3.3.2.
3. Using a solid sample cartridge prior to the primary cartridge (on-line) – Section 3.3.3.
4. Using a valve and loop injector for repetitive volume sample loading (on-line) – Section 3.3.4.
5. Through the pump loading for dilute samples (on-line) – Section 3.3.5.

3.3.1: Direct Syringe Injection onto the Cartridge (Off-Line)

Note: In most cases direct syringe injection is recommended prior to placing the cartridge onto the VersaFlash station.

To Perform Direct Sample Injection:

- a) Using a non threaded male Luer syringe, draw up an aliquot of sample of desired volume.
- b) Insert the male Luer of the syringe into the hole on either end of the cartridge until it fits tightly into the cartridge end-fitting.

Figure 3-9:



- c) Inject the sample into the cartridge.
- d) Put the VersaPak cartridge into the VersaFlash Station so that the sample is on the inlet side of the cartridge (i.e. bottom for upward flow, top for downward flow).

3.3.2: Vacuum Aspiration onto the Cartridge (Off-Line)

Cartridges can be loaded via the VersaVac Sample Loading Station when one or more cartridges (as many as six) are to be loaded at the same time. This method also allows the loading of samples through SPE tubes for pretreatment prior to application into the VersaPak cartridge.

The setup of the VersaVac requires a vacuum source as shown in Figure 3-10

Figure 3-10:



- a) To aspirate sample into the inlet of the cartridge, place the cartridge(s) onto the male Luer fitting(s) on the lid of the VersaVac.
- b) Place an empty syringe barrel or SPE tube on the inlet to the cartridge. Cap off the unused male Luer fittings using female Luer caps (Cat. No. 57098).
- c) To apply the sample(s), pour an aliquot of the desired volume of sample material into the empty syringe barrel or SPE tube and turn on the vacuum (Figure 3-11). Allow the sample material to empty into the cartridge(s).

Note: Slow loading under low vacuum is recommended.

- For a partial separation in the cartridge while it is on the VersaVac, follow the sample with a volume of solvent matching the starting mobile phase fluid that will be used when the cartridge is transferred to the VersaFlash Station.
- d) Once the sample is on the cartridge, remove the syringe barrel or SPE tube and transfer the cartridge to the purification station for final purification and elution of desired components.

Figure 3-11:



3.3.3: Using a Solid Sample Cartridge

If the sample material exhibits low solubility in the mobile phase to be used in the flash purification, it may be necessary to apply the sample through the use of a Solid Sample Cartridge using the following procedure:

- a) Adsorb the sample material onto a solid support media such as silica or celite. The media is then dried *in vacuo* along with the adsorbed sample material.
- b) Pour a desired volume of adsorbed media into the appropriate sized Solid Sample Cartridge.

3-12



- c) Once the desired volume of adsorbed media is in the Solid Sample Cartridge, backfill to the fill line with non-adsorbed packing media such as celite. Periodically tap the bottom of the cartridge gently on a hard surface to settle the packing material without voids. This will help insure a uniformly packed cartridge leading to proper desorption.



- d) Place a frit (smooth side up) onto the open end of the Solid Sample Cartridge and slide the frit carefully down into the cartridge.

- e) When the frit is securely held inside the opening of the cartridge, place the inlet end-fitting over the frit so that the O-ring on the fitting is in contact with the sides of the cartridge and place in the VersaFlash station as shown in Figure 3-14.



- f) Turn the VersaFlash handle to the idle position with it pushed in. Lower the Upper Platen so that it is resting on top of the end-fitting to be pressed into the solid sample cartridge, and tighten the securing knob.

- g) Turn the handle 180 degrees to the **Operate** position. The lower platen will rise securing the top end-fitting in place. If the end-fitting does not push in completely (flush with the frit), repeat steps f) and g) until it is completely seated in the cartridge. The Solid Sample Cartridge is now fully assembled.



- h) Return the handle to the idle position and pull it out. Stack the VersaPak cartridge in line after (downstream of) the solid sample cartridge so that the sample will be eluted onto the VersaPak cartridge.

3.3.4: Using the Valve-and-Loop Accessory

When a fixed volume of sample fluid is to be repetitively applied to one or more VersaPak cartridges, the valve and loop accessory (Cat. No. 57683, 1mL) is a useful alternative to direct syringe loading. The loop enables finite volumes to be premeasured and ready for loading even while a purification is occurring on the VersaFlash purification station. (Additional sample loop volumes are available.)

3.3.4.1: Set-up

- a) Mount the valve-and-loop accessory on a ringstand close to the VersaFlash Station.
- b) Select the loop that will be used for loading the VersaPak cartridge. Standard valve-and-loop accessory injection valves in 1, 5 and 10mL volumes are available. Follow the instructions with the valve-and-loop assembly to connect the loop to the proper fittings on the valve.
- c) Connect the inlet tube of the valve to the outlet of the pump. Then connect the outlet tube of the valve to the inlet of the VersaFlash Station.

Caution: Always use mobile phase fluid that is compatible with the sample and sample solvent. Precipitation and/or emulsion can result in serious blockage and/or contamination of the flow path.

3.3.4.2: Operation

- a) With the pump off, and the valve in the LOAD position, fill the 25mL syringe, supplied with the valve-and-loop kit, and connect it to the loop loading tube. Place the overflow tube into a flask to collect the overflow sample fluid.
- b) Apply the volume of sample material to completely fill the loop.
- c) Turn the valve to the INJECT position and turn on the pump at a preset flow rate.
- d) Leave the valve in the INJECT position until the sample volume is completely moved into the VersaPak cartridge. To load another sample, return the valve to the LOAD position and repeat the procedure as described above.

3.3.5: Trace Enrichment (Through the pump sample loading)

When sample material is present in a matrix fluid at very low concentration, it is possible to concentrate the sample component on a VersaPak cartridge by passing large volumes of matrix fluid through the cartridge while trapping the sample component via interaction with the stationary phase. Often the solute is found in water sources, requiring a VersaPak C18 reversed phase cartridge for the concentration of the dilute sample component.

3.3.5.1: Set-up

Caution: Be sure that all fluid is filtered through a fine mesh non-adsorptive filter prior to entering the pump. Fine particles in the matrix fluid can damage the internal mechanism of many pumping devices.

- a) After the matrix fluid has been filtered, use a 2 liter flask or bottle to contain the fluid. Place a stainless steel Inlet Filter (Cat. No. 59277) on the inlet tube to the pump and place the filter at the bottom of the sample matrix vessel.
- b) On the outlet end of the VersaFlash Station, place a large vessel (>3 liters) for the collection of effluent.
- c) Select a VersaPak cartridge of the appropriate size and selectivity. Place the cartridge in the VersaFlash Station and set in the **OPERATE** position.
- d) Set the flow rate of the pump and start the pump.
- e) Allow enough time for the trace sample component to adsorb onto the packing media.
- f) Reverse the VersaPak Cartridge (Rev-Elution) and switch over to a stronger reversed phase mobile phase (e.g.: water:MeOH), and elute the sample component of interest.

3.4: Rev-Elution

Rev-Elution is a powerful technique to reverse the flow through a VersaPak cartridge by revolving the cartridge 180 degrees. Rev-Elution is used to elute fully retained or slow moving substances without the need to travel the entire length of the cartridge before eluting. This technique reduces band spreading, reduces contamination of desired substances and conserves solvent.

Rev-Elution is performed as follows.

- a) Following the elution of non-retained and fast moving substances, stop the flow by turning off the pump.
- b) Remove the cartridge from the VersaFlash Station.
- c) Return the cartridge to the VersaFlash Station by turning it 180 degrees from the original position. (The retained substance is now on the outlet end of the cartridge.)
- d) Turn the pump on and collect the eluting substance.

Chapter 4: Maintenance and Troubleshooting

4.1: Maintenance

The VersaFlash Station is designed to minimize the effort required for separation.

Before using the station:

- Check the station for the presence of particulate matter. If particulate matter is observed, clean the station and remove any particulate matter that may have deposited on the outside.
- Check the station for leaks. If a leak is observed, gently tighten the offending fitting (do not overtighten).
- Replace the cartridge (if necessary)

After finishing a series of separations:

- Remove the eluting solvent by rinsing with two to three volumes of water. If the solvent used is not miscible with water, use an intermediate solvent such as isopropanol first.
- On a periodic basis it may be necessary to perform maintenance on the pump (e.g. replace brushes, gears, seals) as described in the manual supplied with the pump.
- The threads on the Upper Platen Securing Knob should be lubricated with a lithium bearing grease approximately every 500 cycles to insure smooth operation.

4.2: Troubleshooting

- **Improper seating of the cartridges in the PEEK seal assembly:** Turn the handle to the idle position and pull out. Adjust the cartridge back and forth while pushing the handle in, so that the cartridge seals properly in the PEEK Seal Assembly.

VersaFlash handle does not turn all the way to the operate position: It is not critical that the handle turns all the way to the operate position. The patent pending design allows the Lower Platen to lock into position at any point along the clockwise rotation. How far the handle turns is dependent on the pressure applied to the Upper Platen when securing it on top of the cartridge. The more pressure applied, the less likely you will be able to turn the handle all the way to the operate position.

Specifications

VersaFlash High Throughput Flash Purification Station

Height: 27.5 in.

Width: 8 in.

Length: 11 in.

Weight: 27 lbs.

Systems, Cartridges, Kits and Accessories

Systems

Item	Cat. No.
VersaFlash System I (VersaFlash Station, Pump, VersaVac, Starter Kit)	97730-U
VersaFlash System II (VersaFlash Station, VersaVac, Starter Kit)	97731-U
VersaFlash Station	97732-U

Silica Cartridges

Size	Qty.	Cat. No.
40 x 75mm	12	97704-U
	96	97705-U
40 x 150mm	6	97706-U
	48	97707-U
80 x 150mm	2	97708-U
	12	97709-U
80 x 300mm	1	97710-U
	6	97711-U

C18 Cartridges

Size	Qty.	Cat. No.
40 x 75mm	2	97700-U
40 x 150mm	1	97701-U
80 x 150mm	1	97702-U
80 x 300mm	1	97703-U

Pumps

Item	Qty.	Cat. No.
Assembled Pump, 120V	1	97734-U
Assembled Pump, 220V	1	97737-U

Kits & Accessories

Item	Cat. No.
Starter Kit	97739-U
Solid Sample Cartridge Kit	97738-U
Cartridge Stacking Assembly (40 x 40mm)	97740-U
Cartridge Stacking Assembly (40 x 80mm)	97741-U
Cartridge Stacking Assembly (80 x 80mm)	97742-U
Solvent Flush Connector	97743-U
VersaVac Sample Loading Station	97750-U

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This Data Sheet Contains Important Information About The Product.

VersaFlashSM

High Throughput Flash Purification Station
(Patent pending)
Cat. No. 97732-U



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SUPELCO

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VersaFlash™



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This system is covered by a one year limited warranty.

A copy of the warranty is included in this manual.

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Printed in the United States.

VersaFlash™

Station

Cartridges

Pumps

Kits



Accessories

Stacking
Connection

Replacement
Parts

Warnings and Safety Information

1. Install the VersaFlash station in a fume hood or a vented enclosure capable of handling solvent fumes associated with VersaFlash operation in accordance with OSHA and internal health, safety and environmental guidelines.
2. Store VersaPak™ cartridges in the original packaging in a cool, dry location.
3. Before disposing of spent VersaPak cartridges, expel solvent by applying an air flow or by vacuum using the VersaVac™ manifold.
4. Dispose of all VersaPak cartridges in accordance with health, safety and environmental guidelines.
5. Install the VersaFlash station in a location free of temperature fluctuations greater than +/- 3°C (+/- 5°F). For best results maintain a temperature of approximately 24°C.
6. Keep all solvent bottles covered and stored in a flameproof solvent cabinet.
7. For best pump performance, filter all solvents prior to use with the VersaFlash station. The Supelco 10µm stainless steel inlet filter (Cat. No. 59277) is recommended.
8. Thoroughly clean and rinse solvent reservoir glassware prior to use with the VersaFlash station.
9. Do not attach clamps or ringstand apparatus to the uprights of the VersaFlash station as this may damage the smooth operation of the system.
10. Do not force the handle of the VersaFlash station when securing a VersaPak cartridge into the OPERATE position.
11. Do not attempt to open VersaPak cartridges. Supelco will not be responsible for VersaPak cartridges that have been opened or repacked.
12. Use of any repacked or non Supelco approved cartridges may void the warranty of the VersaFlash station.
13. Immediately report any leaks from the VersaFlash station to Supelco Technical Service.
14. When changing solvents, flush the VersaFlash station using the solvent flush connector. This is required after step-wise or continuous gradient purification.
15. Follow all safety precautions indicated in the manual provided with the pump.
16. Attaching accessories such as a UV detector or fraction collector downstream of the VersaFlash station can create additional backpressure on the station and the cartridges. Care should be taken not to exceed the maximum pressure ratings of the cartridges.
17. The crimped ends of the VersaPak cartridge are designed only to hold the end-fittings, frits and packing material in place during shipment. They are not designed to withstand normal operating pressures for flash chromatography. In order for the cartridges to work properly without leakage, they must be securely held in place between the Upper and Lower Platens (refer to VersaFlash Station Operating Instructions).
18. The spring loaded PEEK seals are designed to leak before the cartridge o-rings leak. If leakage is coming from the PEEK seal, then the operating pressure is exceeding the specified maximum. To correct, simply decrease the pump flow rate, and the PEEK seal should automatically seal.

◀ Figure 1-1:
The Supelco VersaFlash
High Throughput Flash
Purification System

VersaFlashTM

High Throughput Flash Purification Station

Chapter 1: Introduction

The Supelco VersaFlash System is a high throughput flash purification system (Figure 1-1) that is used for rapid separation of substances from a sample so that they can be used for further evaluation.

The complete system consists of three components (also sold separately):

- A VersaFlash Station which holds the VersaPak cartridge in place
- A pump which is used to deliver the mobile phase to perform the separation.
- A VersaVac sample loading station for loading samples onto cartridges prior to purification.

A variety of cartridges designed to be used with the unit are available from Supelco. A listing of available cartridges is provided on page 13.

VersaPak cartridges are pre-compressed, disposable cartridges containing an adsorbent or silica based bonded phase sorbent, quality-controlled to meet your stringent separation requirements. Cartridge compression reduces particle interstitial spacing and wall effects to provide even mass transfer and balanced chemical interaction. The

spherical geometry of the particles greatly reduces the possibility of particulate fines due to fracture within the cartridge bed. The result is faster separations, greater band definition and lower back-pressure.

The unique, patent pending symmetrical cartridge design leads to a variety of features that will allow you the versatility to easily complete flash separations. The versatile design means that the cartridge is bi-directional, leading to the ability to let the solvent travel through the cartridge in either direction and even reverse the flow during the separation, should you encounter a slow moving, or tightly retained compound that you would like to elute quickly and with less solvent (Rev-Elution).

The symmetrical cartridge design also allows the cartridges to be stacked one on top of the other, leading to the ability to expand length and/or combine phase chemistries for multiple, simultaneous separations.

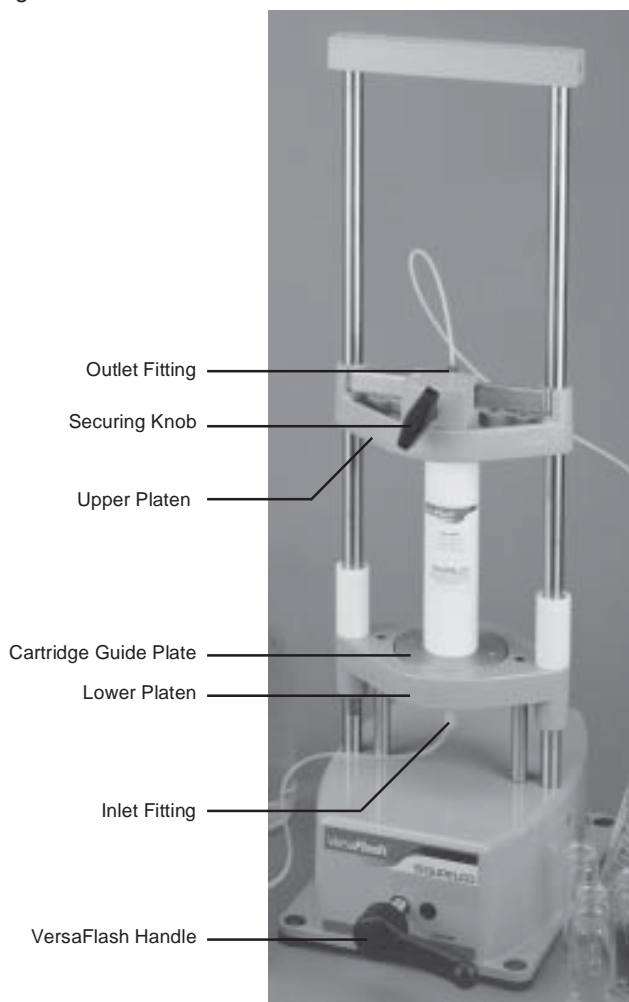
An additional advantage of the VersaPak cartridges is the way in which they seal in the VersaFlash High Throughput Flash Purification (HTFP) station. This allows users to employ all four sizes of cartridges in the same system, eliminating the need to purchase and change-out additional expensive compression barrels.

Chapter 2: Installation

2.1: Overview

This chapter describes how the system is to be installed in the laboratory and includes a short protocol to verify that it is operating properly. Once you have completed the installation procedure, the system is ready to operate. Figure 2-1 shows the key components critical to the VersaFlash Station operation.

Figure 2-1: The VersaFlash Station



2.2: Unpacking

The complete VersaFlash system is comprised of the following components (sold separately).

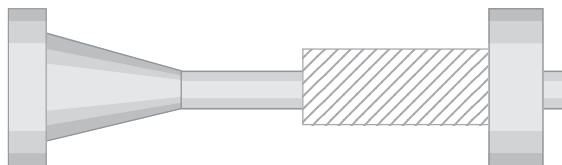
- A VersaFlash Station which holds the VersaPak cartridge in place
- A pump which is used to deliver the mobile phase to perform the separation.
- A VersaVac sample loading station for loading samples onto cartridges.

2.3: Locating the VersaFlash Station

Place the VersaFlash Station in a fume hood or a vented enclosure in a position with easy access to the handle and the Upper Platen.

Place the pump next to the VersaFlash Station so that the outlet of the pump faces the inlet tubing of the VersaFlash Station.

- a) Cut a length of tubing to be connected to the outlet of the pump. The length should be sufficient to reach the inlet of the VersaFlash Station. Use a safety razor or other sharp instrument to cut the plastic tubing. The cut end must be smooth and square.
- b) Slide the knurled Delrin nut onto the tubing.
- c) Slide the Tefzel ferrule onto the tubing with the beveled edge facing the nut. Align the outer surface of the ferrule flush with the end of the tubing.



- d) Insert the fitting into the outlet fitting of the pump. It should be finger tight so that no leaks occur.

Note: The ferrule and the nut can be reused multiple times, so long as the ferrule is not scored or scratched or the threads on the nut are not stripped. Over-tightening can result in leaks.

2.3: Locating the VersaFlash Station (cont.)

Note: If the ferrule is not placed in the correct direction as shown in c), the fitting will leak.

- e) Cut another length of tubing to be used to deliver solvent to the inlet of the pump. The length should allow for immersion to the bottom of a large flask or solvent bottle.
- f) Follow the same procedure (steps b and c) to place a fitting on one end of the tubing.
- g) Insert the fitting into the inlet fitting of the pump and tighten.
- h) Place a 10 μ m stainless steel filter (Supelco Cat. No. 59277) on the open end of the inlet tubing and place the tube with the filter into a flask containing distilled water.
- i) Place a Solvent Flush Connector (from the starter kit) into the VersaFlash Station following the procedure in section 3.2.
- j) Cut another length of tubing for the outlet of the VersaFlash Station. There should be ample length to reach the point of collection of the effluent from the VersaFlash Station.
- k) Follow the aforementioned procedure to put a fitting on the end of the tubing and connect the fitting to the outlet of the Solvent Station.
- l) Connect the loose end of the pump outlet tubing to the inlet fitting of the VersaFlash Station.

Leak Test Procedure

- m) Place the end of the outlet tubing from the VersaFlash Station into a collection flask.
- n) Turn on the pump (see pump manual). Set pump speed to 2.5 on the dial.
- o) Watch the flow path and check for leaks at all fitting connections. Tighten fittings as needed.

Note: If leaks occur at the inlet or outlet of the VersaFlash System after tightening of the fittings, re-tighten leaking fittings.

- p) When the system is free of leaks, it is ready for use.

Caution: Follow OSHA and internal safety guidelines as applied to flammable and hazardous solvents.

Chapter 3: General Operating Procedure

3.1: Overview

This chapter describes the general operating protocol for the system and includes all the information required for operating the system on a routine basis.

An overview of the system is presented in Figure 3-1.

3.2: Cartridge Loading

To load a cartridge:

Note: Be sure that the cartridge loading procedure is done with the pump off.

- a) Place the handle of the VersaFlash station in the **Idle** position and pull it out as shown in Figure 3-2, then place the VersaPak cartridge on the lower platen so that the inlet hole aligns with the PEEK seal (shown in Figure 3-3) of the lower platen.
- b) Push the handle inward causing the Lower Platen to rise. The handle is now in the Idle position as shown in Figure 3-4.

Figure 3-1: The VersaFlash High Throughput Flash System



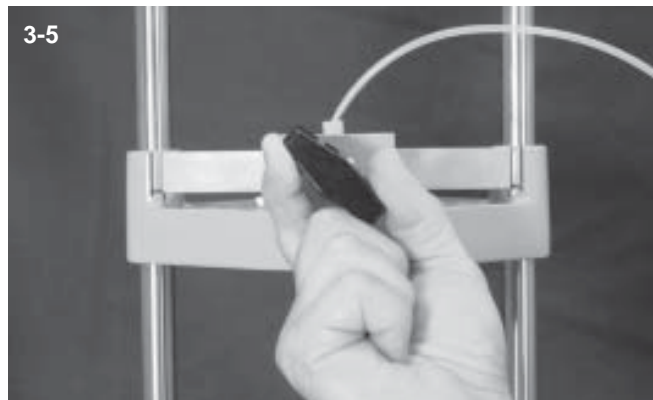
- c) Lower the Upper Platen by turning the Securing Knob (Figure 3-5) counterclockwise and guiding it downward until it securely meets with the outlet end fitting of the cartridge and the PEEK seal is aligned with the outlet hole of the cartridge. Continue to lower the Upper Platen until it rests flush with the cartridge end fitting.
- d) Turn the Securing Knob clockwise until the Upper Platen is secure and the cartridge is held tightly in place.
- e) Turn the handle clockwise until it reaches the **Operate** position and the cartridge is tightly held between the Upper and Lower platens.

Note: Do not overtighten. It is not critical that the handle turns all the way to the operate position. The patent pending design allows the Lower Platen to lock into position at any point along the clockwise rotation. How far the handle turns is dependent on the pressure applied to the Upper Platen when securing it on top of the cartridge. The more pressure applied, the less likely you will be able to turn the handle all the way to the operate position.

- f) Ensure that the bottom cartridge guide plate cannot be depressed. By using your thumb and forefingers, try to depress it on the Lower Platen as shown in Figure 3-8.

Note: It is critical that the station is set up so that the Upper and Lower Platens securely hold the cartridge end-fittings in place. The Bottom Guide Plate should be fully depressed.

- g) Turn on the pump and adjust the flow rate as necessary.



3.3: Sample Loading

There are at least five ways to load a sample onto a VersaPak Cartridge. They are:

1. Direct syringe injection onto the cartridge (off-line) – Section 3.3.1.
2. Vacuum aspiration onto the cartridge (off-line) – Section 3.3.2.
3. Using a solid sample cartridge prior to the primary cartridge (on-line) – Section 3.3.3.
4. Using a valve and loop injector for repetitive volume sample loading (on-line) – Section 3.3.4.
5. Through the pump loading for dilute samples (on-line) – Section 3.3.5.

3.3.1: Direct Syringe Injection onto the Cartridge (Off-Line)

Note: In most cases direct syringe injection is recommended prior to placing the cartridge onto the VersaFlash station.

To Perform Direct Sample Injection:

- a) Using a non threaded male Luer syringe, draw up an aliquot of sample of desired volume.
- b) Insert the male Luer of the syringe into the hole on either end of the cartridge until it fits tightly into the cartridge end-fitting.

Figure 3-9:



- c) Inject the sample into the cartridge.
- d) Put the VersaPak cartridge into the VersaFlash Station so that the sample is on the inlet side of the cartridge (i.e. bottom for upward flow, top for downward flow).

3.3.2: Vacuum Aspiration onto the Cartridge (Off-Line)

Cartridges can be loaded via the VersaVac Sample Loading Station when one or more cartridges (as many as six) are to be loaded at the same time. This method also allows the loading of samples through SPE tubes for pretreatment prior to application into the VersaPak cartridge.

The setup of the VersaVac requires a vacuum source as shown in Figure 3-10

Figure 3-10:



- a) To aspirate sample into the inlet of the cartridge, place the cartridge(s) onto the male Luer fitting(s) on the lid of the VersaVac.
- b) Place an empty syringe barrel or SPE tube on the inlet to the cartridge. Cap off the unused male Luer fittings using female Luer caps (Cat. No. 57098).
- c) To apply the sample(s), pour an aliquot of the desired volume of sample material into the empty syringe barrel or SPE tube and turn on the vacuum (Figure 3-11). Allow the sample material to empty into the cartridge(s).

Note: Slow loading under low vacuum is recommended.

- For a partial separation in the cartridge while it is on the VersaVac, follow the sample with a volume of solvent matching the starting mobile phase fluid that will be used when the cartridge is transferred to the VersaFlash Station.
- d) Once the sample is on the cartridge, remove the syringe barrel or SPE tube and transfer the cartridge to the purification station for final purification and elution of desired components.

Figure 3-11:



3.3.3: Using a Solid Sample Cartridge

If the sample material exhibits low solubility in the mobile phase to be used in the flash purification, it may be necessary to apply the sample through the use of a Solid Sample Cartridge using the following procedure:

- a) Adsorb the sample material onto a solid support media such as silica or celite. The media is then dried *in vacuo* along with the adsorbed sample material.
- b) Pour a desired volume of adsorbed media into the appropriate sized Solid Sample Cartridge.

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- c) Once the desired volume of adsorbed media is in the Solid Sample Cartridge, backfill to the fill line with non-adsorbed packing media such as celite. Periodically tap the bottom of the cartridge gently on a hard surface to settle the packing material without voids. This will help insure a uniformly packed cartridge leading to proper desorption.



- d) Place a frit (smooth side up) onto the open end of the Solid Sample Cartridge and slide the frit carefully down into the cartridge.

- e) When the frit is securely held inside the opening of the cartridge, place the inlet end-fitting over the frit so that the O-ring on the fitting is in contact with the sides of the cartridge and place in the VersaFlash station as shown in Figure 3-14.



- f) Turn the VersaFlash handle to the idle position with it pushed in. Lower the Upper Platen so that it is resting on top of the end-fitting to be pressed into the solid sample cartridge, and tighten the securing knob.

- g) Turn the handle 180 degrees to the **Operate** position. The lower platen will rise securing the top end-fitting in place. If the end-fitting does not push in completely (flush with the frit), repeat steps f) and g) until it is completely seated in the cartridge. The Solid Sample Cartridge is now fully assembled.



- h) Return the handle to the idle position and pull it out. Stack the VersaPak cartridge in line after (downstream of) the solid sample cartridge so that the sample will be eluted onto the VersaPak cartridge.

3.3.4: Using the Valve-and-Loop Accessory

When a fixed volume of sample fluid is to be repetitively applied to one or more VersaPak cartridges, the valve and loop accessory (Cat. No. 57683, 1mL) is a useful alternative to direct syringe loading. The loop enables finite volumes to be premeasured and ready for loading even while a purification is occurring on the VersaFlash purification station. (Additional sample loop volumes are available.)

3.3.4.1: Set-up

- a) Mount the valve-and-loop accessory on a ringstand close to the VersaFlash Station.
- b) Select the loop that will be used for loading the VersaPak cartridge. Standard valve-and-loop accessory injection valves in 1, 5 and 10mL volumes are available. Follow the instructions with the valve-and-loop assembly to connect the loop to the proper fittings on the valve.
- c) Connect the inlet tube of the valve to the outlet of the pump. Then connect the outlet tube of the valve to the inlet of the VersaFlash Station.

Caution: Always use mobile phase fluid that is compatible with the sample and sample solvent. Precipitation and/or emulsion can result in serious blockage and/or contamination of the flow path.

3.3.4.2: Operation

- a) With the pump off, and the valve in the LOAD position, fill the 25mL syringe, supplied with the valve-and-loop kit, and connect it to the loop loading tube. Place the overflow tube into a flask to collect the overflow sample fluid.
- b) Apply the volume of sample material to completely fill the loop.
- c) Turn the valve to the INJECT position and turn on the pump at a preset flow rate.
- d) Leave the valve in the INJECT position until the sample volume is completely moved into the VersaPak cartridge. To load another sample, return the valve to the LOAD position and repeat the procedure as described above.

3.3.5: Trace Enrichment (Through the pump sample loading)

When sample material is present in a matrix fluid at very low concentration, it is possible to concentrate the sample component on a VersaPak cartridge by passing large volumes of matrix fluid through the cartridge while trapping the sample component via interaction with the stationary phase. Often the solute is found in water sources, requiring a VersaPak C18 reversed phase cartridge for the concentration of the dilute sample component.

3.3.5.1: Set-up

Caution: Be sure that all fluid is filtered through a fine mesh non-adsorptive filter prior to entering the pump. Fine particles in the matrix fluid can damage the internal mechanism of many pumping devices.

- a) After the matrix fluid has been filtered, use a 2 liter flask or bottle to contain the fluid. Place a stainless steel Inlet Filter (Cat. No. 59277) on the inlet tube to the pump and place the filter at the bottom of the sample matrix vessel.
- b) On the outlet end of the VersaFlash Station, place a large vessel (>3 liters) for the collection of effluent.
- c) Select a VersaPak cartridge of the appropriate size and selectivity. Place the cartridge in the VersaFlash Station and set in the **OPERATE** position.
- d) Set the flow rate of the pump and start the pump.
- e) Allow enough time for the trace sample component to adsorb onto the packing media.
- f) Reverse the VersaPak Cartridge (Rev-Elution) and switch over to a stronger reversed phase mobile phase (e.g.: water:MeOH), and elute the sample component of interest.

3.4: Rev-Elution

Rev-Elution is a powerful technique to reverse the flow through a VersaPak cartridge by revolving the cartridge 180 degrees. Rev-Elution is used to elute fully retained or slow moving substances without the need to travel the entire length of the cartridge before eluting. This technique reduces band spreading, reduces contamination of desired substances and conserves solvent.

Rev-Elution is performed as follows.

- a) Following the elution of non-retained and fast moving substances, stop the flow by turning off the pump.
- b) Remove the cartridge from the VersaFlash Station.
- c) Return the cartridge to the VersaFlash Station by turning it 180 degrees from the original position. (The retained substance is now on the outlet end of the cartridge.)
- d) Turn the pump on and collect the eluting substance.

Chapter 4: Maintenance and Troubleshooting

4.1: Maintenance

The VersaFlash Station is designed to minimize the effort required for separation.

Before using the station:

- Check the station for the presence of particulate matter. If particulate matter is observed, clean the station and remove any particulate matter that may have deposited on the outside.
- Check the station for leaks. If a leak is observed, gently tighten the offending fitting (do not overtighten).
- Replace the cartridge (if necessary)

After finishing a series of separations:

- Remove the eluting solvent by rinsing with two to three volumes of water. If the solvent used is not miscible with water, use an intermediate solvent such as isopropanol first.
- On a periodic basis it may be necessary to perform maintenance on the pump (e.g. replace brushes, gears, seals) as described in the manual supplied with the pump.
- The threads on the Upper Platen Securing Knob should be lubricated with a lithium bearing grease approximately every 500 cycles to insure smooth operation.

4.2: Troubleshooting

- **Improper seating of the cartridges in the PEEK seal assembly:** Turn the handle to the idle position and pull out. Adjust the cartridge back and forth while pushing the handle in, so that the cartridge seals properly in the PEEK Seal Assembly.

VersaFlash handle does not turn all the way to the operate position: It is not critical that the handle turns all the way to the operate position. The patent pending design allows the Lower Platen to lock into position at any point along the clockwise rotation. How far the handle turns is dependent on the pressure applied to the Upper Platen when securing it on top of the cartridge. The more pressure applied, the less likely you will be able to turn the handle all the way to the operate position.

Specifications

VersaFlash High Throughput Flash Purification Station

Height: 27.5 in.

Width: 8 in.

Length: 11 in.

Weight: 27 lbs.

Systems, Cartridges, Kits and Accessories

Systems

Item	Cat. No.
VersaFlash System I (VersaFlash Station, Pump, VersaVac, Starter Kit)	97730-U
VersaFlash System II (VersaFlash Station, VersaVac, Starter Kit)	97731-U
VersaFlash Station	97732-U

Silica Cartridges

Size	Qty.	Cat. No.
40 x 75mm	12	97704-U
	96	97705-U
40 x 150mm	6	97706-U
	48	97707-U
80 x 150mm	2	97708-U
	12	97709-U
80 x 300mm	1	97710-U
	6	97711-U

C18 Cartridges

Size	Qty.	Cat. No.
40 x 75mm	2	97700-U
40 x 150mm	1	97701-U
80 x 150mm	1	97702-U
80 x 300mm	1	97703-U

Pumps

Item	Qty.	Cat. No.
Assembled Pump, 120V	1	97734-U
Assembled Pump, 220V	1	97737-U

Kits & Accessories

Item	Cat. No.
Starter Kit	97739-U
Solid Sample Cartridge Kit	97738-U
Cartridge Stacking Assembly (40 x 40mm)	97740-U
Cartridge Stacking Assembly (40 x 80mm)	97741-U
Cartridge Stacking Assembly (80 x 80mm)	97742-U
Solvent Flush Connector	97743-U
VersaVac Sample Loading Station	97750-U

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