DryVap[™] Users Guide

Navigational Tips

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Navigational Tips

Sections (Chapters) are indicated by a boxed in number in the upper left corner. Clicking this object when a number is inside it will link back to the beginning of the section

BUTTONS

Navigating through this manual is through hyperlinked buttons and hyperlinked text.

Example of a button:

1-2 Product Safety Notice

GENERAL NAVIGATION

3 Ways to go to next page

- 1. Depressing the space bar.
- 2. Left-mouse click anywhere on a page where a button is not present.
- 3. Move scroll button of mouse down.

• 2 Ways to go to previous page

- 1. Press the letter "P".
- 2. Move scroll button of mouse up.

• Go to Home (first page)

Clicking on the Horizon icon in the upper right corner.

• Returning to last viewed page.

Clicking on the button to the left of the Horizon icon (upper right hand corner) returns to the previously viewed page.

Section footer

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To Go to a Specific Page

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Section 0: Navigational Tips



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1-9 Chemical Compatibility with Sulfinert Coatings



| Certification Mark: | | CE |
|-------------------------|---|---------------------------------|
| Product: | Evaporation equi Evaporator/Conc | pment entrator System |
| Model(s): | DryVap Model 50 | 00 |
| Parameters: | Rated Input Voltage: Rated Frequency: Rated Input Current: | 120-240 V AC 47-63 Hz 5 A |
| Tested according to: | CAN/CSA -C22.2 No.610 UL 61010-1:2004 EN 61010-1:2001 IEC 61010-2-010:2005 | 10-1-04 |

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Symbols used in this manual

Indicates a caution concerning operations that may lead to injury to persons or damage to property if not performed correctly. In order to use the instrument safely, always pay attention to these cautions and read the manual.



NOTICE

Indicates notes for operation or additional explanations. Reading these is recommended.

This Safety Notice has been provided to inform and guide the operator in safe practices to avoid injury, and is intended to cover general safety requirements for a laboratory. Each laboratory is responsible for implementing and communicating its unique safety requirements and program to all workers.

If you have any questions please call Horizon Technology at 1-800-997-2997 (inside NH call 603-893-3663)

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• Eye protection in the form of safety glasses or goggles is mandatory when operating the DryVap® Concentrator System. If solvent liquids or vapors come into contact with the eyes, follow the appropriate first aid procedures set forth in the laboratory's safety manual.

•Lab coats must be provided for protection and convenience. They should be worn at all times when operating the DryVap Concentrator System.

Protection of the hands when working with solvents or any hazardous material is essential. Wear gloves selected on the basis of the hazard.
If solvents or other chemicals come into contact with the skin, follow the appropriate first aid procedures set forth in the laboratory's safety manual.







Equipment Safety 1 of 2

•The DryVap® Concentrator System is designed to dry and evaporate solvents. It has been tested and approved for use with the following solvents; <u>acetone</u>, <u>acetonitrile</u>, <u>ethyl acetate</u>, <u>hexane</u>, <u>methanol</u>, <u>methylene chloride</u>, <u>MTBE</u>, <u>and</u> <u>petroleum ether</u>. Using the DryVap Concentrator System in a manner other than what is specified by Horizon Technology, Inc. can result in personal injury or compromise the performance of the equipment and quality of data. Please contact Horizon Technology, Inc., before using different solvents, or solvent mixtures.

•The DryVap Concentrator System is designed for bench top operation. All solvent vapors are removed via vacuum. The vacuum pump must be exhausted into a fume hood to avoid the discharge of potentially toxic vapors and fumes into the laboratory atmosphere. The equipment must be set-up and operated in a well-ventilated area. The system can also be operated in a fume hood.

•Because solvent vapors are removed from the DryVap Concentrator System via the vacuum line, it is recommended to place a liquid trap between the DryVap Concentrator System and the vacuum source. This will minimize the total amount of solvent being exhausted through the vacuum pump, and will allow collected solvents to be recycled. For optimum solvent recovery, use the Horizon Reclaimer[™] Solvent Recovery system.

•Only a diaphragm vacuum pump, or a vacuum pump specifically designed to handle high levels of solvent vapors should be used. Rotary vane vacuum pumps should not be used.



•If flammable solvents like hexane are to be evaporated, a vacuum pump with an explosion-proof motor is required.

•Do not work with volatile solvents without adequate ventilation from a chemical fume hood, or other protective devices.



•Only the concentrator tubes supplied with the DryVap Concentrator System should be used. These have been specifically dimensioned to work with the critical functions of both the heater and optical sensors. The tubes are under vacuum when the equipment is in operation. The use of any other concentrator tubes can pose a risk of implosion.

•Examine each concentrator tube for cracks, scratches, or any damage prior to use. Do not use any tube that appears to have some damage, as this can cause an implosion.



•<u>The DryVap Concentrator System immersion</u> heaters get very hot during operation. (Fig.1)



•The concentrator tubes and Lid Assemblies can get hot during the concentration phase <u>if</u> <u>high boiling solvents are used.</u> (Fig.2)

•The DryVap Concentrator System must be properly grounded for safe operation. Only use a power cord with a ground plug and connect it to a grounded outlet.

•<u>Disconnect the power cord before working on</u> the unit.



Chemical Safety

Section 1: Product Safety Notice an Certification

•The DryVap® Concentrator System is used with organic solvents that can pose inhalation, skin, and ingestion hazards with potential chronic health effects. Some of the solvents may also be flammable, which could cause fire and/or explosion hazards. Chlorinated solvents are, in general, not flammable, while non-chlorinated solvents are often flammable. However, chlorinated solvents do decompose when burned, resulting in high concentrations of toxic vapors. All solvents must be handled using appropriate personal protection equipment and in a properly operating fume hood to eliminate inhalation hazards. For handling and safety instructions refer to the **Material Safety Data Sheet (MSDS)** for the specific chemical. Refer to OSHA Standard 29 CFR, Part 1910.1052 for occupational exposure to methylene chloride.

•MSDS (Material Safety Data Sheets) are the source for chemical hazard information including basic information on the manufacturer or distributor, identification of the chemical, the product's hazardous ingredients, physical data, fire and explosion data, toxicity information, protection information, and more. The laboratory is responsible for having a MSDS for every chemical or substance being used. It is also the laboratory's responsibility to make the MSDS available and accessible to all employees and to provide training in the safe handling of hazardous chemicals. MSDS can be obtained from the vendor.

•All hazardous solvents and chemicals must be disposed in accordance with appropriate Federal, State and local regulations.

•The DryVap Concentrator System requires the use of a pressurized gas source for the sparge gas. The recommended source is a <u>zero grade (ultra low hydrocarbon)</u> <u>nitrogen gas.</u> The operator must be aware of the potential hazards of reactivity and toxicity, as well as asphyxiation, of even the referred to "harmless" gases, such as nitrogen. The large amount of potential energy resulting from compression of the gas makes the cylinder a potential rocket or fragmentation bomb if dropped. To prevent this, secure the gas cylinder to avoid tipping.







(Zero Grade)



| | | Boiling | Flash | Auto Ignition | Flammable limits | • | |
|-----------|-------------------------|----------|---------|------------------|---------------------|-------|---|
| | | Point | Point | Temp | in air by vo | ol. | |
| CAS# | Solvent | (deg C) | (deg C) | (deg C) | LEL | UEL | MSDS Source |
| 75-09-2 | Methylene Chloride | 39.8 | none | 556 | 12.0% | 23.0% | http://www.jtbaker.com/msds/englishhtml/M4420.htm |
| 1634-04-4 | Methyl tert-butyl ether | 55.2 | -27 | 435 | 1.6% | 8.4% | http://www.jtbaker.com/msds/englishhtml/B7222.htm |
| 67-64-1 | Acetone | 56.5 | -20 | 465 | 2.6% | 12.8% | http://www.jtbaker.com/msds/englishhtml/A0446.htm |
| 67-56-1 | Methanol | 64.5 | 11 | 464 | 6.7% | 36.6% | http://www.jtbaker.com/msds/englishhtml/M2015.htm |
| 110-54-3 | Hexane | 68.0 | -23.3 | 234 | 1.1% | 7.5% | http://www.jtbaker.com/msds/englishhtml/H2379.htm |
| 8032-32-4 | Petroleum Ether | 20 to 75 | -18 | 288 | 1.10% | 5.9% | http://www.jtbaker.com/msds/englishhtml/P1696.htm |
| 141-78-6 | Ethyl acetate | 77.0 | -4 | 426 | 2.0% | 11.5% | http://www.jtbaker.com/msds/englishhtml/E2850.htm |
| 75-05-8 | Acetonitrile | 81.6 | 2 | 524 | 4.4% | 16.0% | http://www.jtbaker.com/msds/englishhtml/A0518.htm |
| 108-88-3 | Toluene | 110.6 | 7 | 422 | 1.1% | 7.1% | http://www.jtbaker.com/msds/englishhtml/T3913.htm |

| | | NFPA 704 Code | | | |
|-----------|-------------------------|---------------|-------------|------------|---------|
| | | Health | Flamability | Reactivity | Special |
| CAS# | Solvent | | | | |
| 75-09-2 | Methylene Chloride | 2 | 1 | 0 | |
| 1634-04-4 | Methyl tert-butyl ether | 1 | 3 | 0 | |
| 67-64-1 | Acetone | 1 | 3 | 0 | |
| 67-56-1 | Methanol | 1 | 3 | 0 | |
| 110-54-3 | Hexane | 1 | 3 | 0 | |
| 8032-32-4 | Petroleum Ether | 2 | 4 | 1 | |
| 141-78-6 | Ethyl acetate | 1 | 3 | 0 | |
| 75-05-8 | Acetonitrile | 2 | 3 | 0 | |
| 108-88-3 | Toluene | 2 | 3 | 0 | |



NFPA = National Fire Prevention Association http://www.nfpa.org/index.asp



Notice: Data obtained from J.T. Baker MSDS site links listed above for reference only. For definitive information about the chemicals used in your laboratory, refer to the MSDS data sheets provided by the supplier of your chemicals and solvents.

Max. Contact Time with Glass For Selected Solvent BP Temperatures





BP for solvents under 12 and 15 inches Hg vacuum were calculated using the Antoine Vapor-Pressure equation and Table 5.9 (Vapor pressures of various organic compounds) p5.39 Lange's Handbook of Chemistry 14th ed.

Notice: Use RoSPA information for reference only. Observe your Federal, State, or Local Laws for safety. Wear protective gloves when handling high boiling solvents.

Data from RoSPA (link below)

http://www.rospa.com/productsafety/articles/temperatures.htm

1



Sulfinert® Treated Metal Surfaces

The station lid, immersion heater, sparge tube, and rinse tube are coated With a Restek performance coating called Sulfinert®. The chart to the right shows the chemical compatibility of the coating extracted from their literature. Sulfinert is a vapor deposited silicon and will be degraded if exposed to pH's greater than 7 or with reactive acids such as HF.

NOTE AND CAUTION: Although, Sulfinert is listed as "good" with strong acids and brines, any scratch or damage to the coating will expose the underlying metal to the corrosives. <u>Do not attempt to concentrate</u> <u>acidic digests (aqueous solutions) with DryVap as the components for</u> <u>solvent reclamation (e.g. vacuum pump , regulators, and Reclaimers are</u> <u>not compatible with these solutions).</u>

If there is a question on the DryVap system chemical compatibility with solvents or solutions, contact Horizon Technology for further information.

| Compatibility with Sulfinert® Coating | | | | |
|---------------------------------------|-----------|------|------|--|
| | Excellent | Good | Poor | |
| Aldehydes | | | | |
| Acetic Acid | | | | |
| Acetone | | | | |
| Alcohol | | | | |
| Amines | | | | |
| Ammonium Hydroxide | | | | |
| Arsenic | | | | |
| Aromatics | | | | |
| Benzene | | | | |
| Brine | | | | |
| Carbon Dioxide | | | | |
| Carbon Disulfide | | | | |
| Dimethyl Disulfide | | | | |
| Dimethyl Sulfide | | | | |
| Ethyl Mercaptan | | | | |
| Fatty Acids | | | | |
| Ferric Chloride | | | | |
| Formaldehyde | | | | |
| Hydrocarbons | | | | |
| Hydrochloric Acid | | | | |
| Hydrofluoric Acid | | | | |
| Hydrogen Peroxide | | | | |
| Hydrogen Sulfide | | | | |
| Ketones | | | | |
| Mercury | | | | |
| Mercury Oxides | | | | |
| Methyl Mercaptan | | | | |
| Nitric Acid | | | | |
| Phosphoric Acid | | | | |
| Potassium Hydroxide | | | | |
| Extracted from Restek Literature | | | | |

Introduction 1 of 2



INTRODUCTION

The DryVap[®] Concentrator System is designed to automatically remove water (dry) and concentrate samples through evaporation of the extraction solvent, for GC, GC/MS, and HPLC/MS analysis. Organic solvents from SPE (solid phase extraction) or LLE (liquid-liquid extraction) are automatically dried of any residual water using Horizon's DryDisk[™] membranes, and then concentrated to volume using a combination of heat, vacuum, and sparge gas. The DryVap Concentrator System provides bench top operation capable of handling up to 6 samples simultaneously with each station operating independently. Sample volumes can range from 2 ml to 200 ml. The easy to use DryVap Concentrator System sets new standards for rapid solvent drying and concentration, and reproducibility of results.

The DryVap Concentrator System allows testing facilities to:

- · Save time and provide an increased level of productivity
- Improve recoveries
- Improve reproducibility
- · Improve operator safety by reducing exposure to solvents

The DryVap's Master Control Panel is used to set the following drying and concentration parameters for the individual stations; *Dry Volume, Heater Power*, and *Heater Timer*. The Master Control Panel has an additional feature for setting the *Auto Rinse Mode* parameter. This feature allows the user the option to set an automatic rinse that will wash the concentrator tubes with solvent to maximize recoveries. Each station is also equipped with a Station Control Panel for operating the individual stations. The Station Control Panel includes the following features; *Start, Stop, Pause/Resume, and Manual Rinse* keys, along with a set of LED lights to indicate the current status for each station.

Introduction 2 of 2

2



The following are features of the DryVap Concentrator System:

• Automatic in-line solvent drying with DryDisk membranes: Uses 65 mm membranes with 225 mL DryDisk Assembly. This process replaces sodium sulfate drying.

• Six (6) programmable Stations: Each position operates independently providing versatility with a wide range of solutions. Six samples can be handled simultaneously.

• Utilizes a combination of heat, vacuum, and sparge gas, to control the concentration process: Each parameter can be adjusted independently.

• Automatic heater shutoff: Heaters turn off when the solvent level drops below an internal thermocouple sensor.

• **Optical liquid sensors for endpoint detection**: Concentration process stops when the solvent meniscus reaches the optical sensor. Final vol. is dependent on glassware selection.

• Individual Immersion Heating Elements: Provides heat directly to the sample; no water bath or hot block.

• Variable timer for ending the concentration process: Allows dirty and opaque samples to be processed.

• Individually sealed concentrator tubes: No cross contamination or loss of analytes.

• Preserves samples under inert gas with no heat when finished: Sample can be left in tube for longer periods without requiring immediate attention.

• Four selectable solvent dry times: Handles volumes of 0, 20, 100, and 200 mL samples.

- Individual front panel status displays: Identifies the current status of each station.
- Easy viewing of evaporation process to endpoint.
- Designed for bench top operation: All solvent vapors are removed via vacuum and vented to a fume hood.
- Optional DryVap Software: Used for monitoring and programming methods.

• Optional Direct-to-Vial Evaporation tubes: Eliminates manual sample transfer to GC vials.

Section 2: Introduction





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- 3-2 Liquid Trap Components
- 3-3 Master Control Panel
- 3-6 Station Control Panel
- 3-7 Station Lid Assembly
- 3-8 EV Tubes and Cradle Assembly
- 3-9 DryDisk Assy. and DryVap Controls
- 3-10 DryVap Demonstration (Animated)



Use Figures 3-1 through 3-10 to identify the major components of the DryVap® Concentrator System.



Figure 3-2: Base of DryVap System

3



Use Figures 3-1 through 3-10 to identify the major components of the DryVap[®] Concentrator System.



Figure 3-3: Liquid Trap Assembly

3. 1 Master Control Panel:

3

The Master Control Panel is used to set the parameters for each of the 6 stations. Refer to Figure 3-4a. In order to provide the most versatility, when an individual station is started, the parameters are read from the Master Control Panel, and stored in the individual station memory. This means that different parameters can be selected for each station, and run independently. Simply dial in the new parameters and press *Start*.

DRY VOLUME: The volume of solvent extract (20, 100, or 200 ml) to be dried using the DryDiskTM membrane. If the solvent extract will not be dried prior to concentration, the *Dry Volume* is set to 0.

HEATER POWER: Heat is used for the concentration of the sample extract. During the heating stage, the heater cycles between the on and off states.

NOTE: When heat is being applied, Heat Power 5 is the preferred setting as the solvent is brought to the BP under vacuum the quickest and evaporates the sample fastest without degradation. The BP under vacuum is the temperature limit for all the power settings.)

Off: Heater is not used and only sparge gas and vacuum are applied to the sample. (There are additional features available with this setting such as sparge heat which is accessible through software commands. Refer to section 5-2 for more information.)

- 1: Heater is on 40 % of the time (24W)
- 2: Heater is on 55 % of the time (33W)
- 3: Heater is on 70 % of the time (42W)
- 4: Heater is on 85 % of the time (51W)
- 5: Heater is on 100 % of the time (60W)



Fig. 3-4a Master Control Panel

U 🔶 3-4

3. 1 Master Control Panel (Continued)

HEATER TIMER: When using settings 1-5, the heater is turned off by time. When the time expires, the run is finished. This feature is activated only when >1 mL endpoint volumes are desired. The reasons for this can be specifications of a method, samples being opaque making them incompatible with optical liquid level sensor endpoints, or samples that cannot be brought down to 1 mL or less in volume.

NOTICE: Heat Timer must be OFF to allow optical liquid level endpoints. The heater turns off by an embedded thermocouple sensor inside the immersion heater. The heater automatically turns off when the liquid level reaches the top of the coils.

The default factory time settings are listed below, custom timer values can be achieved through the optional software application DryVap Programmer:

Off = Heater turns off automatically by Thermocouple sensor Heater Timer 1 = 5 minutes Heater Timer 2 = 10 minutes Heater Timer 3 = 15 minutes Heater Timer 4 = 20 minutes Heater Timer 5 = 25 minutes

Refer to section 6 for selection various endpoint volumes methods.



Fig. 3-4b Master Control Panel

3.1 Master Control Panel (Continued)

AUTO RINSE MODE: Settings for automatic rinse of Lid Assembly and concentrator tubes;

Off: No rinse.

1: Rinses once when the solvent level in the concentrator tube reaches the liquid level sensor (0.4 or 0.9 mL depending on EV tube selection). The vacuum and sparge continues the concentration process until the solvent level again reaches the optical liquid level sensor, then the system goes to the *Finish* state and shuts off.

2: Rinses once after the thermocouple sensor reading shuts off the heater. The vacuum and sparge continue the concentration process until the solvent level reaches the optical liquid level sensor (0.4 or 0.9 mL depending on EV tube selection). The station then goes to the *Finish* state and shuts down.

3: A combination of modes "1" and "2". Rinses once after the heater shuts off and a second time when the solvent level reaches the optical liquid level sensor (0.4 or 0.9 mL depending on EV tube selection). Vacuum and sparge continues until the solvent level again reaches the liquid level sensor, then the station goes to *Finish* and shuts off.

4: This rinse mode is used when using a **Heater Timer** setting. This mode rinses once when the heater shuts off. The concentration process ends after the rinse and the station goes to the **Finish** state and shuts off. The station **does not** continue vacuum and sparge to the optical liquid level sensor.

5: Rinses once when the solvent level in the concentrator tube reaches the optical liquid level sensor (0.4 or 0.9 mL depending on EV tube selection). The station then goes to the *Finish* state and shuts off. The station *does not* continue vacuum and sparge back to the liquid level sensor.

POWER LED: The Power LED indicates that the system is on. If the LED flashes, it indicates that one or more stations in the system are not connected.



Fig. 3-4c Master Control Panel

3.2 Station Control Panel

The Station Control Panel is how the individual stations are Started, Stopped, Paused / Resumed, and manually rinsed. Each station has LED's which display the current status of each station. Refer to Figure 3-6.

•Start: Begins the drying/concentration process for the station.

[*Helpful hint:* When the station is in the heat state (Heat, Vacuum and sparge LEDs are illuminated), pressing the Start button at this time will terminate the heat state and move the sample processing to the sparge/vacuum state.]

•Stop: Aborts the process for the station.

•*Pause / Resume*: The *Pause* key interrupts the process for the station and the *Resume* key resumes the process from where it was interrupted.

NOTICE: It is recommended <u>not to pause during the heating</u> <u>process</u> when heat timer is off and the thermocouples sensor is used for turning the heater off as the heater may not turn off properly. In this situation stop the run and restart (set Dry vol. to zero to have the station return to heat state quicker).

•*Manual Rinse*: Allows the operator to manually actuate the Rinse Solvent feature when desired. A concentrator tube must be in position, the vacuum source on, and the Rinse Solvent Bottle filled with the desired solvent. Pressing the *Manual Rinse* key will activate the rinse solvent function for 2 seconds. The key can be pressed as many times as desired to rinse the assembly.









3.3 Lid Assembly

3

The Lid Assembly makes a seal on the concentrator tube and incorporates the heater and three components. Refer to Figure 3-7.

•*Lid Assembly:* The Lid Assembly is the circular Stainless Steel body which holds the heater, the Sparge Gas Tube, and the Rinse Solvent Tube. This part has been coated with Sulfinert[®] for maximum inertness.

•*Gasket Seal:* The Gasket Seal is a two part construction; a silicone gasket, surrounded by a PTFE collar. The PTFE collar covers three sides of the silicone gasket and provides an inert surface for the concentrator tube to seal on, while the silicone gasket provides a soft, flexible surface, which the PTFE collar can compress against. The Gasket Seal can be removed from the lid assembly for cleaning or replacement.

•Sparge Gas Tube: The Sparge Gas Tube serves two purposes; one, to deliver the sample into the concentrator tube, when the DryDisk[™] feature is used, and two, to introduce the sparge gas into the concentrator tube, during the concentration process. The sparge gas is controlled via the gas pressure regulator on the left, front side of the system. The Sparge Gas Tube has been coated with Sulfinert[®] to provide maximum inertness.

•*Heater*: A 60-Watt heater is used in the concentration process. The heater automatically turns off when the solvent level drops below the internal thermocouple located just above the 90 degree bend of the heater. When the heater shuts off, approximately 6.5 ml of sample will be left. If the *Heater Timer* function is used, the heater will automatically turn off based on the timer setting. This part has been coated with Sulfinert[®] to provide maximum inertness.

•*Rinse Solvent Tube*: The rinse solvent is introduced through this tube to wash the Lid Assembly and the concentrator tube walls. This part has been coated with Sulfinert[®] to provide maximum inertness.

•*Vacuum Port*: The opening in the Lid Assembly which allows vacuum to enter the concentrator tube and remove the solvent vapors.





Section 3: DryVap System Overview

DryVap System Overview



3.4 Evaporation Tubes:

3

• The Evaporation tubes hold a maximum of 200 mL. Depending on your application, there are different tips for the tubes, 1 mL tip, 0.5 mL tip, no tip and direct to 1.5 mL GC vial tips. (Refer to the parts list section for part numbers , more specific details and required accessories



3.5 Cradle Assembly:

The Cradle Assembly is made up of the Cradle and the Cradle Basin. See the description below for information on these parts.

•*The Cradle*: The Cradle is the white plastic piece, which holds the concentrator tube in proper alignment under the Lid Assembly. It positions the tube the proper distance from the optical liquid level and tube sensors to operate properly.

•*Cradle Basin*: The Cradle Basin is the lower metal assembly beneath the Cradle. It houses two sensors; one optical set for determining the liquid level, and one mechanical set for determining the presence of the tube. The Cradle Basin contains three spring-loaded feet, to allow the Basin to "float" and ensure an optimal seal between the Lid Assembly and the lip of the concentrator tube.

•*Liquid Level Sensor*. The optical liquid level sensor is located just below the white Cradle. The sensor is positioned to stop the concentration process at 0.9 or 0.4 mL depending on the EV tube used.

•*Tube Sensor*. The mechanical tube sensor is located in the Cradle Basin. This sensor is used to confirm the presence of a concentrator tube. Only when a tube is in position, will a station advance to the *Ready* state. If a tube is removed when the station is idle, the *Ready* LED will turn off and a run will not be permitted. If a tube is removed while a station is running, the station will abort.





3.6 DryDisk[™] Device:

3

•225 ml DryDisk Assembly: The 225 ml DryDisk Assembly consists of the DryDisk Basin, a Support Screen, the 225 ml Glass Reservoir, and the Locking Ring. The 225 ml DryDisk Assembly fits into a Luer fitting, located on the platform area in front of each station. Use the 225 ml DryDisk Assembly for sample volumes greater than 20 ml, but less than 200 ml.

3.7 Beeper Volume Control:

When a Station Control Panel advances to the *Finish* state, the *Finish* LED will flash and a beeper will sound. The volume of the beeper is controlled by the volume knob located on the back side of the unit. The volume can also be turned completely off if desired. The beeper will continue to sound until the concentrator tube is removed from the cradle, or when the *Stop* key is pressed.

3.8 Communication Port:

The Communication Port allows software parameter changes to be made to each individual Station Control Panel. Please consult Horizon Technology, Inc. for instructions.

3.9 Main Power Switch:

The main power switch is housed in an assembly which connects the power cord to the power outlet. This assembly also contains the main fuse.



Figure 3-10: Back of DryVap System



3.10 DryVap Demonstration

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Click the link to the PowerPoint Show file shown below. This 11 page file shows how DryVap processes a 200 mL sample with DryDisk. (If the link is broken, go to the DryVap Demonstrations folder on the User's Guide CDROM and launch the "DryVap Demo Module.pps".

Each slide of the 11 slides runs through a sequence of animations. When the animations for the particular slide has finished, the navigation button console found in the upper right corner will move in a oval pattern notifying the viewer to click to the next slide.

This series of slides illustrate how DryVap[™] works.





SAMPLE:

200 mL extracted sample consisting of DCM with some residual water.

OBJECTIVE:

Remove water and concentrate sample down to 0.9 mL.

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Operation Theory 1 of 2

4.1 DryVap[®] Concentrator System: Theory of Operation

The DryVap[®] Concentrator System is designed to automatically remove residual water (dry) and concentrate solvent extracts for GC, GC/MS, and HPLC/MS applications. In addition, the DryVap system can remove all solvent from the sample for applications requiring recovery of dissolved material. The DryVap System is unique in that it combines the condensation principle of the Kuderna-Danish, and the gentle blow down technique of nitrogen evaporators; all in one package, and is completely automated. This combination allows for fast sample concentration rates, with very high recoveries. The following describes the three major phases of the DryVap System operation.

Drying:

Solvent extracts can be dried (residual water removed) prior to the concentration step by pouring the solvent into a DryDisk™ device. The DryDisk device is a 225 ml glass reservoir holding a 65 mm DryDisk membrane. Vacuum pulls the solvent through the DryDisk membrane and transfers the dried solvent to the concentrator tube. The residual water remains in the DryDisk device, unable to penetrate the membrane. The drying step is an option, and can be ignored if desired. The drying function is ignored by setting the Dry Volume to 0. In order to ensure that residual water is not pulled through the DryDisk membrane, the vacuum delivered to the DryVap System must be maintained at no higher than 15" Hg. Lower vacuum settings of -12" Hg is recommended for applications where light-end recovery is important. Vacuum regulator settings lower than 12" Hg may not completely transfer sample contents from DryDisk assy. into the concentrator tube.

In order to minimize any sample being left in the transfer line from

the DryDisk device to the evaporation tube, nitrogen gas is introduced through energizing a 2-position Teflon valve upon which the DryDisk device is connected to. The gas sweeps all remaining traces of the sample directly into the concentrator tube. To ensure complete transfer and to minimize sample carry-over, it is recommended to use a small volume (2 - 4 ml) of fresh solvent to flush the transfer line. This is added just before the completion of the **Dry Volume** timer.

Concentration:

The DryVap System uses an internal heater vs. an external water bath, or hot block. By directly heating the solvent, applying vacuum, and simultaneously keeping the concentrator tube sealed during the process, solvent condensation occurs inside of the tube. This condensation is beneficial in retaining the analytes, while the solvent evaporates. In addition, the vacuum lowers the boiling point of the solvent, which allows the evaporation process to occur at lower temperatures. The ability to operate at lower temperatures also increases analyte recoveries.

The concentration is accomplished using a combination of heat, sparge gas (nitrogen), and vacuum. The heating element is in direct contact with the solvent (sample). Heating the solvent directly is much more efficient than trying to heat the solvent from an external source. In addition, by turning the heater off, while sample is still left in the tube, ensures better recoveries of the more volatile compounds. The samples when concentrated go to the *Finish* state and can remain in the tubes unattended, as the heat, sparge and vacuum are off and the sample is covered with nitrogen without any adverse impact on the recovery values.

Another unique feature is the sparge gas, which is introduced into the sample. Introducing the sparge gas into the sample helps to keep the sample mixed, but more importantly, when the solvent level drops below the tip of the sparge gas tube (approximately 7 mL of sample left), the gas flow is now directed gently onto the surface of the solvent. (continued)

U 🔶 4-1

Operation Theory 2 of 2

U 🔶 4-2

This operation closely mimics a standard nitrogen blow down technique for optimal recoveries, but by incorporating this technique into the DryVap System, the process is now fast and completely automated.

The heater is turned off automatically using algorithm that uses information obtained from an internal thermocouple embedded in the immersion heater. The thermocouple mode of heater control is activated when **Heater Timer** is (set to "OFF"). The heater remains on until the internal thermocouple senses that the solvent level has dropped to the heating element; at this point the heater is turned off. (**NOTICE**: The minimum volume of sample for the thermocouple sensor to work properly is 15 mL). The vacuum and sparge gas continue to concentrate the sample to the optical endpoint (0.9 ml or 0.4 mL, depending on the EV tube used). When the optical liquid sensor is tripped, the system goes to *Finish*. At the completion of the run, an audible beeper sounds, and continues until the operator removes the concentrator tube from the station, or presses the *Stop* key.

If the *Heater Timer* is used, the heat power will shut off when the timer expires. At this point, the system goes to the *Finish* state and shuts off. (*Heater Timer* is selected when non-optical endpoints are desired. Heater Timer requires specific starting volumes of sample and knowledge of evaporation rates.)

Manual Rinse and Auto Rinse Mode:

There are two ways the rinse function can be activated; manually and automatically. The manual rinse can be activated from each Station Control Panel, but only when a concentrator tube is in position and the Lid Assembly is properly sealing a concentrator tube. Vacuum is used to pull the rinse solvent into the tube. The manual rinse is designed to help wash down the internal parts of the station and improve recoveries. The default manual rinse time is 2 seconds and can be modified using HyperTerminal or DryVap software for parameter T34 for any particular station. The *Auto Rinse Mode* allows various automatic rinses to occur during various stages of the concentration process. The duration of each rinse is defaulted to 2 seconds. The duration time can be modified using HyperTerminal or DryVap on parameter T35 for any particular station.

Auto-Rinse Modes:

Off = No rinse.

- 1 = Rinse once when the sample reaches the optical liquid level sensor, and continue with vacuum and sparge gas until the liquid level again reaches the liquid level sensor. The run is complete and the station goes to *Finish*.
- 2 = Rinse once when the heater shuts off, and continue the vacuum and sparge gas until reaching the optical liquid level sensor. The run is complete and the station goes to *Finish*.
- **3** = A combination of modes "1" and "2". Rinses once after the heater shuts off, and a second time when the optical liquid level is reached. Vacuum and sparge gas continue until the liquid level again reaches the optical liquid level sensor. The run is complete and the station goes to *Finish*.
- 4 = This mode is used with the *Heater Timer* function. This mode rinses once when the heater is shut off by the timer. The run is complete and the station goes to *Finish*. The concentration *does not* continue vacuum and sparge gas to the liquid level sensor.
- **5** = Rinses once when the solvent level in the evaporation tube reaches the liquid level sensor. The run is complete and the station goes to *Finish*. The concentration **does not** continue to liquid level sensor again.

Refer to Sections 5-5 through 5-8 for flow diagrams showing the possible pathways and states.



4.2 Operating Procedures

Step 1) Turn the DryVap[®] Concentrator System on using the ON/OFF switch located on the back, lower left-hand side.

Step 2) Turn the main nitrogen gas source on and adjust the gauge to 50 – 60 psi. Set the pressure gauge on the DryVap System to the desired pressure (typically 20 psi). This should have been done during the initial system checkout.

Step 3) Turn the main vacuum source on and adjust the main vacuum level to maximum vacuum. It is necessary to have an inline vacuum regulator, not a bleed valve, either on the vacuum pump, or in-line between the DryVap System and the vacuum pump. Adjust the vacuum regulator to -12 inches Hg. If using a different vacuum setup, it is necessary to set the vacuum delivered to the DryVap System to -12 inches Hg.

Step 4) Not Using DryDisk: If no drying is to be used, pour the sample directly into a evaporation tube, and load the filled tube into the Cradle. Use the handle to lift the Lid Assembly up for the station that is to be used and load the concentrator tube into the Cradle. The Cradles are located right in front of each Station Control Panel. Gently lower the Lid Assembly until it makes contact with the concentrator tube. Always use the handle on the lid to raise or lower the Lid Assembly. Remember to press down firmly on the Lid, such that the Cradle Basin's feet get slightly compressed. Skip to Step 10).

Step 5) Using DryDisk[™]: If a sample is to be dried with the DryDisk, place an empty 200 mL evaporation tube into the Cradle by using the handle to lift the Lid Assembly. The Cradles are located right in front of each Station Control Panel. Gently lower the Lid Assembly until it makes contact with the evaporation tube.

Always use the handle on the lid to raise or lower the Lid Assembly. Remember to press down firmly on the Lid, such that the Cradle Basin's feet get slightly compressed. *Notice 1:* When a evaporation tube is in the Cradle, the tube sensor detects it and places the station in the *Ready* state. The *Ready* light will be displayed on the Station Control Panel. The station will not start unless it is in the *Ready* state.

Step 6) Remove the DryDiskTM Luer-slip plug from the DryDisk port on the station to be used. **Do not** discard the Luer-slip plug . (Newer systems tether the plug to the front cover).

Step 7) Use the 225 ml DryDisk Assembly and follow the directions that came with the DryDisk Assembly to assembly this device.

Step 8) Select the proper *Dry Volume* setting from the Master Control Panel; 20, 100, or 200 ml. When the dry feature is used, the volume selected initiates a timer, which is the amount of time that the vacuum will pull on the bottom side of the DryDisk. The following are the actual times that will be used to pull the solvent extract through the DryDisk device.

- a. 20 ml = 1 minutes and 30 seconds.
- b. 100 ml = 3 minutes
- c. 200 ml = 4 minutes and 30 seconds

Notice 2: Even though the DryVap® System has been designed with minimal tubing lengths and dead volumes, a good suggestion would be to use a solvent wash bottle to wash out the DryDisk device, when the sample is almost completely through the DryDisk membrane. This extra solvent wash will help ensure that the entire sample has been pulled into the concentrator tube, and that the connecting line between the DryDisk port and the sparge gas tube (where the sample gets introduced into the evaporation tube), gets flush with clean solvent. (continued)



Notice 3: Based on the cleanliness of the solvent extract, or the amount of emulsion that might be present, it might be beneficial to use a longer dry time, by selecting a larger volume size. For example, if a 100 ml sample is to be dried, but it also has an emulsion present, it would be best to use the 200 ml setting, as this will pull the extract through the DryDisk for a minute and a half longer. The added time helps ensure that the entire sample gets pulled through the DryDisk, and into the evaporation tube.

Step 9) Pour the sample directly into the DryDisk assembly.

Step 10) Select the parameters for each station from the Master Control Panel. Refer to Section 4.1 of the manual for a definition of each parameter.

A. Select the desired *Heater Power* setting. For most work, the max setting of 5 is recommended.

B. For applications where the endpoint volume is 1 mL or less, where the optical liquid sensor in the cradle determines the endpoint, set the Heater Timer to OFF. The heater is then turned off by the thermocouple sensor. Only If it is desired that the concentration process run for only a specified time, or if the sample is known to turn opaque during the concentration process, than the *Heater Timer* is used. Each setting is incremented by 5 minutes. Therefore:

•Off = (timer is off) Thermocouple Sensor turns heater off

- •Heater Timer 1 = 5 minutes
- •Heater Timer 2 = 10 minutes
- •Heater Timer 3 = 15 minutes
- •Heater Timer 4 = 20 minutes
- •Heater Timer 5 = 25 minutes
- C. Set the Auto-Rinse Mode to the desired setting:
 - Off = No rinse.

•1 = Rinse once when the sample reaches the optical liquid level sensor, and continue with vacuum and sparge gas until the liquid level again reaches the liquid level sensor. The run is complete and the station goes to *Finish*.

•2 = Rinse once when the heater shuts off, and continue the vacuum and sparge gas until reaching the optical liquid level sensor. The run is complete and the station goes to *Finish*.

•3 = A combination of modes "1" and "2". Rinses once after the heater shuts off, and a second time when the optical liquid level is reached. Vacuum and sparge gas continue until the liquid level again reaches the optical liquid level sensor. The run is complete and the station goes to *Finish*.

• 4 = This mode is used with the *Heater Timer* function. This mode rinses once when the heater is shut off by the timer. The run is complete and the station goes to *Finish*. The concentration *does not* continue vacuum and sparge gas to the liquid level sensor.

• **5** = Rinses once when the solvent level in the evaporation tube reaches the liquid level sensor. The run is complete and the station goes to *Finish*. The concentration **does not** continue to liquid level sensor again.

Step 11) Once all of the DryDisk[™] devices and/or and the concentrator tubes are filled with the samples, press the *Start* key on each Station Control Panel for which there is a sample.

Step 12) Once a station is started, if the sample is to be dried, the *Dry* and *Vacuum* LED will come on, and pull the solvent through the DryDisk for the specified time. Once the dry timer expires, the *Dry* LED will turn off, and the *Heat*, *Vacuum*, and *Sparge* LED's will be on (the *Heat* LED will only turn on, if a *Heat Power* setting of 1 – 5 has been selected). If no samples are to be dried, the *Heat*, *Vacuum*, and *Sparge* LED's will turn on immediately after pressing the *Start* key. Based on the actual status of the station, these LED's will change, indicating the current status of each station.



Notice 4: If some of the sample is still left in the DryDisk, press the Stop key on the station, and press Start a second time. The dry sequence will begin again, and pull the sample through the DryDisk.

Step 13) If an Auto Rinse Mode is selected, a rinse will be dispensed during the concentration process, based on the mode selected

Notice 5: If all stations are to be run using the same set of parameters, first select the desired parameters at the Master Control Panel. Prepare each station and press the Start key on each Station Control Panel. As long as the Master Control Panel parameters remain the same, each station will use the same parameters for each run.

Notice 6: If each station is to be run using a different set of parameters, first pour the samples into the DryDisk[™] devices, or the concentrator tubes. Second, select the parameters for the first station from the Master Control Panel and press the Start key for that station. Reset the parameters for the next station from the Master Control Panel and then press the Start key for the station. Remember, when pressing the Start key on a Station Control Panel, the Station Control Panel loads the selected parameters from the Master Control Panel to that station.

Helpful hint; keep a record of the conditions set on the Master Control Panel if running different methods. ACAUTION Be careful of the heater as the surface may be hot. Hot surface. Do not touch



Sep 14) When the process is complete, the Finish LED will come on, and the beeper will sound. Press the Stop key to silence the beeper, lift the lid Assembly up, using the handles, and remove the concentrator tubes from the Cradles.

Step 15) Process the sample as would normally be done after a concentration process.

4.3 Additional Operating Procedures

Once a sample run has been started, there are two options available to interact with the run. The operator can elect to **Pause** and **Resume** the run, or to the **Stop** run. See the information below for more detail.

NOTICE: Pause/Resume can be used during the Dry State, Heat State or Sparge State. If implemented during the Heat State (when heat timer is OFF), the software algorithm that monitors the thermocouple will be have insufficient data to predict when to turn the heater off (as the heat has been turned off during the pause state). In this situation it is recommended to Stop and restart the run. (If Drying was used, just turn off the Drying step before restarting).

•Pause / Resume Key: If a sample needs to be tended to before the end of the concentration process, the **Pause** key can be pressed. By pressing the **Pause** key, all timers will be suspended, and the heater, vacuum, and sparge gas (if any are on), will be turned off. The LED's on the Station Control Panel will flash 1 time per second, indicating the stage the station was in. Once the sample has been tended to, the **Resume** key can be pressed. The station will return to the run state, and the sample will be concentrated to the final station set point.

•Stop Key: If the run is to be aborted, the Stop key should be pressed. Upon pressing the Stop key, all functions will be turned off, except the sparge gas function, which will continue the sparge gas process for 5 seconds. This is to ensure that all sample is evacuated from the sparge gas tube, and to keep the sample under a nitrogen atmosphere. Once the 5 second timer has expired, the **Finish** LED will flash and the beeper will sound. Press the **Stop** key to silence the beeper, or remove the concentrator tube from the Cradle.



Fig. 4-1

Flashing LEDs other than Ready or Finish, indicates station has been PAUSED.

4.4 Control Station Panel Finish LED

To provide a level of diagnostic information, the Station Control Panel *Finish* LED will flash a specific number of times per second, to indicate that an action was taken, or that a problem has occurred with a sample. See the information below for more details.

1 Flash / second: One flash / second of the *Finish* LED indicates that the *Stop* key has been pressed. The *Finish* LED will continue to flash until the *Stop* key is pressed a second time, or until the evaporation tube is removed from the Cradle. The beeper will also sound until the *Stop* key is pressed or the tube removed from the Cradle.

2 Flashes / second: Two flashes / second of the *Finish* LED indicates that there was an Overheat condition of the heater. The beeper will also sound. Press the *Stop* key to clear the fault. The Overheat setting of the heater is a Factory setting, but this setting can be changed. Please contact Horizon Technology, Inc. for more details on making specific software changes.

3 Flashes / second: Three flashes / second of the *Finish* LED indicates that the timer for either the Heater, or the Sparge Gas purge has been exceeded. The default setting for both timers is set to one hour. As an example, if a Heater setting of 1 is selected for a high boiling solvent, and the thermocouple does not sense the liquid level within one hour of starting the run, the heater timer will be exceeded, and the station will shut down. The *Finish* LED will flash three timers / second and the beeper will sound. Press the *Stop* key to clear the fault.



Fig. 4-2

A flashing Finish Light provides diagnostic information.



4.5 Learn Mode Programming

4

The Learn Mode is a unique feature which allows each Station's heater to be calibrated for the characteristics of a new solvent or solvent mixture. As each station runs independently, the Learn Mode should be used on all stations which will run the new solvent. (Software is not required for the Learn Mode).

NOTICE: Before using the Learn Mode, it is important to know the following;

•Each of the six heaters is calibrated at the factory using Learn Mode to obtain an offset value called T13 .

•The DryVap is calibrated with 25 mL of methylene chloride (DCM), heat power 5, and -12 in Hg. The value obtained is stored in each station. The typical T13 range is 60 to 300).

•These factory settings work well for DCM, hexane, and petroleum ether. Recalibration for these solvents should not be necessary.

•High boiling solvents such as ethyl acetate and acetonitrile, or even higher boiling solvents will require the Learn mode to operate properly. If the factory settings are used it is common for the heaters to turn off late.

•Lower T13 values turn the heater off earlier. Access to the T13 values can be through a serial port connection between the PC and the DryVap system. Programming can be performed with HyperTerminal or the optional application DryVap Programmer. Refer to the software manuals provided in the CDROM for more information. (HyperTerminal or DryVap Programmer is NOT required to use the Learn Mode).

•Users should keep record of the T13 values obtained from Learn Mode, so that they can be entered through software without having to redo the Learn Mode to return to original conditions or to enter a new method.



Fig. 4-3

Depress the Pause/Resume button down for 10 seconds and the Ready Light will begin to flash. This indicates that the station has been placed in the Learn Mode for heater calibration

•Factory T13 values along with the complete set of T-Values used in programming the DryVap system are provided in a document found in the DryVap System papers during installation. Horizon Technology maintains records in house and the information can be provided by contacting service. The information Horizon needs is the serial number of the DryVap system.

•Learn Mode requires a minimum of 20 mL of solvent to run the test. If the Learn Mode is being applied to high boiling solvents such as ethyl acetate, use the typical volume of solvent used in the application for best results.



4.5 Continued (LEARN MODE STEPS)

4

Step 1) Turn on the DryVap[®] Concentrator System, and set the vacuum to the desired level.

Step 2) Place a concentrator tube with a minimum of 20 ml of the solvent to be calibrated into the desired station. The *Ready* LED must be on.

Step 3) Set the heater to power level 5, (Dry Volume, Heat Timer, and Auto Rinse are all OFF)

Step 4) Press the *Pause / Resume* key and hold it for 10 seconds.

Step 5) The *Ready* LED will flash as an indicator that the station is in the Learn Mode.

Step 6) Press the Start key to begin the run.

Step 7) The run will begin as normal, and the heater will turn on. When the solvent level reaches the desired level above the heater coil (~ 1 mm from the top of the heater), press the <u>Start key</u> again.

Note 1: Pressing *Stop* at this time will terminate the calibration run.

Note 2: As the level of the solvent approaches the top surface of the heater, observe the tip of the sparge gas tube. When the tip of the sparge gas tube just becomes exposed from the surface of the solvent, press Start. Refer to Fig 4-6.

Note 3: Use the Quick time movie tutorials for a physical demonstration of the Learn Mode. Click links on the right or locate files in the demo folder of the User's Guide CDROM

Step 8) The software has now been calibrated for the solvent just run. The run will continue to the optical liquid sensor end point, or it can be stopped if desired.

Notices

1. A convenient way to determine when to turn the heater off is to first turn off the sparge gas. Now, the only turbulence in the solvent will be due to the boiling of the solvent. press the *Start* key which will turn the heater off.

2. If at any time the heater does not turn off at the desired solvent level, run the Learn Mode program to correct the software algorithm.

3. If the station proceeds directly to a flashing Finish light, then the offset value determined was below an internally set min. value. (Default minimum is 60 and is found in parameter T38, if samples froth more than usual then this value may be lowered to obtain a lower T13 value.) Redo the calibration.

4. DryVap Programmer users can make manual adjustments to the T13 heater calibration factor. Make changes in units of 20. Raising the number turns the heater off later, lowering the number turns the heater off earlier.



Fig. 4-4

Fig. 4-5



Fig. 4-6


Sparge Heat Function/ Shutdown Procedure



4.6 Sparge Heater Function

4

Notice 1: The Sparge Heater Function is a feature that is accessible only with a HyperTerminal connection or with DryVap Software. This feature is disabled with the factory settings.

Notice 2: Programming and Monitoring of Operation and Optimization is best performed using DryVap Software.

Sparge Heat is a target temperature mode of operation that is typically used to speed the evaporation of solvent surrounding the heater just after the heater has just been turned off (automatically) when running In the normal mode of heater operation. In the normal mode of heater operation, the temperature is controlled by the evaporation of the solvent (BP of the solvent under vacuum). Once the solvent has evaporated or is about to evaporate away from the heater coil, the heater would get very hot as the vapor does not control the temperature of the heater as well as the solvent. Sparge heat keeps the power on the heater as long as the target thermocouple value is not exceeded. The thermocouple monitors the temperature and quickly pulses the heater to maintain the desired set point. When the heater pulses, the *Heat* LED will turn on.

There are two T-List parameters, T6 Sparge Heat, and T7 Sparge Temp which need to be non-zero value to activate. T6 is the Sparge Heat power which is typically set to the value of 161 (equivalent to heat power 1 of 24W). T7 is the sparge heat target thermocouple value which is set to the value obtained for the thermocouple value reached when the solvent is boiling. Although there are ballpark values for each solvent, due to the variation of coupling of the internal thermocouple with the heating element, responses can vary. An example for DCM would be around 30 units, however depending on the heater, the value could be higher or lower by about 15 units. It is recommended to use a target thermocouple value slightly lower than the BP value under vacuum.

Application of sparge heat should be attempted only when the non-sparge heat mode performance has been determined (e.g. % recovery, run time etc.).

Note: If a station has been programmed to run with sparge heat, this target temp. heater running mode can replace the normal heater running mode by turning heat power off on the Master Control Panel (The station then looks to see if T6 and T7 have been programmed). DryVap Monitor provides indicator lights for Sparge heat and which state it is operating under.

4.7 Shutdown Procedure

Once all samples have been concentrated, follow the steps below to shutdown the DryVap® System.

Step 1) Turn off the main power switch.

Step 2) Turn off the gas supply. If the gas supply is also being used to supply other lab instruments, than adjust the gas regulator on the DryVap System to zero.

Step 3) Turn off the vacuum pump/source unless this vacuum source is also being used to supply other lab instruments. If using a diaphragm vacuum pump, bleed the system by allowing air into the vacuum lines. This action will extend the useful life of the diaphragm.

Step 4) It is recommended that the concentrator tubes be left in each station. This will ensure that heaters and internal parts remain clean.



The DryVap[™] System uses advanced software logic to operate the system. The following charts and diagrams present the operational logic for a DryVap station. Note the logic diagrams include an advanced feature called Sparge Heat which can only be programmed into the station through commands sent through a serial port connection with a PC running either HyperTerminal or DryVap Programmer.

DryVap State Diagrams for Code v1.3

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- 5-9 Internal Station Parameters (T-Lists)

Six Main Station States/ Indicator LEDs





The DryVap[™] system has six main station states as illustrated above. LED Indicator lights for each state are shown for easier identification. In the logic flow charts shown later in this section, there is a short transitional state between the Sparge and Finish State called the Hold state. The Hold state default duration is 5 seconds where vacuum is terminated and vented to atmosphere while nitrogen sparge continues. This blankets the sample with the inert gas while is stays in the Finish state.



The heat state can be modified through the settings on the master control panel and through modification of the station board's T-List values for "Sparge Heat".

| SYMBOL | MASTER CONTROL SETTINGS | ACTION |
|---|--|--|
| HEAT STATE I Heat, Sparge & Vacuum Leave State by Thermocouple (or Tmax, Time Out) | Heat Power ON (set 1-5) Heat Timer OFF (Sparge Heat Settings in Station Not Used even if programmed) | Heater is turned off by the thermocouple sensor algorithm, when heater turns off, the next state is the sparge state which continues evaporation down to the optical endpoint, then goes to Finish. |
| HEAT STATE II Heat, Sparge & Vacuum Leave State by Heat Timer (or Tmax, Algorithm) | Heat Power ON (set 1-5) Heat Timer ON (set 1-5) (Sparge Heat Settings in Station Not Used even if programmed) | Heater turns off based on heater timer, when heater turns off, the station goes to the Finish state. |
| HEAT STATE III Sparge Heat, Sparge & Vacuum Leave State by Heat Timer | Heat Power OFF Heat Timer ON (set 1-5) (Sparge Heat Settings in Station are Used to Moderate Power) | Target temperature mode, sparge heat, is used instead of Heat power. (Station must be programmed by software to activate sparge heat settings). When the heater turns off, the station goes to the Finish State. |
| HEAT STATE IV NO Heat, Sparge & Vacuum Leave State by Heat Timer | Heat Power OFF Heat Timer ON (set 1-5) (Sparge Heat Settings in Station are set to zero) | The Heat State is made to act like the sparge state (sparge, vacuum and NO heat). Sparge heat settings values are the default factory settings OFF, where T6 and T7 are zero. When the heat timer has elapsed, the station goes to the Finish State. |

Note: Using the optional accessory DryVap software enables sparge heat to be activated and optimized, along with custom times for the 5 heat timer settings. HyperTerminal may also be used for this function.



The sparge state II can be activated by using the optional accessory DryVap software for programming the station or with HyperTerminal.

| SYMBOL | MASTER CONTROL SETTINGS | ACTION |
|--|--|---|
| SPARGE STATE I Sparge & Vacuum Leave State by Liquid Level Sensor, Prg.Sprg. Tmr. or Time Out | Sparge State Does Not Use Master Control Settings. Sparge Heat Settings in Station are set to zero (default). | This state follows the heat state. No heat is applied. When the optical sensor is tripped, the sparge state is left and proceeds to the Finish state |
| SPARGE STATE II Sparge Heat, Sparge & Vacuum Leave State by Liquid Level Sensor, Prg.Sprg. Tmr. or Time Out | Sparge State Does Not Use Master Control Settings. Sparge Heat Settings in Station are set are activated. | This state follows the heat state. Sparge is activate through software, and target temperature mode of gentle heating is activated. This speeds up the evaporation time of the liquid just covering the heater coils until the liquid is no longer in contact with the heater coil. This state is left and goes to the Finish state. (It is advised to compare recovery of analytes against the normal non-sparge heat to confirm no major losses have occurred.). |

Note: DryVap software (optional software) or HyperTerminal, enables sparge heat to be activated and optimized. Software programming of the station also enables additional features and modifications to the running method, such as disabling the liquid sensor and changing the sparge state duration to convert the Sparge State into a custom sparge timer mode.



Section 5: Programming Logic

DryVap State Diagram for Code v1.3: Auto Rinse Modes 1 and 3



5-5

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DryVap State Diagram for Code v1.3: Auto Rinse Mode 2



5-6

removed

DryVap State Diagram for Code v1.3: Auto Rinse Mode 4 5 5-7 READY DRY HEAT SPARGE FINISH Start DRY STATE YES End Timer READY (Vacuum) Dry Vol. ON? STATE Leave State by 100 200 mL Dry Vol. Timer DRY 11 VOLUME 20 NO No Tube Tube HEATER POWER NO TUBE STATE HEATER TIMER AUTO RINSE 2 MODE OFF POWER **HEAT STATE II** Heat Pwr ON? YES Heat, Sparge & Vacuum End Timer & Leave State by Heat Timer Heat Tmr ON? (or Tmax, Algorithm) NO HOLD STATE 5 sec **HEAT STATE III** Sparge, End Timer Auto Rinse Sparge Heat, Sparge & Vacuum No Vacuum YES → Heat Pwr OFF? YES Leave State by Heat Timer Leave State Is Sparge Heat & After 5 sec Programmed? Heat Tmr ON? NO HEAT STATE IV End Timer NO Heat, Sparge & Vacuum Leave State by Heat Timer **FINISHED STATE** Leave state when No Tube or Stop pressed Stop is pressed

or sample tube removed

Section 5: Programming Logic

DryVap State Diagram for Code v1.3: Auto Rinse Mode 5



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Section 5: Programming Logic



Each DryVap station board has loaded into it 38 T-List parameter values. An example of the a T-List value is T13 which is the heater offset value used to calibrate heaters. This parameter can be optimized through front panel control using the "Learn Mode" or through serial port communication with a PC running either HyperTerminal or DryVap software.

The DryVap software option enables the user to customize timer durations for sparging, rinsing, heating or drying along with saving the changes as reloadable method files.

For more information about the DryVap Software or HyperTerminal, refer to the manuals located in the DryVap Software folder on the DryVap User's Guide CDROM.

> Color Legend Dry Times Finishing Sample Max Time States Sparge Heat Conditions Heater Off Algorithm Cradle Sensors Heat Timers Rinse Timers Do Not Change

| Parameter | Parameter name | Value | Units | Value variance |
|-----------|--------------------|----------|-------------|-----------------------|
| Т0 | 0 mL Volume | 3 | x 0.1 sec | (0.3 sec) fixed |
| T1 | 20 mL Volume | 900 | x 0.1 sec | (1.5 min) fixed |
| T2 | 100 mL Volume | 2100 | x 0.1 sec | (3.5 min) fixed |
| Т3 | 200 mL Volume | 2700 | x 0.1 sec | (4.5 min) fixed |
| T4 | Spit Duration | 20 | x 0.1 sec | (2 sec) fixed |
| T5 | Sparge Time | 36000 | x 0.1 sec | (60 min) max time |
| T6 | Sparge Heat | 0 | units | 0-255 |
| T7 | Sparge Temp | 0 | units | 0-100 mV |
| Т8 | Hold Time | 50 | x 0.1 sec | (5 sec) fixed |
| Т9 | Max Temp | 700 | units | fixed |
| T10 | Tube Trip | 700 | units | |
| T11 | Level Trip | 300 | units | varies with station |
| T12 | Delay (D) | 60 | sec | fixed |
| T13 | Offset (Off) | (75-250) | units | Varies with heater |
| T14 | Count (C) | 1 | units | fixed |
| T15 | Power OFF | 0 | units | fixed (equiv. to 0W) |
| T16 | Power 1 | 161 | units | fixed (equiv. to 24W) |
| T17 | Power 2 | 189 | units | fixed (equiv. to 33W) |
| T18 | Power 3 | 213 | units | fixed (equiv. to 42W) |
| T19 | Power 4 | 235 | units | fixed (equiv. to 51W) |
| T20 | Power 5 | 255 | units | fixed (equiv. to 60W) |
| T21 | Samples 0 | 0 | sample size | fixed |
| T22 | Samples 1 | 200 | sample size | fixed |
| T23 | Samples 2 | 162 | sample size | fixed |
| T24 | Samples 3 | 118 | sample size | fixed |
| T25 | Samples 4 | 101 | sample size | fixed |
| T26 | Samples 5 | 90 | sample size | fixed |
| T27 | TIME OFF | 36000 | x 0.1 sec | (60 min) max time |
| T28 | TIME 1 | 3000 | x 0.1 sec | 5 minutes (fixed) |
| T29 | TIME 2 | 6000 | x 0.1 sec | 10 minutes (fixed) |
| T30 | TIME 3 | 9000 | x 0.1 sec | 15 minutes (fixed) |
| T31 | TIME 4 | 12000 | x 0.1 sec | 20 minutes (fixed) |
| T32 | TIME 5 | 15000 | x 0.1 sec | 25 minutes (fixed) |
| T33 | Rinse Vacuum | 20 | x 0.1 sec | 2 sec (fixed) |
| T34 | Manual Rinse | 20 | x 0.1 sec | 2 sec (fixed) |
| T35 | Auto Rinse | 20 | x 0.1 sec | 2 sec (fixed) |
| T36 | Stop Delay | 50 | x 0.1 sec | 5 sec (fixed) |
| T37 | Min offset | 60 | units | 60 fixed |
| T38 | Level Sensor Delay | 20 | x 0.1 sec | 2 sec (fixed) |

LAUNCH DryVap Software Manual

LAUNCH DryVap HyperTerminal Manual



Optical Endpoints

Select Target Endpoint Volume



This section is designed to aid the user in achieving the desired endpoint volume by specifying the;

- Selection of EV Tube type
- Accessories (if any)
- Method
 - Master control settings
 - Sparge & vacuum settings
 - Station programming

Note: Endpoint volumes are approximate.



There are two programming approaches to obtain a 10 mL endpoint.

OPTION 1: Thermocouple Sensor Approach

This approach is the easiest to set up and perform as it permits any volume from 20 mL up to 200 mL to automatically be brought down to the liquid level above the heater ~5 to 6 mL. The sample is transferred to a 10 mL volumetric. A few mL of rinse solvent can be added to the EV tube to complete the transfer, followed by bringing the sample volume to mark on the volumetric flask.

OPTION 2: Heater Timer Approach

This approach can bring the sample very close to the 10 mL endpoint but requires the starting sample volume to always be the same. It also requires knowing the evaporation rate of the solvent under the running conditions.

Click button for Option1

10 mL Endpoint (Thermocouple Sensor)

Click button for Option 2

10 mL Endpoint (Heat Timer)

10 mL Endpoint Volume (Option 1: Thermocouple Sensor)





10 mL Endpoint Volume (Option 2: Using Heat Timer)





Section 6: DryVap Endpoint Volume Method Guide

>1 mL to 5 mL Endpoint Volume





Section 6: DryVap Endpoint Volume Method Guide

1 mL Direct to GC vial

6





Section 6: DryVap Endpoint Volume Method Guide

1.0 mL Endpoint



Required Glassware 200 mL Evaporator Tube

1 mL Tip 1 mL optical endpoint

Part No.; 03-1588-01 Qty 1

Comments

6

This method is standard and straightforward. The 1 mL optical endpoint EV tube is not a standard item with DryVap and needs to be ordered separately.



0.9 mL Endpoint



Required Glassware 200 mL Evaporator Tube

1 mL Tip 0.9 mL optical endpoint (Standard Tube with DryVap system)

Part No.; 03-1588-04 Qty 1

Comments

6

This method is standard and straightforward. The 0.9 mL optical endpoint EV tube is a standard item with DryVap.





0.8 mL Endpoint



Required Glassware 200 mL Evaporator Tube

1 mL Tip 0.9 mL optical endpoint

Part No.; 03-1588-04 Qty 1

Comments

This method is standard and straightforward. The 0.9 mL optical endpoint EV tube is a standard item with DryVap.

Requires two (2) C-Shape Cradle Spacers PN: 02-2110; Qty 1

The C-Shaped Cradle Spacer when placed in the cradle slot, raises the EV tube up which results in lowering position of the optical liquid sensor on the tip. Each spacer lowers the endpoint volume by 0.05 mL. Two spacers can be stacked on top of each other for a maximum optical volume reduction of 0.1 mL. The optical endpoint volume for the 03-1588-04 EV tube with 1 mL tip can be lowered from 0.9 mL down to 0.85 mL with one spacer or 0.8 mL with two spacers.



RUNNING CONDITIONS Master Control 100 200 mL VOLUM 1 Select Dry Vol. (Optional) HEATER POWER 1 2 Set Heat Power to 5 HEATER TIMER 1 3 Set Heat Timer to OFF AUTO RINSE 4 Set Auto Rinse Mode to OFF 5 Set Sparge Gas to 20 psig 6 Set Vacuum to -12 in Hg. **Sparge Gas** Vacuum

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Section 6: DryVap Endpoint Volume Method Guide

0.5 mL Endpoint



Required Glassware 200 mL Evaporator Tube

0.5 mL Tip 0.5 mL optical endpoint

Part No.; 03-1588-02 Qty 1

Comments

This method is standard and straightforward. The 0.5 mL optical endpoint EV tube is not a standard item with DryVap and needs to be ordered separately.



0.4 mL Endpoint



Required Glassware 200 mL Evaporator Tube

0.5 mL Tip 0.4 mL optical endpoint

Part No.; 03-1588-05 Qty 1

Comments

6

This method is standard and straightforward. The 0.4 mL optical endpoint EV tube is not a standard item with DryVap and needs to be ordered separately.



0.3 mL Endpoint



Required Glassware 200 mL Evaporator Tube

0.5 mL Tip 0.4 mL optical endpoint

Part No.; 03-1588-05 Qty 1

Comments

6

This method is standard and straightforward. The 0.4 mL optical endpoint EV tube is not a standard item with DryVap and needs to be ordered separately.

Requires two(2) C-Shape Cradle Spacers PN: 02-2110; Qty 1

The C-Shaped Cradle Spacer when placed in the cradle slot, raises the EV tube up which results in lowering position of the optical liquid sensor on the tip. Each spacer lowers the endpoint volume by 0.05 mL. Two spacers can be stacked on top of each other for a maximum optical volume reduction of 0.1 mL. The optical endpoint volume for the 03-1588-04 EV tube with 1 mL tip can be lowered from 0.9 mL down to 0.85 mL with one spacer or 0.8 mL with two spacers.



RUNNING CONDITIONS Master Control 100 200 mL DRY VOLUN 1 Select Dry Vol. (Optional) HEATER POWER 1 2 Set Heat Power to 5 HEATER TIMER 1 3 Set Heat Timer to OFF AUTO RINSE 4 Set Auto Rinse Mode to OFF 5 Set Sparge Gas to 20 psig 6 Set Vacuum to -12 in Hg. **Sparge Gas** Vacuum



Complete Removal of Solvent





Recommended Glassware* 200 mL Evaporator Tube with solid tip

Use this tube for most convenience in sample transfer for sample volumes > 1mL.

Part No. 03-1588-03; Qty 1

Solid Tip EV Tube:

6

The 0.4 mL optical endpoint EV tube is not a standard item with DryVap and needs to be ordered separately.

Solvent Removal:

Since the goal is to remove solvent from a sample and there is no optical endpoint sensor being used, sparge gas pressure can be increased up to 30 psi as long as there is no splattering of the solvent. Unless the sample is of similar volatility to the solvent, the vacuum can be increased up to the limit of the regulator which about -18 inches of Hg. Greater vacuum levels will require removal of the regulator and will be as high as the vacuum pump limit which is -25 inches Hg. Caution: Higher vacuum may cause the solvent to boil violently splattering sample on the sides of the EV tube.

Important:

The clear glass tip prevents the liquid optical sensors from tripping. When the heater turns off using the thermocouple sensor, the liquid level will be just above the heater coils. The sparge state will continue on with sparge gas and vacuum for 1 hour (default factory program) and then time out and go to the Finish state where the Finish LED on the station will flash three times a second (indicating a false error condition).

Using DryVap Programmer or HyperTerminal, parameter T5 (sparge time) can go as 1 hour 48 min 55 sec. (The maximum value to input is 65355 which the duration time of the sparge state in tenths of seconds.)

RUNNING CONDITIONS

Master Control



HEATER TIMER

1 Select Dry Vol. (Optional)

2 Set Heat Power to 5



3 Set Heat Timer to OFF

4 Set Auto Rinse Mode to OFF



5 Set Sparge Gas to 20-30 psig

Sparge Gas





Vacuum



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Cleaning Between Runs w/DryDisk

Sample Transfer

Solvent Exchange

Changing Rinse Solvents



DryVap Weekly Cleaning

The following is a recommended procedure for rinsing and cleaning of the Horizon Technology DryVap automated concentrator.

Pre-Clean of metal surfaces.

- 1. Remove Evaporation (EV) tubes from DryVap.
- 2. To prevent damage to the optical sensors located in the station cradle, cover the cradle with a weighing pan.
- 3. Wear protective eyewear.

4. With a toothbrush dipped in Isopropyl Alcohol (IPA) or similar alcohol gently scrub the metal surfaces of the DryVap insert such as the heater and the sparge tube.

5. Replace EV Tubes.

DryVap Rinse Procedure with DryDisk installed.

- 1. Replace EV Tubes after sample removal into the DryVap.
- 2. Remove DryDisk assembly and empty residual water to waste.
- 3. Rinse DryDisk assembly (with DryDisk still installed) 2-3 times with Acetone to waste to remove water.
- 4. Replace DryDisk into DryVap.
- 5. Using 50:50 Dichloromethane/Acetone as the DryVap rinse solvent, perform 3 manual rinses of each DryVap EV tube.
- 6. Ensure the Dry Volume is set to greater than zero.
- 7. With a rinse bottle rinse the DryDisk assembly with approximately 5 ml of DCM to ensure the DryDisk is covered.
- 8. Press start on the DryVap Station to draw the DCM through to EV Tube and when all the solvent is removed from the DryDisk press Stop on the DryVap Station.
- 9. Repeat steps 7 & 8 2-3 times.

10. Raise DryVap lid and before removal of EV Tube rinse heater and Sample/N2 nozzle with DCM. It is suggested that a gentle squirt of DCM on the stem of the heater ensures the DCM rinses the entire heater coil.

- 11. Remove EV tube and empty rinse solvents to waste.
- 12. Rinse EV tube 2-3 times with DCM.
- 13. Invert EV tube to dry.

14. Replace EV tubes to DryVap. The EV tubes are now ready for next sample.

NB: The DryVap rinse procedure above can be used routinely and is recommended as a thorough rinsing procedure between samples. It is imperative that the order of each step above is adhered to, to prevent possible back siphoning from the EV tube to the DryDisk, which could cause carryover issues.



Cover the cradle when cleaning heaters, to keep the sensors and the tube flag in optimal condition.



Standard DryVap® Sample Handling Procedures

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This section describes some of the procedures used with the DryVap to get the best recoveries for the analysis. Tasks covered include how to transfer the final sample to a GC vial, how to clean the DryVap tubes, and how to perform solvent exchange.

1. Transferring the dried and concentrated extract from the evaporation tube to the GC vial.

The objective is to quantitatively transfer the contents of the evaporation tube to the GC vial and obtain the highest recovery values from the drying and concentrating steps. Although we would always like to achieve 100% recovery of all compounds, some percentage is always lost during the process regardless of the technique employed. Therefore, it is important to perform the transfer step efficiently to minimize sample loss.

Analytes are lost either through evaporation or by adhering to the glass walls of the tube and/or heater. Studies have shown that very little loss occurs during the active heat stage when the solvent is boiling away. Once the heater shuts off, however, and the system enters the sparge mode analytes can be lost to the glass walls or to evaporation. The fraction remaining on the glass surfaces can be recovered by solvent rinsing; those that have evaporated are lost from the process. Recoveries can be optimized with solvent rinsing but many methods specify that the final volume should be 1 mL so the volume of rinse solvent is limited.

The procedure below is used to optimally rinse the tube and efficiently transfer the contents of the DryVap tube to a GC vial. Figure 1 shows a DryVap tube. The red area highlights the important region of the tube that should be rinsed.

• Remove the evaporation tube from the DryVap and confirm that he final volume if 0.8 - 0.9 mL.

• Fill a 250 uL syringe with compatible solvent and position the needle near the top of the red area.

• Slowly dispense the liquid while moving the needle completely around the circumference of the tube, washing down the sides into the nipple.

• Shake the tube to guide the rinse liquid into the nipple of the tube.

• During steps 3 and 4 you will find that some of the rinse solvent has evaporated while wetting the sides of the glass. Repeat steps 3 and 4 until the volume has reached the 1 mL mark in the nipple.

• Use a Pasteur pipette to transfer the liquid from the nipple to the GC vial.

• Start by removing approx. 0.5 mL and make sure that the sample does not drip out of the pipette during transfer.

• Use the pipette to withdraw the remaining liquid in the nipple. Again, make sure that liquid does not drip out of the pipette during the transfer step.

• Add internal standards and store the sample as necessary.





2. Cleaning Procedures

7

Carryover is an issue for all concentration techniques. The cleaning objective is to remove residual material from the DryVap and prevent carryover from one sample to the next. Standard procedures for cleaning glassware should be followed. The DryVap heaters must also be cleaned between samples. Three different cleaning procedures are recommended depending on the situation. First is a normal cleaning procedures used for typical analysis of semi-volatiles in the ppm range. Second is a trace cleaning for analysis such as pesticides and PCBs at ppb levels. The third procedure is used periodically to remove material accumulated from processing dirty samples. Blank samples should be processed to validate cleaning procedures.

Normal cleaning.

•Follow standard cleaning procedures for glassware.

•Rinse glassware with a small amount of acetone. Other solvents may be more suitable depending on the analysis. Be aware that "squirt bottles" sometimes contaminate solvents with phthalates and other plasticizers.

•Place a DryVap tube in place and use the manual rinse feature to rinse the lid surfaces.

•Hold a beaker underneath the DryVap heater and use a squirt bottle to rinse down the heating coil and the sparge tube.

Trace level cleaning.

•Follow standard cleaning procedure for glassware used in trace level analysis.

•Rinse glassware with a small amount of acetone. Other solvents may be more suitable depending on the analysis. Be aware that "squirt bottles" sometimes contaminate solvents with phthalates and other plasticizers.

•Place a DryVap tube in place and use the manual rinse feature to rinse the lid surfaces.

•Hold a beaker underneath the DryVap heater and use a squirt bottle to rinse down the heating coil and the sparge tube.

•Take a small amount of methanol and use a soft bristles toothbrush to gently scrub the heater and sparge tube.

•Wipe down the lid surface with methanol.

Periodic Cleaning.

•Follow the procedures outlined for Trace level cleaning.

•Dissolve a small amount of baking soda in warm water.

•Use a soft bristled toothbrush to scrub the surface of the heater and sparge tube. Wipe down the lid surface with a cloth moistened cloth. •Repeat the cleaning procedure for trace level work.

3. DryVap Solvent Exchange Technique

The objective is to exchange the sample solvent from methylene chloride (DCM) to a second solvent that is compatible with an ECD detector. If there is too much DCM remaining in the sample it will overload the detector. Hexane is the solvent of choice because it dissolves pesticides and PCBs boils at 69°C compared to 40°C for DCM. Typically solvent exchange is performed by evaporating most of the DCM off the initial extract then adding an aliquot of hexane and continuing the evaporation. This step may be repeated until the sample extract is void of DCM.

KD, Roto-Vap and other techniques apply heat to the entire sample to evaporate the solvent. The DryVap, on the other hand, applies heat with an immersion heater and only the liquid that comes in contact with the heater is evaporated. The liquid contained in the DryVap tube nipple does not get hot. Furthermore, diffusion into and out of the nipple of the tube is slow. The hexane is added to the evaporation tube for solvent exchange the liquid in the nipple must be mixed with the added solvent. This is done by taking a Pasteur pipette and drawing up the liquid and mixing it with the main body of liquid.

The procedure below is used to exchange the extraction solvents for ECD analysis.

•Remove the evaporation tube from the DryVap and confirm that he final volume if $0.8-0.9\mbox{ mL}.$

•Add 30 mL hexane to the tube.

•Use a Pasteur pipette to draw the liquid from the nipple up and dispense it into the 30 mL of added hexane. Repeat two more times. •Replace the tube to the DryVap station.

•Replace the tube to the Dryvap statio

•Start the evaporation.

-Once the second evaporation is complete to 0.9 mL the exchange is complete



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Changing Rinse Solvents

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Replacing 240V Fuses

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Heater Shutoff Test Procedure

Vacuum Integrity Test

There are only a few procedures that should be considered routine maintenance for the DryVap® Concentrator System. These procedures are listed below. Please contact Horizon Technology, Inc. directly for any other specific questions.

8.1 Gasket Seals

8

The Gasket Seals which are pressed onto the Lid Assembly are used to provide a vacuum tight seal on the lip of the concentrator tubes. Over time, the surface of the Gaskets may distort slightly, such that it becomes more difficult to achieve the necessary vacuum seal. A quick remedy for a during a concentration run, it is possible to rotate the concentrator tube, while the tube is in the Cradle, follow this procedure.

•Lift the Lid Assembly completely up.

•Gently grab the Gasket Seal and pull it down from the Lid Assembly. The Gasket is pressed onto the Lid Assembly and will be snug.

•Once the Gasket is free from the Lid Assembly, carefully lower the Gasket and slide it around the heater and sparge gas tube. Note the side which was facing down towards the concentrator tubes,

Using methanol and a KimWipe, gently clean the top side of the Gasket Seal. Be careful not to wet the inner silicone ring.
Turn the Gasket Seal over, such that the cleaned surface is now facing **down**, and position the Gasket Seal back onto the Lid Assembly. Press the Gasket Seal firmly into place.

•This procedure should be repeated when necessary to restore the vacuum seal.



8.2 Cleaning the Heaters

Rinsing a heater immediately after running a dirty sample will minimize build-up of organic deposition. Based on the cleanliness of the samples, it might be necessary to periodically clean the heaters. The heaters are coated with Sulfinert[®], which provides a very inert surface. However, the heaters can be cleaned by using a KimWipe which has been wetted with methanol. Simply wipe the heater with the wetted KimWipe.

•If organic material has carbonized onto the heater surface and does not readily come off with a methanol dampened KimWipe, then a soft toothbrush dipped in a baking soda solution with gentle brushing can be used to loosen the material. Rinse well with distilled water to remove the baking soda. DO NOT leave the baking soda on the heater coils for any extended period as the paste is slightly alkaline and will slowly attack the Sulfinert coating.

•If a toothbrush is not available, first place a cover over the cradle under which the heater is to be cleaned, then put some baking soda in one pan and some distilled water in another pan. Wearing nitrile type gloves, wet your pointer finger with the water and then contact the baking soda to make a firm paste. Gently rub the paste on the heater coils, for the region between the coils, use a folded KimWipe to access these tight regions. Rinse well with distilled water to remove the baking soda. DO NOT leave the baking soda on the heater coils for any extended period as the paste is slightly alkaline and will slowly attack the Sulfinert coating.

A CAUTION

Do not use any abrasive cleaners or rough paper to rub or clean the heaters. This could damage the Sulfinert coating, exposing an active surface site on the heater.



Distilled Water



Cover the cradle when cleaning heaters, to keep the sensors and the tube flag in optimal condition.





8.3 Changing Rinse Solvents

If a different rinse solvent is to be used, it will be necessary to first flush the original solvent from the lines. Follow these steps.

• Lift the Rinse Solvent Line (which contains the fritted filter) from the solvent bottle and place the fritted filter into a beaker.

- Place a concentrator tube into each of the stations and lower the Lid Assembly.
- Turn on the vacuum pump / source and set the vacuum to 12" Hg.

• Press the *Manual Rinse* key on each station, starting with Station #6.. Repeat this operation several times. Each time the *Manual Rinse* key is pressed, the rinse solvent valve will open for 2 seconds. When the tube is under vacuum, this action will pull the solvent through the DryVap System.

Notice: It is necessary for a vacuum tight seal to be created during this operation. Press down firmly on the Lid Assembly to help ensure a tight seal.

• When no more solvent is been pulled into the concentrator tubes, place the Rinse Solvent line into the new solvent to be used.

• Repeat the above operation of activating the *Manual Rinse* key until a steady stream of solvent is pulled into each concentrator tube.

Replacing the Fuse (120V Operation)





not fit in the reverse orientation.

Use the same screw driver to remove the red fuse holder (B).

Replace blown fuse, reinsert fuse holder close fuse panel, reinsert power cord and power system up.

> If the unit does not power up after replacing fuses contact Horizon Tech.

Replacing the Fuse (240V Operation)





8

Remove the power cord from the power mains. Use a small screw driver to open the panel (A).



Use the same screw driver to remove the red fuse holder (B).



Pull the fuse holder out. Note the square on the fuse holder must be on this side when reinserting holder back into power mains. It will not fit in the reverse orientation.



Replace fuses on both sides of holder. Use the two adjacent clips for these short fuses.

E

Uses **two** 5A 5x20mm fuses PN: 13-0308 (for 1 fuse)

Replace blown fuse, reinsert fuse holder close fuse panel, reinsert power cord and power system up.



To Convert a 120 V to 240V





2. Remove large 5A fuse from other side of holder



3.Follow Steps D and E above.

If the unit does not power up after replacing fuses contact Horizon Tech.

Note: To convert a 240V to a 120V system. The clip will be missing contact Horizon for a clip.

Section 8: Maintenance

Tighten Fittings on Sparge/Dry Valves

If the sparge gas flow is bubbling excessively, the fittings to the Sparge/Dry Valves may have backed off. This sometimes occurs as the inert valves are made of Teflon. (Finger tighten only, do not use a wrench as this may damage the soft Teflon valve material.)



1. Remove Front Cover

2. Finger Tighten Each Side Compression Screw (Line to Station Lid). Tighten fittings clockwise.

Do this for all 6 stations



8-6

3. Finger Tighten Each Bottom Compression Screw (nitrogen feed line). The nitrogen feed line is black polyurethane tubing. Turn fitting counter –clockwise when viewing valve from top.

Do this for all 6 stations

4. Put back front cover, and test and check sparge gas flow rate.





8-7 Heater Shutoff Test Procedure:

This test procedure will allow visual confirmation that the internal thermocouple is turning off the heater at the proper time. That is, is the heater being turned off while still immersed in the solvent (*Heater* LED turns off), or is the *Heater* LED still on while the solvent level drops below the heater body? Follow the steps below to conduct this test.

Turn on the vacuum pump and adjust the vacuum regulator to 12"Hg.

Turn on the nitrogen gas and adjust the gas pressure to 20 psi on the DryVap® System.

Fill a concentrator tube with approximately 20 ml of solvent.

Place the tube into the station that has the questionable heater. Lower the Lid Assembly down onto the concentrator tube.

Dial in the following parameters at the Master Control Panel:

Dry Volume = 0

8

Heater Power = 5

Heater Timer = Off

Auto Rinse Mode = 0

Press the *Start* key on the Station Control Panel.

During the solvent evaporation process, carefully monitor the solvent level relative to the heater body. At the maximum heater power setting of 5, the solvent should aggressively boil, making it easy to determine when the heater is shut off. When the heater shuts off, the boiling immediately stops.

If the solvent level begins to drop below the heater body, while the heater is still on (i.e. the solvent is boiling), refer to Section 4.5 and recalibrate the heater.


8.8 Concentrator Tube / Vacuum Integrity Test Procedure:

The DryVap® Concentrator System is designed to operate with 12" Hg of vacuum in the Concentrator Tube. One way to determine if an adequate vacuum seal has been achieved between the Concentrator Tube and the Lid Assembly Gasket Seal is to monitor the actual temperature of the solvent being evaporated. The following chart shows the temperature of methylene chloride (DCM), and hexane, as a function of vacuum. Follow the steps below to test the vacuum seal.

Notice: A best way to measure the temperature of the solvent inside of the Concentrator Tube is with an Infrared Thermometer. These IR thermometers are available from Radio Shack.

Step 1) Fill a Concentrator Tube with 25 ml of either methylene chloride (DCM) or hexane.

Step 2)Place the tube into any one of the 6 Cradles. **Step 3**) Lower the Lid Assembly down onto the top of the Concentrator Tube; press down firmly.

Step 4) Set the Heater Power to a setting of 5 (max power). **Step 5)** After a few minutes when the solvent has been boiling, using the IR thermometer, measure the temperature of the solvent. Point the probe directly at the bottom of the tube, and towards the internal heater.

Step 6) Record the actual temperature of the solvent and using the chart, determine the vacuum level inside of the tube. If the vacuum level is less than the necessary 12" Hg, than a leak is present. The most likely leak source will be between the lip of the Concentrator tube and the bottom of the Gasket Seal. See Section 8.1 for the procedure to correct an issue with the Gasket Seals.



Notice: If the temperature indicates a lower vacuum, turn off the sparge gas and rerun the test. The addition of sparge gas into the concentrator tube will reduce the overall vacuum level.



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9



200 mL Evaporation (EV) Tube with 1 mL Graduated Tip, Optical endpoint 0.9 mL (Standard EV Tube for DryVap)



200 mL Evaporation (EV) Tube with 1 mL Graduated Tip, Optical endpoint 1.0 mL



9



200 mL Evaporation (EV) Tube with 0.5 mL Graduated Tip, Optical endpoint 0.4 mL



200 mL Evaporation (EV) Tube with 0.5 mL Graduated Tip, Optical endpoint 0.5 mL





200 mL Evaporation (EV) Tube with solid tip.

PN: 03-1588-03; Qty 1



Select this tube when the objective is to totally remove solvent from the sample or for evaporating the sample to volumes > 1 mL (no optical endpoint).

Lower Optical Endpoint Volume with C-Shape Cradle Spacers PN: 02-2110; Qty 1

The C-Shaped Cradle Spacer when placed in the cradle slot, raises the EV tube up which results in lowering position of the optical liquid sensor on the tip. Each spacer lowers the endpoint volume by 0.05 mL. Two spacers can be stacked on top of each other for a maximum optical volume reduction of 0.1 mL. The optical endpoint volume for the 03-1588-04 EV tube with 1 mL tip can be lowered from 0.9 mL down to 0.85 mL with one spacer or 0.8 mL with two spacers. The optical endpoint volume for the 03-1588-05 EV Tube with 0.5 mL tip can be lowered from 0.4 mL down to 0.35 mL with one spacer or 0.3 mL with two spacers.







Select this tube when convenience is an important factor. Sample is directly concentrated into a GC vial. Endpoint volume is approx. 0.9 mL and works with optical liquid sensor in the DryVap system. When sample is concentrated, simply remove EV tube and unscrew the GC vial from the coupler. Place a GC cap on the vial and put into autosampler.

| Part # | Qty | Description |
|------------|------|--|
| 49-2225 | 1 | KIT EV Tube, Coupling, GC Vials & Cradle Spacer |
| | | Kit includes; |
| | | 200 mL Evaporator Tube, tapered base, GC Vial thread (12x32mm), (Qty 1) |
| | | GC Vial Coupling for 12x32 mm GC Vial (Qty 1) PN 03-1657 |
| | | Box of 100, 12x32 mm GC Vials and Caps (Qty 1 Box) |
| | | Cradle Adapter for 12x32 mm vials (Qty 1) |
| 49-2231 | 1 | KIT EV Tube, Coupling & GC Vials |
| | | Kit includes; |
| | | 200 mL Evaporation Tube, tapered base, GC Vial thread (12x32mm), (Qty 1) |
| | | GC Vial Coupling for 12x32 mm GC Vial (Qty 1) PN 03-1657 |
| | | Box of 100, 12x32 mm GC Vials and Caps (Qty 1 Box) |
| 49-2232 | 1 | KIT, GC Vial Couplings (Qty 6) |
| 03-1657 | 1 bx | 12x32 mm GC Vials, Box of 100 |
| 27-2255 | 1 bx | GC Vial Caps, Box of 100 |
| 03-1653-01 | 1 | 200 mL Evaporation Tube, tapered base, GC Vial thread (12x32mm) |
| 50-2384 | 1 | GC Vial Coupling for 12x32mm GC Vial (Qty 1) |
| _02-1656 | 1 | Cradle Adapter for 12x32 mm vials (Qty 1) |



Section 9: Parts and Accessories

Call Horizon (1-800-997-2997) for current prices and delivery schedules

Parts and Accessories

9





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Tube Rack, holds six 200 mL EV tubes or six 225 mL DryDisk Assemblies.



9

PN: 01-1963; Qty 1

The DryVap tube rack is specifically designed to hold up to 6 EV tubes (upright or inverted) and or DryDisk Assemblies in any combination.

| Part # | Qty | Power Cords |
|---------|-----|---------------------------------------|
| 26-0300 | 1 | Power Cord (USA) |
| 26-2120 | 1 | Power Cord (Australia) 8 ft. |
| 26-2121 | 1 | Power Cord (Continental Europe) 8 ft. |
| 26-2122 | 1 | Power Cord (Italy) 8 ft. |
| 26-2123 | 1 | Power Cord (UK) 8 ft. |

| Part # | Qty | Spare Parts |
|------------|-----|--|
| 50-0912-02 | 1 | Vacuum Tubing Kit (8 ft FEP tubing plus fittings) |
| 50-0913 | 1 | Sparge Gas Tubing Kit (10 ft green tubing plus fittings) |
| 50-0911 | 1 | Solvent Rinse Kit |
| 50-0937 | 1 | Sealing Ring/ Gasket for DryVap station lid |



Note: Starting in 2008, the vacuum pump regulator PN 50-0947 is included with the DryVap Systems rather than with the vacuum pumps. The vacuum pumps are now preconfigured by Horizon to readily accept this vacuum pump regulator. There is no need to order this item separately.

| A. SELECT DRYVAP SYSTEM (either 115V or 230V version) | | | | | | |
|---|--------------|-----|---|-------------------|--|--|
| Part # | Voltage | Qty | Description | | | |
| 50-0916-01 50-0916-03 | 115V 230V | 1 | DryVap [®] Concentrator System Model 5000 Includes; | | | |
| | | | 200 mL Concentrator Tube, tapered base, 0.9 mL tip (Qty 6) | PN 03-1588-04 | | |
| | | | Tube Rack, 6 positions (Qty 1) | PN 01-1963 | | |
| | | | Solvent Rinse Kit (Qty 1) | PN 50-0911 | | |
| | | | vacuum pump regulator assembly (with gauge) | PIN 50-0947 | | |
| B. SELECT SOLVENT RECLAMATION SYSTEM FROM CHOICE 1 OR 2 | | | | | | |
| CHOICE 1 | | | | | | |
| Part # | Voltage | Qty | Description | | | |
| 50-2155-01 | 115V 60Hz | 1 | The Reclaimer | | | |
| 50-2155-02 | 230V 50Hz | | (Recovers up to 95% solvent vapors with DryVap system) | | | |
| 50-2155-03 | 208V 60Hz, | | Includes; | | | |
| | 200V 50Hz | | 10 L Solvent Carboy, Tubing and Fittings | | | |
| CHOICE 2 | 1 | | | | | |
| 50-1895-02 | | 1 | Dual Reservoir Liquid Trap Kit (Designed for DryVap to collect condensed solvent vapors to prot Includes; Blue Tub Bottle Holder 2.5 liter solvent bottles (Qty 3) Special manifold caps to thread onto solvent bottles (Qty 2) Complete set of brass fittings for connection to vacuum hose 16 ft. of FEP vacuum hose | tect vacuum pump) | | |





NEPM = Non Explosion Proof Motor

EXPM* = Explosion Proof Motor

ATEX^{*} = [European Commission] Equipment intended for use in potentially explosive atmospheres *NOTE: Explosion Proof Motor Vacuum Pumps must be installed by a licensed electrician.

50-2480-01 – <u>**Gast NEPM 120V 60Hz Plumbed</u></u> - Plumbed in the Dryvap style, No regulator, (but plumbed to accept vacuum regulator 50-0947 which is now included with the DryVap systems). Has head vacuum gauge and exhaust line included.</u>**

50-2480-02 – <u>Gast NEPM 220V 60Hz Plumbed</u> - Plumbed in the Dryvap style, No regulator, (but plumbed to accept vacuum regulator 50-0947 which is now included with the DryVap systems). Has head vacuum gauge and exhaust line included.

49-2481-01 – <u>Gast NEPM Pump Plumbing Kit</u> – Includes all the parts that are on the above pump assemblies, minus the pump itself. This kit is for customers who do not order a Gast pump from us. *Note:* Customers who require a 220V pump will need to figure out on their own how to connect the electrical cord on the pump (220V pumps do not come with a power cord – just a short pig-tail)

50-2483-01 – <u>KNF EPM 120-220V 60Hz Plumbed</u> – Plumbed in the DryVap style, No regulator, (but plumbed to accept vacuum regulator 50-0947 which is now included with the DryVap systems). Has head vacuum gauge and exhaust line included.

50-2483-02 – <u>KNF EPM 220V 50Hz 1PH Plumbed</u> – Plumbed in the DryVap style, No regulator, (but plumbed to accept vacuum regulator 50-0947 which is now included with the DryVap systems). Has head vacuum gauge and exhaust line included.

50-2483-03 – <u>KNF EPM 220V 50Hz 3PH Plumbed</u> – Plumbed in the DryVap style, No regulator, (but plumbed to accept vacuum regulator 50-0947 which is now included with the DryVap systems). Has head vacuum gauge and exhaust line included.

49-2481-02 – <u>KNF EPM Pump Plumbing Kit</u> – Includes all the parts that are on the above pump assemblies, minus the pump itself. This kit is for customers who do not order a KNF pump from us.



Table of Contents Links

- A-1 Quick Reference Guide
- A-2 Solvent BP vs. Vacuum Chart
- A-3 EV Tube Volumes for the Tapered Section
- A-4 Vacuum Pump Service Manuals
- A-5 DryVap Specifications
- A-6 Limited Warranty

Α



Master Control Panel



| Dry Volume Settings: | | | | |
|------------------------|---|--------------|--|--|
| 0 mL | = | OFF | | |
| 20 mL | = | 1.5 minutes. | | |
| 100 mL | = | 3.0 minutes | | |
| 200 mL | = | 4.5 minutes | | |
| Heater Power Settings: | | | | |

The 60 Watt heater is "pulsed" to deliver the desired power. The settings below indicate the amount of power being applied.

Off = Heater is OFF (Unless sparge heat is programmed into the station and heat timer is ON.) 1 = 24W

- 2 = 33W
- **3** = 42W
- 4 = 51W
- 5 = 60W (full power)

Heater Timer Settings:

Off = Heater turns off automatically by thermocouple sensor

- 1 = 5 minutes
- $\mathbf{2} = 10$ minutes
- $\mathbf{3} = 15$ minutes
- $\mathbf{4} = 20$ minutes
- 5 = 25 minutes

Auto-Rinse Modes:

Off = No rinse.

1 = Rinse once when the sample reaches the optical liquid level sensor, and continue with vacuum and sparge gas until the liquid level again reaches the liquid level sensor. The run is complete and the station goes to Finish.

2 = Rinse once when the heater shuts off. and continue the vacuum and sparge gas until reaching the optical liquid level sensor. The run is complete and the station goes to Finish.

3 = A combination of modes "1" and "2". Rinses once after the heater shuts off, and a second time when the optical liquid level is reached. Vacuum and sparge gas continue until the liquid level again reaches the optical liquid level sensor. The run is complete and the station goes to *Finish*.

4 = This mode is used with the *Heater Timer* function. This mode rinses once when the heater is shut off by the timer. The run is complete and the station goes to *Finish*. The concentration *does* not continue vacuum and sparge gas to the liquid level sensor.

5 =Rinses once when the solvent level in the evaporation tube reaches the liquid level sensor. The run is complete and the station goes to *Finish*. The concentration **does not** continue to liquid level sensor again.





DCM and Hexane are common solvents used for sample extractions. The dotted line box indicates the BP range of the two solvents between 10 and 20 inches of Hg vacuum. Increasing the vacuum decreases the vapor pressure on all solvents and analytes. Vacuum exceeding 20 inches Hg show the beginning of convergence of the vapor pressures and BPs.

Appendix 3: EV Tube Volumes for the Tapered Section







Appendices

Α



Vacuum pump head will need periodic maintenance. The Horizon Non-Explosion Proof Motor Vacuum pumps are based on the Gast pump. Models built before June 2002 will have heads as shown in Fig. 1. Models after June 2002 will have heads as shown in Fig 2. Click on Fig. 1 or Fig. 2 to launch the respective Gast Service Manual (Acrobat file ".pdf). If you have the KNF Neuberger Vacuum pump with Explosion Proof Motor click on Fig. 3.





Product Specifications

Performance:

Α

The unit has six independently operating drying and concentrating stations. Each station can dry and concentrate 200 ml of methylene chloride in less than 45 minutes. Other solvent times will vary. The resulting concentrate volume is 0.9 ml +/- 20%.

The system will automatically dry and transfer a sample to the concentrator tube using Horizon's DryDisk[™] membrane.

The system will automatically stop the concentration process via optical liquid level sensing, or at a preprogrammed time.

The Rinse Solvent function is auto programmable or manually operated.

Utility Requirements:

Vacuum: an inert pump and regulator to maintain 1 SCFM at -15"Hg.

Sparge gas: Nitrogen or other inert gas at 60 psi. Rinse solvent: Optional rinse solvent.

Environmental:

Operating Temperature:20 to 40 degrees CStorage Temperature:10 to 50 degrees CRelative Humidity:0 to 90% (non condensing)Altitude:7,500 feet

Wetted Materials and Construction:

Metals: 300 series stainless steel coated with Sulfinert® Plastics, Polymers, Composites Sample in contact with: PTFE (Teflon), PFA (Teflon), ETFE (Tefzel), UHMWPE (ultra high molecular weight polyethylene) Waste in contact with: FEP tubing Glass: Borosilicate glass

Solvent Compatibility:

Acceptable solvents: acetone, acetonitrile, ethyl acetate, hexane, methanol, methylene chloride, MTBE, and petroleum ether.

Unacceptable solvents: water, any organic solvent with a B.P. above 85C @ STP

Physical Properties:

Maximum size (inches) – 27 5/8" wide x 18 $\frac{3}{4}$ " high x 17 $\frac{1}{4}$ " deep (excluding open doors and external plumbing and accessories, i.e. power cord)

Weight (lbs) – 82 lbs

Electrical Properties:

Power Consumption: 340 VA (at 120V) Input Voltage:120-240 VAC, 47 – 63 Hz Fuse: 5 AMP MAXIMUM (1 Fuse, US .25" X 1.25") (2 Fuses, Europe 5mm x 20 mm) Fuse Type 250V, 5A, SLO BLO **Regulatory Requirements:**

Agency Approvals: UL 61010C laboratory instruments, per acceptable NRTL (TUV)



Limited Warranty

Horizon Technology, Inc. ("Horizon") warrantees the DryVap® Concentrator System (Product) against defects in material or workmanship as follows:

LABOR: For a period of one (1) year from the date of purchase, if this Product is determined to be defective, Horizon will repair the Product, and will cover all labor charges. The Product must be returned to Horizon, prepaid, for repair. Horizon will pay the return shipping charges.

PARTS: Horizon will supply, at no charge, new replacement parts for a period of one (1) year. Horizon will send the replacement parts directly to the customer site, for customer installation. If the customer does not wish to install these parts, the Product must be returned to Horizon. To obtain warranty service, the Product must be delivered prepaid, in the original packaging. If needed, new packaging is available from Horizon.

To obtain warranty service, the Product must be delivered prepaid, in the original packaging. If needed, new packaging is available from Horizon.

This warranty does not cover customer installation, or set up adjustments and / or Product optimization. The warranty also does not cover cosmetic damage or damage due to acts of nature, accident, misuse, abuse, negligence, or modifications of, or to any part of the Product. This warranty does not cover damage due to improper operation or maintenance, or attempted repair of any electrical components. No other warranties are expressed or implied. For service assistance or resolution of a service problem, or for product information or operation, call: **Horizon Technology, Inc.** 1-(800)-997-2997 1-(603)-893-3663 Or write to: Horizon Technology, Inc. 45 Northwestern Drive Salem, NH 03079 EMAIL: <u>spe@horizontechinc.com</u> Website: www.horizontechinc.com