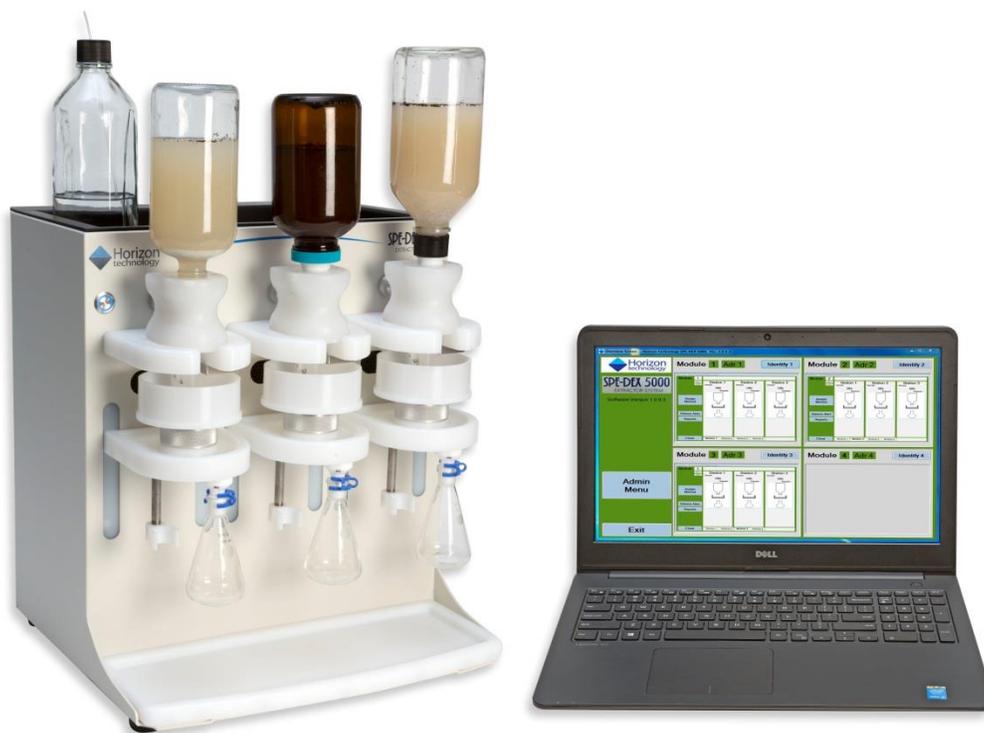


SPE-DEX[®] 5000

Automated Extraction System



Users Guide



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Preface

Your SPE-DEX[®] 5000 Automated Extraction System

Congratulations on your purchase of the SPE-DEX 5000 System, the latest generation of automated Solid Phase Extraction (SPE) Disk systems from Horizon Technology. The SPE-DEX 5000 System offers reliable automation for all SPE disk methods, does not require any external vacuum pumps or gases to process samples, and allows interfacing with a bar code scanner. All method details and run parameters can be exported to a LIMS system for archival purposes.

Modular in design, a single extractor module contains three extractor stations and, as sample processing needs increase, up to four extractor modules can be connected to and run from a single personal computer (PC). This allows up to 12 samples to be run simultaneously for maximum throughput. Each three-station extractor module is designed for the effective extraction of semi/non-volatiles and/or oil and grease from aqueous samples. The module is operated by a software program that can run preprogrammed methods. Alternatively, methods can be user-customized.

We are confident the SPE-DEX 5000 System will be a welcome addition to your laboratory for all your disk SPE sample preparation needs.

FCC (USA)

The SPE-DEX 5000 Automated Extraction System has been tested and found to comply with the limits for a Class A device, pursuant to Part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interferences when the equipment is operated in a commercial environment. This equipment generates and can radiate radio frequency energy and, if not installed properly and used in accordance with the user's manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his/her own expense.

The SPE-DEX 5000 System complies with NRTL and CE certification.

Product Safety

The SPE-DEX 5000 System is designed with user safety in mind. However, the product use is at the discretion and risk of the user or laboratory supervisor/manager. Use the product as described in this manual, particularly referencing Section 1.4, *Product Safety Notice and Certification*.

Statement of Proper Use

The SPE-DEX 5000 System is a fully automated SPE system, designed to automate the extraction of organic and inorganic compounds from aqueous samples. SPE is an effective alternative to conventional liquid-liquid extraction (LLE) and continuous liquid-liquid extraction (CLLE). Both LLE and CLLE are manual techniques, which can lead to errors, worker exposure to organic solvents, emulsions, and numerous other problems. Automated SPE eliminates or minimizes these problems. The SPE-DEX 5000 System can be used in conjunction with DryDisk[®] membrane drying, the XcelVap[®] Evaporation/concentration System, or the DryVap[®] In-line Drying and Concentration System for evaporation of the extracted material to allow for chromatographic measurement or with the Speed-Vap[®] IV for gravimetric measurement for oil and grease.

**WARNING**

To reduce the risk of electrical shock, do not disassemble the SPE-DEX 5000 System. Refer repairs to qualified service personnel.

**WARNING**

Use only the Horizon-supplied power supply AND Horizon-supplied IEC cords. Replace only with Horizon-supplied power supply and cords.

Manual Audience and Intent

This manual, which is intended for all SPE-DEX 5000 System users in the laboratory environment, provides the information needed to operate and maintain the SPE-DEX 5000 System. The information may contain typographical errors or technical inaccuracies and is subject to change without notice. Modifications or enhancements may also be made to the product at any time. For the most current information, consult the Horizon Technology website at:

www.horizontechinc.com.

Conventions

The following table provides examples of conventions used in Horizon Technology manuals.

| Example | Description |
|--|--|
| <i>Declaration of Conformity</i> | Italicized text indicates document and section titles as well as special notes. |
| Setup.Exe | Courier type indicates a program file name. |
| Save | Bold type indicates a button or information displayed on the screen. |
|  WARNING | Symbols to the left of WARNING , CAUTION , or NOTE indicate the type of danger that could be present, such as high voltage, fire, explosion, etc. See the Table of Symbols, below, for details. |

Table of Symbols

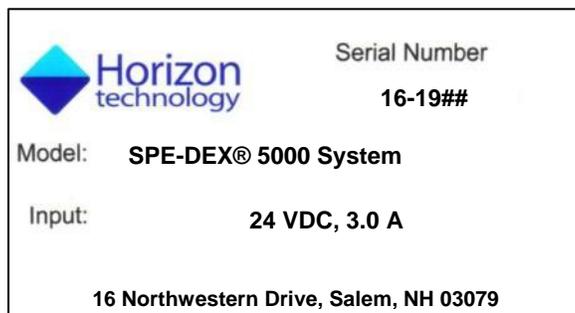
The following symbols point out important information and alert you to potential hazards.

| Symbol | Type | Description |
|---|---------|--|
|  | Warning | A potentially hazardous situation, which if not avoided could result in death or serious injury. |
|  | Note | A safety note for operation or additional explanation. This informs and guides you 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. |
|  | Caution | A caution concerning potential eye injury. Eye protection in the form of safety glasses or goggles is highly recommended when operating the SPE-DEX 5000 System and any chemical processing. If reagents, liquids, or vapors come into contact with the eyes, follow the appropriate first aid procedures set forth in the laboratory's safety manual. |

| Symbol | Type | Description |
|---|---------|---|
|  | Caution | A caution concerning the potential of a fire. |
|  | Caution | A caution to wear protective gloves when handling harmful reagents. |

Serial Number Label

The following is a sample of the serial number label located on the back of the SPE-DEX 5000 System.



Technical Support

Visit the Horizon Technology website for technical information in addition to that provided in this manual: www.horizontechinc.com. If you have questions about the SPE-DEX 5000 System that are not fully addressed in this manual or our website, please contact the Horizon Technology Technical Customer Support Center:

Phone: (603) 893-3663
 Fax: (603) 893-4994
 E-mail: support-service@horizontechinc.com

Customer Support Center hours are Monday through Friday, 8:00 a.m. to 5:00 p.m. EST. The Center provides expert technical support including troubleshooting, repair instructions, service and installation scheduling, and replacement part information.

1 Introduction and Safety

1.1 System Overview

The SPE-DEX 5000 System is the latest generation, modular automated disk extractor system from Horizon Technology. The system consists of two main components:

- Extractor Module
- Software on a standard personal computer (PC)

The SPE-DEX 5000 System can control up to four Extractor Modules. Each Extractor Module can run three samples with a single method, called a “batch mode.” With four modules, the system can run up to 12 samples simultaneously. Each individual Extractor Module can run a different batch method. This keeps the operation simple and logical.

The software provides an intuitive, graphical interface. All solvents, methods, and control parameters are visually displayed. This provides convenient and easy programming and operation. The continuous interactive feedback informs you of the current status of the extractor system. If you encounter a problem or want to interact with a station, you can pause the individual station, make a change, and then resume operation on the station to continue processing the sample.

At the end of the run, all of the data associated with that sample (date, time, user, Extractor Module and station used, method used, length of the total run time, individual run time to filter the water sample, etc.) are stored and available for export to the facility’s LIMS system. This means all steps of the sample preparation procedure can be recorded and archived—from when the sample is first logged into the facility, to the end when the analytical report is generated. A unique SPE-DEX 5000 System feature is the ability to connect a bar code scanner to the PC and scan bar code labels. The bar code serial number is then associated with a specific sample and Extractor.

The SPE-DEX 5000 System is specifically designed to handle the extraction of organic analytes and/or oil and grease from aqueous samples. Capable of using 47 mm, 50 mm, and 100 mm SPE disks and prefilters, the easy-to-use Extractor Module sets new standards for both the speed of sample extraction and the reproducibility of results.

The SPE-DEX 5000 System enables testing facilities (laboratories) to:

- Reduce laboratory costs
- Reduce manpower requirements for testing
- Provide more reliable results by reducing testing variability
- Improve user safety by reducing exposure to solvents
- Provide an increased level of productivity to the laboratory

1.2 Extractor Module

A single Extractor Module houses three individual extractor stations. As mentioned earlier, each module is designed to run in a “batch mode” to make operation of the module simple and straightforward. In batch mode, one, two, or all three stations can be used to process samples, but the Extractor Module will run the *same* analytical method. As samples are typically processed in a batch typically containing up to 24 samples and quality controls, this design approach follows the work flow currently being done in the laboratory.

Each Extractor Module contains an eight-port rotary selector valve (seven solvent ports and one vent port), a chemically compatible solvent dispensing pump, and various solenoid valves. These components are used to deliver all solvents into the Extractor Module, and solvents can be used for conditioning, washing, and/or eluting the SPE disk. Because each Extractor Module is independent, different solvents can be configured and used for a different Extractor Module.

A single controller PC can handle and operate up to four Extractor Modules. This modular approach permits up to 12 samples to be processed automatically. All four Extractor Modules can run the same method, or four different methods can be run on the four Extractor Modules. This allows greater flexibility such as when emergency samples need to be processed immediately.

You simply install the SPE disk, a collection vessel, and the water sample. The Extractor Module automatically introduces all necessary solvents, filters the water sample, rinses the sample bottle, and then extracts the analytes from the disk. The solvent vapor exhaust fan allows benchtop operation when connected to an exhaust system. Or, if desired, the Extractor Module can be operated under a fume hood.

| Specification | Description |
|---------------|--|
| Dimensions | 19 in. W x 19 5/8 in. D x 21 in. H (48.3 x 49.9 x 53.3 cm) |
| Weight | 61.7 lbs (28.0 kg) |
| Power | 24 VDC, 3.0 amp |

1.3 Controller PC Software

The software provides the programming logic and control for up to four Extractor Modules. As each module contains three stations, up to 12 samples can be processed simultaneously. A number of regulatory methods are stored in the software when it arrives. These preprogrammed methods can be used as they are provided, or modified. This allows you to create a variety of optimized methods to handle a full range of samples and disk configurations. Creating custom methods enables you to optimize the operating parameters to meet your unique requirements and/or take advantage of new SPE disk technology as it becomes available.

1.4 Product Safety Notice and Certification

1.5 General Safety



- Eye protection in the form of safety glasses or goggles is highly recommended when operating the SPE-DEX 5000 System. If solvents, 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 should be provided for protection. They should be worn at all times when operating the SPE-DEX 5000 System.



- Protection of the hands is essential when working with solvents or any hazardous material. 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.
- The SPE-DEX 5000 System is designed for benchtop or fume hood operation. If installed on a benchtop, the solvent bottles could be placed under a vapor vent exhaust fan. The end of the exhaust hose should be ducted into a local exhaust device 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.

- Do not work with volatile solvents without adequate ventilation from chemical fume hoods or other protective devices. The solvent waste bottle should also be safely vented.
- The SPE-DEX 5000 System is designed to use the common solvents used for SPE sample preparation. See *Acceptable Solvents* below for a list of the compatible solvents.
- The wastewater recovery container that should be used with the SPE-DEX 5000 System should be of sufficient size to contain the water waste. A 20-liter water waste recovery bottle (P/N 49-5010-01), is available from Horizon Technology.



WARNING

- To ensure user safety, a Water Inlet Valve (WIV) with a sample bottle must be installed on each station, prior to starting each run (see Figure 4-1, page 20). Operating stations without a WIV and sample bottle is not recommended and poses user risk.



- During solvent dispensing, the solvent is sprayed from the solvent nozzle, located on the Water Inlet Valve (WIV) platform. Be sure a WIV and sample bottle are in position before pressing *START* to avoid solvent being sprayed into the atmosphere and possibly causing injury to laboratory workers.

1.6 Chemical Safety

- A Material Safety Data Sheet (MSDS) or Safety Data Sheet (SDS) is 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. The MSDS can be obtained from the vendor.
- All hazardous solvents and chemicals must be disposed in accordance with appropriate Federal, state, and local regulations.
- Optionally, the SPE-DEX 5000 System can use an inert pressurized gas source (typically nitrogen) to maintain a nitrogen atmosphere over the SPE disk during the dry step. The recommended source is a dry grade of nitrogen gas. If a gas cylinder is used, safely secure the gas cylinder to avoid tipping, which may cause injury.



- The SPE-DEX 5000 System uses organic solvents that can pose inhalation, skin, and ingestion hazards with potential chronic health effects. Some of these solvents are also flammable, which could cause fire and/or explosion hazards. 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.
- The SPE-DEX 5000 System is not intended for use with Hydrofluoric Acid.

Acceptable Solvents

Acceptable solvents include acetone, acetonitrile, ethyl acetate, hexane, methanol, methylene chloride, MTBE, toluene, pentane, reagent water and isopropanol. Mixtures of these solvents and acidified (pH 2) or basified (pH 12) reagent water are also acceptable.

1: Introduction and Safety

Notes:

2 Theory of Operation

2.1 Solid Phase Extraction (SPE)

SPE is a cost-effective and efficient alternative to liquid-liquid extraction (LLE) and continuous liquid-liquid extraction (CLLE). Unlike these two techniques, which mix the water sample directly with an organic solvent, SPE filters the water sample directly through a specially modified filter or disk that contains a packing material such as C18, DVB, or HLB. It is during this filtration process that the analytes of interest are retained (concentrated) onto the packing material. Once the entire sample has been filtered, an appropriate solvent is used to extract the analytes from the disk. Because the filtration step is also a concentration step, only a very small volume of solvent is needed to effectively extract the analytes from the disk. There are many benefits of SPE, such as:

- Much less solvent is used.
- No emulsions are formed.
- Faster extraction times are possible.
- Solvent exposure to workers is reduced.
- SPE can be automated.

With these benefits, SPE also typically provides better recoveries than conventional manual extraction techniques.

2.2 SPE-DEX 5000 System

Designed to greatly simplify your SPE experience, the SPE-DEX 5000 System is completely self-contained and fully automated. This means all necessary pumps and valves are contained internally within each Extractor Module, which keeps the surrounding work area clear and accessible. Each Extractor Module contains an eight-port rotary selector valve (seven solvent ports and one vent port), a solvent-compatible liquid dispensing pump, and three flow-controllable vacuum pumps. Individual solenoid valves are used to direct the flow of all liquids and solvents.

The following description provides a complete overview of how the SPE-DEX 5000 System automatically operates.

2.3 The Sample Preparation Process

When using the SPE-DEX 5000 System, four major steps must be performed:

- Step 1: Condition the SPE disk with solvents.
- Step 2: Introduce and filter the water sample through the SPE disk.
- Step 3: Solvent rinse the sample bottle.
- Step 4: Extract analytes from the SPE disk.

Step 1: Condition the SPE Disk with Solvents

Once the SPE disk, water sample, and collection vessel have been loaded onto the Extractor Module, the SPE-DEX 5000 System is ready to run. The first step is to condition the SPE disk, as described below. The following four sub steps of the conditioning cycle are repeated with a series of solvents, as required by the analytical method. Though different solvents will be used, the purpose of the following sub steps is identical from one analytical method to the next.

3.1 Conditioning Solvent: There are two functions for the conditioning solvent. One is to completely remove any manufacturing impurities present in the SPE disk. Removing all impurities ensures that the SPE disk will not contribute to the background contamination. The conditioning reagent may activate or make the sorbent material more compatible with the sample composition and polarity. For example, methanol is typically used as the conditioning reagent for C18 packings. In this case, once the packing is activated, the SPE disk must not be exposed to air until the completion of the water sample filtration cycle.

Introducing the conditioning solvent is done by using an eight-port rotary selector valve and a chemically compatible liquid dispensing pump. The selector valve will rotate to the method-specified solvent, and the liquid dispensing pump will turn on. The pump will run for a specified time to deliver the desired volume of solvent. The conditioning solvent is directed into the down tube of the Water Inlet Valve (WIV). As the WIV will be closed (the water sample is sitting above the closed valve), the conditioning solvent is deflected down and into the SPE disk holder. The selector valve will automatically rotate to the vent port, and all remaining solvent in the line will be dispensed.

3.2 Saturating the Disk: With the solvent dispensed into the SPE disk holder, the saturate command allows the vacuum pump to quickly turn on and pull some of the solvent into the disk. This allows for all of the sorbent contained within the disk to come into contact with the reagent rather than just the top layer. The speed of each pump can be individually adjusted, if necessary, during the extraction process.

3.3 Conditioning Soak Time: The solvents are allowed to soak the SPE disk. Since different solvents have different extraction efficiencies, this parameter allows for variable soaking times (from 0 sec to 5 min) for each conditioning solvent introduced to the SPE disk.

3.4 Conditioning Drain Time: At the completion of the soak time, a conditioning drain time allows the solvent to be removed from the disk. Since different solvents permeate the disk at different rates, adjustable vacuum pump drain times allow for optimal performance. If the conditioning solvent is used to activate the SPE sorbent material, the Conditioning Drain Time would be set to a very short time (1 or 2 seconds) to still keep the disk damp, or set to zero to keep it completely wet. Control over this parameter ensures the disk does not go dry.

Step 2: Introduce and Filter the Sample through the SPE Disk

2.1 Process Sample Step: After conditioning the SPE disk, the WIV automatically opens and introduces the water sample onto the SPE disk. A liquid sensor monitors the presence of the water and will automatically turn on the vacuum pump.

During this filtration step, the analytes of interest are retained onto the sorbent material in the SPE disk. The cleanliness of the sample and the amount of total surface area of the SPE disk used will impact the rate of filtration. Therefore, to provide better control over the filtration rate, it is possible to control the vacuum level applied to the SPE disk holder, which in turn, controls the water sample flow rate through the SPE disk. Samples with few particulates will flow faster than samples with particulate material such as clay, dirt and sediment. Using the Fast Flow Disk Holder or 100 mm disks will typically flow faster than 47 mm and 50 mm disks due to their greater surface area.

As the sample is pulled through the disk by the vacuum, the water level in the disk holder drops and allows more water to flow out of the sample bottle and WIV down tube. This self-leveling, gravity feed design ensures the SPE disk stays “wet” with the sample until the entire sample has been processed. During the water sample step, the liquid sensor monitors the presence of the water. When the entire water sample has been processed and drops below the liquid sensor, the method advances to the next step.

During the water filtration step, more water sample can be delivered from the sample bottle only when the water level drops below the down tube opening. This action allows air to flow into the sample bottle, displacing more water sample, which then flows into the SPE disk holder. If an air leak occurs between the WIV and the sample bottle (due to a chip or crack in the glass), it could be possible for more water to flow out of the sample bottle than the vacuum pump can remove and an overflow condition could occur. To ensure this does not happen, a second liquid sensor—the Overflow Sensor—is constantly monitoring for this condition. If the Overflow Sensor detects the water sample, the WIV will automatically close and that specific extractor station will be paused. Once the problem is corrected, the station operation can be resumed. The Overflow Sensor ensures that a sample is never lost due to an issue with the sample bottle.

- 2.2 Air Dry or Nitrogen Dry Step:** After the water sample has filtered through the SPE disk, residual water will be present, as it will be retained within the pore structure of the SPE disk. The amount of residual water can be reduced by pulling room air or nitrogen through the disk. This is known as the Air Dry Step, and this time can be adjusted by the operator. The actual duration of this step depends on a number of factors, such as the disk being used, how clean or dirty the sample is, if prefilters were used, how dry you want the disk to be, etc.

Step 3: Solvent Rinse the Sample Bottle

- 3.1 Water soluble (polar) rinse solvent:** As with the conditioning step, the selector valve will rotate to the method specified extracting solvent and the liquid dispensing pump will turn on. The pump will run for a specified time to deliver the desired volume of solvent. The extracting solvent is directed to the WIV. As the WIV will be open, the extracting solvent is sprayed up and into the sample bottle. The solvent is allowed to drain down the sample bottle walls and drip into the SPE disk holder. The selector valve will automatically rotate to the vent port, and all remaining solvent in the line will be dispensed. It is important to note that a water soluble solvent is typically used as the first rinse for each method. The water soluble properties act to remove residual water from the sample bottle and the SPE disk. Removing residual water from the sample bottle walls allows the subsequent extracting solvent rinses to properly remove the analytes of interest.

This step uses several solvents to rinse the sample bottle and wash the extractor’s water sample path. Multiple rinses are used to ensure thorough washing of all surfaces. The rinse solvents drain down onto the SPE disk where the extraction occurs. The extractors automatically perform the rinsing without user intervention.

- 3.2 Water Insoluble (nonpolar) rinse Solvent:** A nonpolar solvent can be used to rinse the bottle and extract the analytes that are retained on the bottle interior. It can be used after the polar rinse described in 3.1 or instead of the step. The solvent will also extract analytes that may be retained on particulate captured on top of the SPE disk.

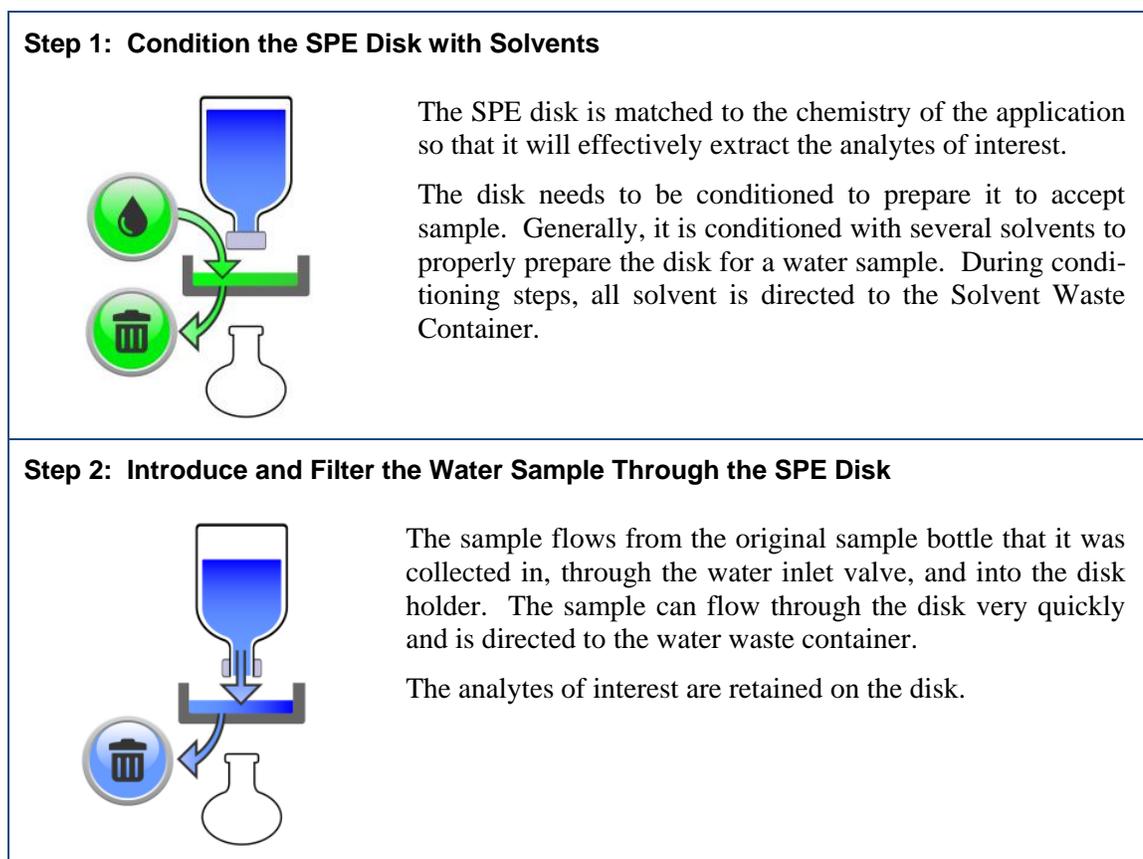
Step 4: Extract Analytes from the SPE Disk

The rinse solvents release the analytes from the SPE disk, allowing them to pass into the collection vessel. A series of rinse solvents are applied to the SPE disk to ensure the extraction of all analytes of interest. The volume of solvent delivered is determined by the method used.

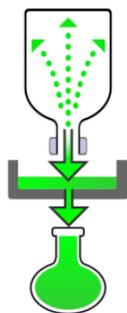
- 4.1 Saturate the Disk:** Once the solvent has been dispensed into the disk holder, the saturate command allows the vacuum pump to be quickly turned on, to pull some of the solvent into the disk. This allows for all of the sorbent contained within the disk to come into contact with the reagent rather than just the top layer.
- 4.2 Extraction Soak Time:** The solvents are allowed to soak the SPE disk. The soak time can be adjusted in the method to yield optimal extraction.
- 4.3 Extraction Collection Time:** This is the time the vacuum pump is turned on and run, and the solvent extract is pulled through the SPE disk and dispensed into the collection vessel. Since different solvents permeate the disk at different rates, the programming will allow for variable collection times for each extracting solvent introduced to the disk.

2.4 Graphical SPE Extraction Process Overview

When an extraction station is running, the software displays graphical icons to show the step that is running and make it easy to visually see what the extractor is doing. A typical extraction process is shown graphically below.

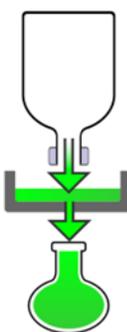


Step 3: Solvent Rinse the Sample Bottle



The sample bottle is rinsed with a stream of solvent that will be used for elution. This ensures that any analytes of interest that adhere to the glass bottle will be carried to the disk and eluted with the other analytes.

Step 4: Extract Analytes from the SPE Disk



Various solvents are used to elute the analytes of interest from the disk and into the collection vessel.

2: Theory of Operation

Notes:

Part I: Getting Started

3 Site Preparation and Unpacking

The *Customer Pre-Installation Form* should be consulted prior to delivery of the system to ensure a successful SPE-DEX 5000 System installation. Contact a Horizon Technology representative if you need this document or have any difficulty fulfilling the requirements.

3.1 Preparing the Site

When setting up the SPE-DEX 5000 System, follow the *Customer Pre-Installation Form* directions and verify:

- Adequate space for solvents, samples, and waste containers
- Ventilation
- Solvents
- Power source suitable for installation
- Nitrogen gas source (optional)

3.2 Facility Requirements

Be sure the site for the SPE-DEX 5000 System meets the facility requirements outlined below.

| Requirement | Specification | | |
|---------------------|---|-----------------|-------------------------------|
| Physical Properties | Dimensions for the SPE-DEX 5000 System: 19 in. W x 19 5/8 in. D x 21 in. H (48.3 x 49.9 x 53.3 cm) Extractor: 61.7 lbs. (28.0 kg) | | |
| Bench Space | Each instance of software can operate up to four extractor modules. At least one inch between each extractor is recommended. A counter depth of 30 inches is preferable but at least 24 inches is required. The table on which the SPE-DEX 4790 System sits must accommodate the combined weight of the Extractor Modules and the Controller PC. | | |
| Gas (optional) | A clean, dry regulated source of nitrogen. The source can be either a tank and regulator, or house nitrogen. The pressure must be a minimum of 60 psi and a maximum of 80 psi. 1/8-inch male NPT fittings are supplied, which will adapt to most nitrogen regulators. The connection also can be made using a custom-supplied 1/8-inch Swage fitting. | | |
| Ventilation | Based on the solvents to be used, each laboratory must decide to operate either on the benchtop or in a fume hood. | | |
| Power | Module | 24 VDC, 3.0 amp | |
| | Power Supply | Line Voltage | 100-240 VAC (to power supply) |
| | | Line Frequency | 50-60 Hz |
| | | Maximum Power | 300 Watts |
| | Uses an IEC type connector to accept various international power cords. | | |
| SPE Disks | 47 mm, 50 mm, or 90 mm disks and/or prefilters. Atlantic® SPE disks and prefilters are available from Horizon Technology and are certified for automation. | | |

3: Site Preparation and Unpacking

| Requirement | Specification |
|------------------|--|
| Solvents | Solvents are required to condition and elute the SPE disks. Typically, methylene chloride, ethyl acetate, hexane, methanol, acetone, reagent water, etc., are required, depending on the method specified. |
| Waste Containers | Two waste containers are required. The waste containers can be any chemically compatible container or carboy. The water waste container needs to be of a sufficient volume to contain the volume of water waste that will be generated. Both containers need to be located lower than the equipment. Due to the weight of the water waste container, it is advisable for this to be placed on the floor. |
| Environmental | Ambient Temperature: 17 to 28 degrees C Ambient Humidity: 10 to 70% relative humidity non-condensing Indoor use only |
| PC Parameters | Processor: (min.) 4th Generation Intel Core i3, or currently available equivalent, 32 or 64 bit Operating System: Windows 7 Professional, 32 or 64 bit Memory: 4 GB DDR (min) Screen: 20" or greater HD LCD for Desktop; 15" or greater for Laptop Graphics: Integrated Hard Drive: (min.) 250 GB 5400 RPM SATA Networking: Ethernet 100 Base T or greater Sound: Required for Alerts Software: Adobe Reader Available USB: 2.0 Port |

3.3 Unpacking the SPE-DEX 5000 System

Follow the steps below when unpacking the SPE-DEX 5000 System. Call a Horizon Technology representative for any assistance required for your installation.

Procedure

- Step 1:** The SPE-DEX 5000 System is delivered in a shipping box. Move the box into the general area where the System is to be set up.
- Step 2:** If several people are available to help unpack the unit, open the top and lift the unit from the box. If one person is initiating this step, position the box, so it is on its side. Slip the unit out of the box and turn the unit upright.
- Step 3:** Two people should lift the unit onto the benchtop



Figure 3-1. Lift the Unit from the Box

- Step 4:** Remove all of the foam pieces.

Step 5: Carefully lift the foam pieces off the sides of the Extractor Module.



WARNING

Due to the weight of the Extractor Module, have two people lift the Extractor Module into place.



Figure 3-2. Removing the Foam Protection

Step 6: Carefully remove the foam piece from above the disk holder platforms. See Fig 3-2 above.

Step 7: Use the checklist in Section 3.4 to ensure that all of the Extractor Module components have been received.

3: Site Preparation and Unpacking



Figure 3-3. Extractor Module Unpacked and Sitting on the Benchtop



Figure 3-4. Vapor Shield option for a station

3.4 Unpacking Checklist

Controller PC

If ordered, check the Controller PC kit (P/N 63-2566) for the following items:

| | |
|--------------------------|--------------|
| <input type="checkbox"/> | PC |
| <input type="checkbox"/> | Power Supply |
| <input type="checkbox"/> | Power Cord |

Extractor Module

Check the Extractor Module kit for the items listed below. They may be contained in several boxes.

| | Component | Part Number |
|--------------------------|---|-------------|
| | Utility Kit | 49-5004-01 |
| <input type="checkbox"/> | Solvent and Water Waste Lines | 50-0225 |
| <input type="checkbox"/> | WIV Wrench | 02-5792 |
| <input type="checkbox"/> | Thermistor Height Tool | 02-3170-05 |
| <input type="checkbox"/> | Hex Wrench | 99-0880 |
| <input type="checkbox"/> | Software and Users Guide CD | 29-3000-01 |
| <input type="checkbox"/> | Software and Users Guide USB | 29-3001-01 |
| <input type="checkbox"/> | Collection Vessel Retaining Clip(s), blue, three (3) | 22-0687 |
| <input type="checkbox"/> | Elution Tube Assembly | 50-5010-01 |
| <input type="checkbox"/> | 20 L Water Waste Recovery Bottle | 49-5010-01 |
| <input type="checkbox"/> | 2.5 L Solvent Waste Recovery Bottle | 50-5025 |
| <input type="checkbox"/> | Start-Up Kit | 50-5041 |
| <input type="checkbox"/> | Water Inlet Valves (WIV), three (3) | 50-5792 |
| <input type="checkbox"/> | 24 Volt Power Supply | 26-2250 |
| <input type="checkbox"/> | Power Cord for Power Supply | Regional |
| <input type="checkbox"/> | USB Communication Cable | 14-2802-02 |
| | Optional -Solvent Delivery Tubing Kit (1 needed to operate system) | |
| <input type="checkbox"/> | Solvent Lines without caps | 49-5003 |
| <input type="checkbox"/> | Solvent lines with caps (GL-45) and bottles | 49-5002 |
| <input type="checkbox"/> | Solvent lines with caps (38-430) and bottles | 49-5001 |
| | Optional - Disk Holders (3 needed to operate the system) | |
| <input type="checkbox"/> | 47 mm Disk Holder with riser | 50-5018-01 |
| <input type="checkbox"/> | Fast Flow Disk Holder | 50-2767 |
| <input type="checkbox"/> | 100 mm Disk Holder | 50-0809-01 |
| | Optional- Elution Glassware (3 needed to operate the system) | |
| | VOA Vial Adapter, 19/22 | 160-0001 |
| | Erlenmeyer, 125-mL, 19/22, with stopper | 27-0476-01 |
| | Erlenmeyer, 250-mL, 19/22 | 03-5000-01 |

3: Site Preparation and Unpacking

| | |
|---|------------|
| <input type="checkbox"/> Optional- One Pass Kit carbon cartridge holder, liquid lines and glassware for one-pass process for volatiles/more polar compounds | 49-2794 |
| <input type="checkbox"/> Optional-Vapor Duct Hose Kit (for operation outside a hood) | 49-5006-01 |
| <input type="checkbox"/> Optional- Vapor Shield for shielding one station from escaping vapors or allowing a nitrogen blanket to be used for sensitive samples | 50-5012 |
| <input type="checkbox"/> Optional- Nitrogen Supply Line (Needed if nitrogen gas is to be used), includes internal components for nitrogen handling installed at the factory, also includes jumper for multiple units connected to one nitrogen source | 50-5085 |
| <input type="checkbox"/> Optional-USB Hub to connect multiple SPE-DEX 5000 Modules to one PC | 63-2820-01 |

Additional Requirements

The following items are also required to complete the installation. These parts need to be supplied by the customer.

| | |
|---|--|
| <input type="checkbox"/> Solvents to Perform the Proper Chemistry. | |
| <input type="checkbox"/> SPE Extraction Disks: 47 mm, or 90-mm size Prefilters are also recommended for handling samples with particulates | |

Optional Accessories

| | |
|---|--|
| <input type="checkbox"/> Gas Supply of Dry Nitrogen, minimum 60 psi (maximum 80 psi) | |
| <input type="checkbox"/> Cap Adapters. Contact Horizon Technology for correct sizing and part number for the bottles you want to use. | |

Notes

4 System Components

Familiarize yourself with all parts and functions of the SPE-DEX 5000 System before you start to use it. This section provides an overview of Extractor Module and software features.

4.1 Extractor Module Components: Front

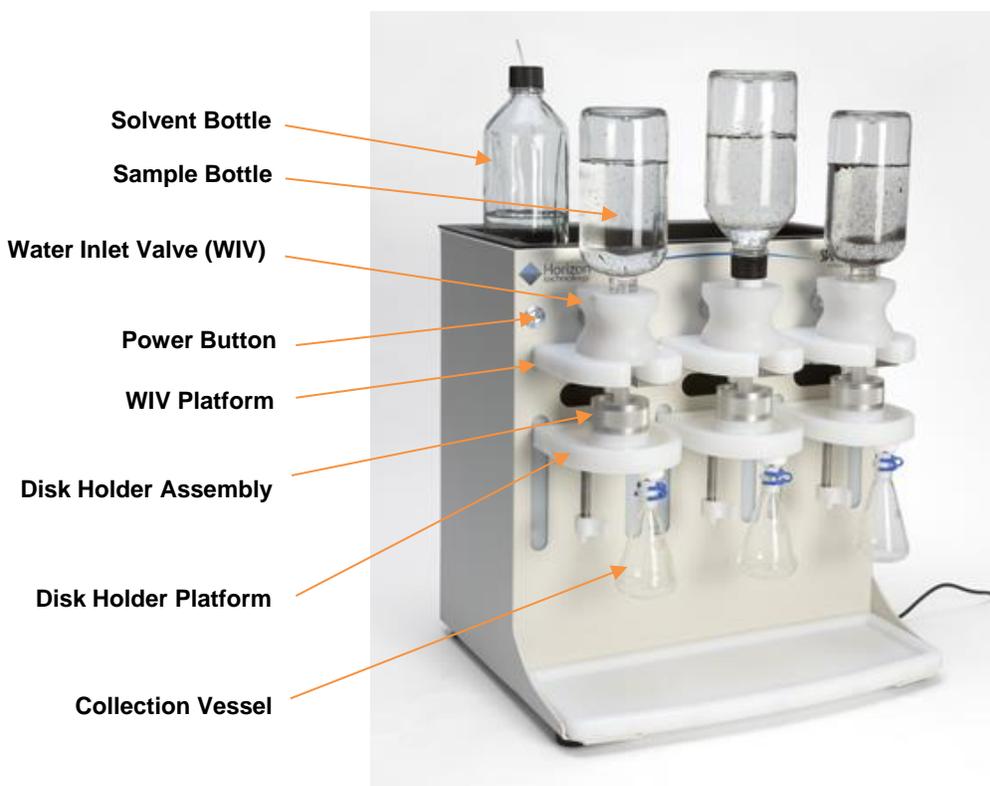


Figure 4-1. Front of the SPE-DEX Extractor Module

The major components on the front of the Extractor Module are:

Solvent Bottle

A bottle that holds a solvent to be used.

Sample Bottle

A bottle that holds the water sample to be processed. The Extractor Module can accommodate a wide range of sample bottles. With the use of optional cap adapters available from Horizon Technology, original sample collection bottles with openings ranging in size from 33 x 400 mm to 89 x 400 mm can be used directly on the Extractor Module. The original sample bottle is used, as the extracting solvent will be automatically sprayed into the bottle to wash any retained analytes off the interior of the bottle.

Water Inlet Valve (WIV)

A stop cock type valve that will automatically open to allow the water sample to be dispensed into the Disk Holder Assembly.

Power Button

Power button which turns the module on/off. The button will light when power is on.

| | |
|----------------------|--|
| WIV Platform | A support platform used to hold the WIV and sample bottle. This platform also houses the Solvent Delivery Port, which is used to deliver solvent to the SPE disk holder, or the sample bottle. |
| Disk Holder Assembly | A single or a multi-piece assembly that holds and seals the SPE disk during the sample preparation process. |
| Disk Holder Platform | An adjustable platform that holds the disk holder assembly. The platform can be adjusted to various heights, based on the holder assembly or extraction device used. |
| Collection Vessel | A vessel used to collect the extracting solvent from the SPE disk. As the extracting solvent does not require a closed system to be dispensed into the elution vessel, any open container can be used. However, if a sealed collection vessel is desired to minimize solvent vapors escaping from the vessel, a 19/22 taper container can be used and attached to the taper fitting. |

4.2 Liquid Sensors and Solvent Delivery Port Area Components

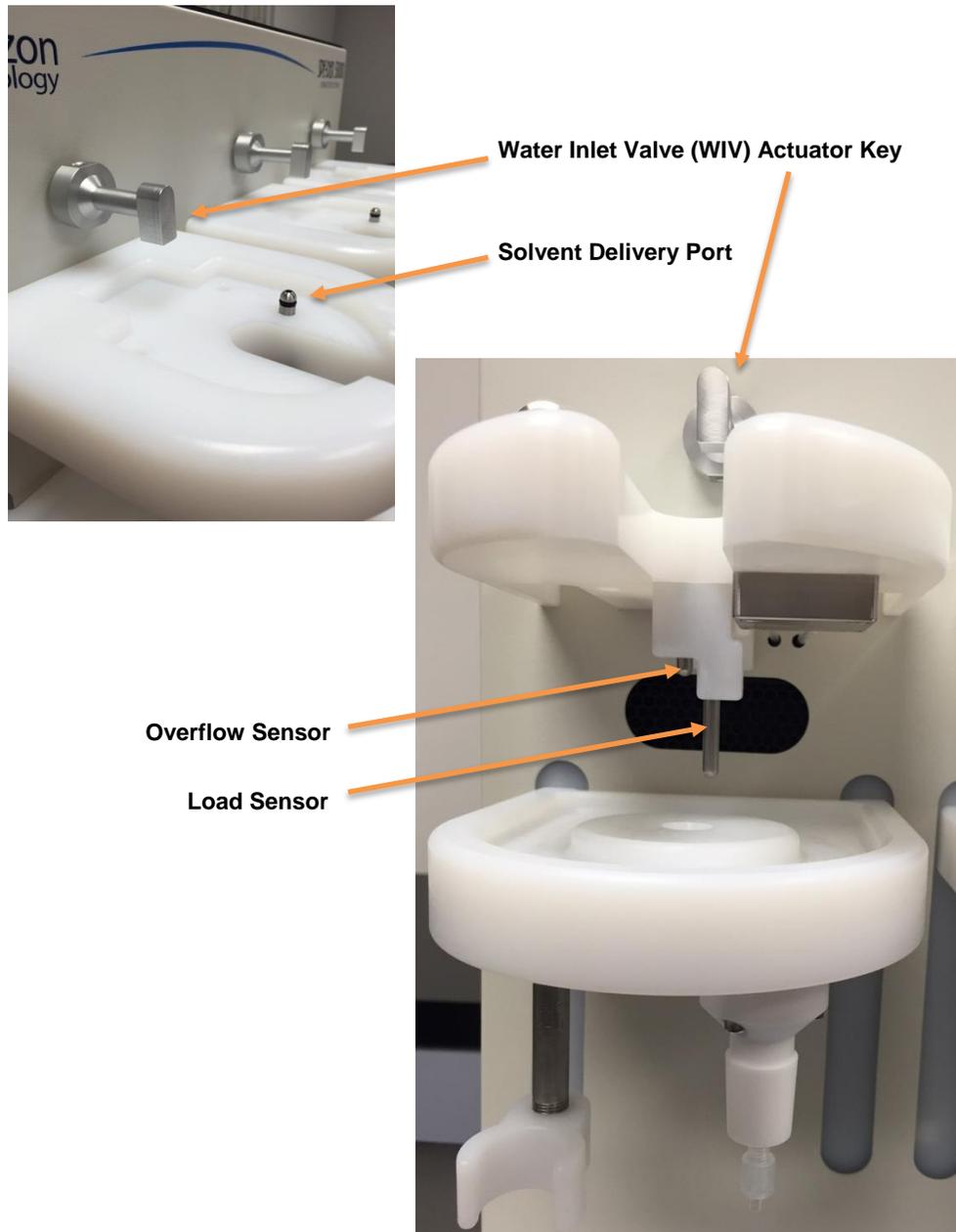


Figure 4-2. Detail of the Liquid Sensors and Solvent Delivery Port Area

The major components in the Liquid Sensors and Solvent Delivery Port area are:

WIV Actuator Key

A component that keys into the WIV assembly and opens and closes the WIV to introduce the water sample onto the SPE disk.

Solvent Delivery Port

The port from which all solvent is dispensed. When the WIV is closed, the solvent will be directed to the SPE disk holder. When the WIV is open, the solvent will be directed up and into the sample bottle.

- Overflow Sensor** A liquid sensor used to detect if the water sample is close to overflowing the SPE Disk Holder. If the water sample is detected, the WIV automatically closes and a warning message is displayed. When the problem has been corrected, the station can be resumed to complete processing the sample.
- Load Sensor** A liquid sensor used to monitor the presence of the water sample, and monitor when the entire water sample has been filtered through the SPE disk. When the entire sample has been processed, the method will advance to the next step.

4.3 Collection Vessel Area Components

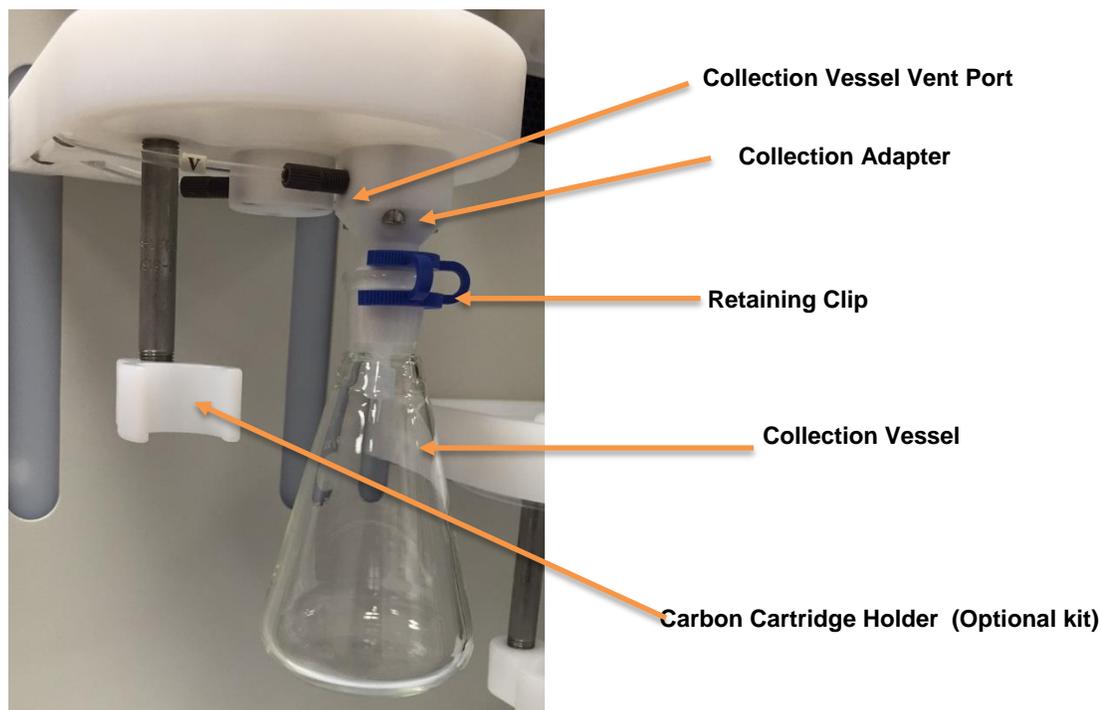


Figure 4-2. Detail of the Collection Vessel Area

The major components in the Collection Vessel area are:

- Collection Vessel Vent Port** A port that directs solvent vapors in the Collection Vessel, out the Solvent Vapor Exhaust Port, located on the backside of the module. This allows all solvent vapors to be contained.
- Retaining Clip** A clip that secures the 19/22 taper glass collection vessel to the tapered Collection Adapter.
- Collection Vessel** A vessel that collects the eluting solvent.
- Collection Adapter** An adapter used to attach a 19/22 tapered glass collection vessel.
- Carbon Cartridge Holder** A holder used to support a 20 cc Carbon Cartridge (optional kit).

4.4 Extractor Module Components: Rear

As shown in Figure 4-3 on the next page, the major components on the rear of the Extractor Module are:

| | |
|--|--|
| Solvent Tubing Guides | Guides that keep the tubing from the Rotary Selector Valve neat and organized. |
| Solvent Drip Guard | A guard that protects the Communication and Power Supply ports from unintentional solvent spills. |
| PC Communication Port | A port that connects the PC communication cable to the Extractor Module. |
| Power Supply Port | A port that provides power to the Extractor Module. |
| Solvent Vapor Exhaust Fan | A fan that connects a vapor exhaust hose to the Extractor when used in benchtop mode without a fume hood. |
| Water Waste Ports, Solvent Vapor Vent, and Solvent Waste Ports | Ports that connect the solvent and water waste lines to the Extractor Module. The Solvent Vapor Vent allows the solvent vapor to be contained. |
| Solvent Inlet Valve | A rotary selector valve used to deliver solvents to the Extractor Module. |
| Inert Gas Inlet | A port that allows nitrogen gas to be used, in place of room air, to remove residual water from the SPE disk. |
| Solvent Inlet | Connects the rotary selector valve to the module. |

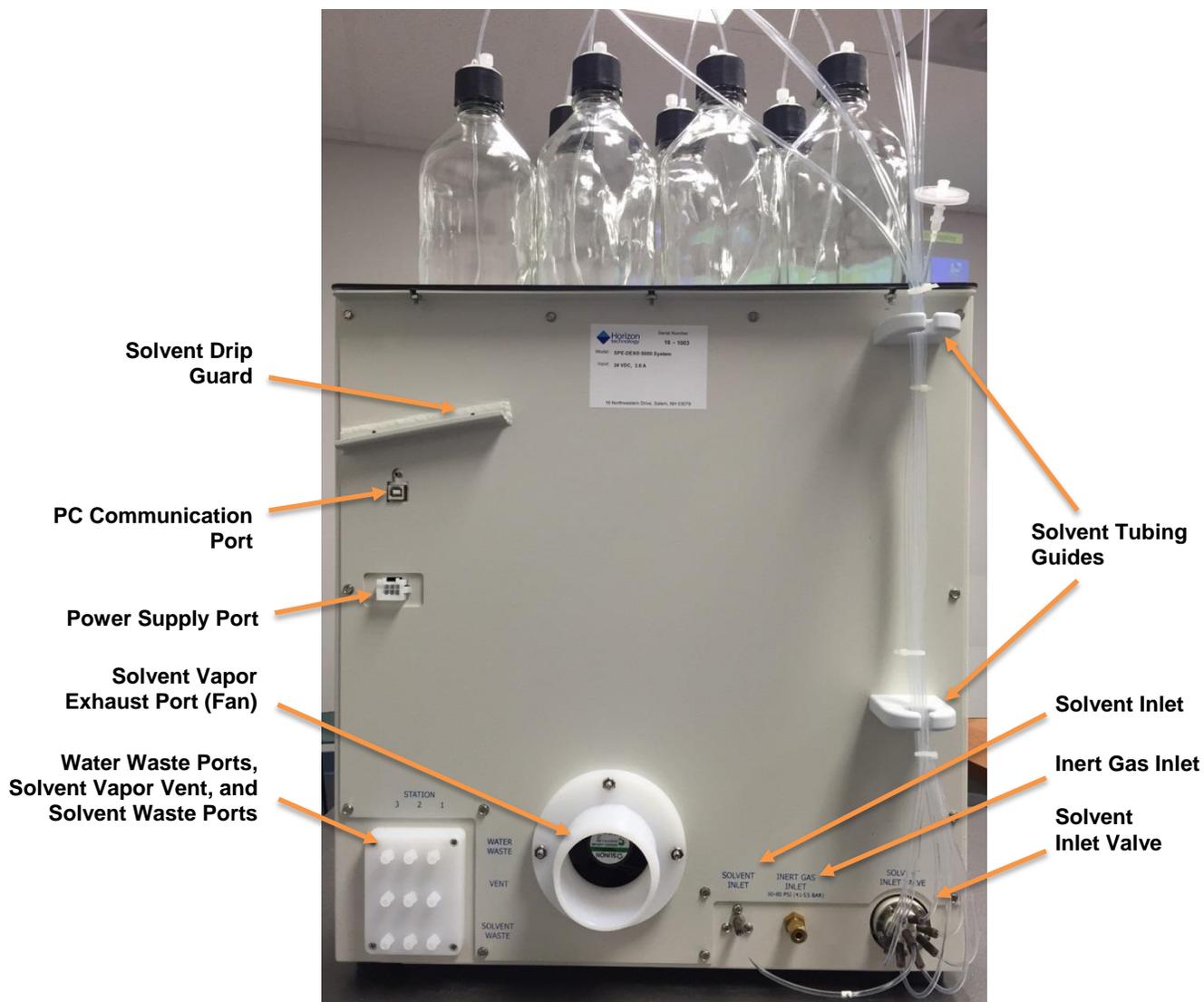


Figure 4-3. Rear of the SPE-DEX 5000 System

4: System Components

Notes:

5 System Installation

Installing the SPE-DEX 5000 System involves three procedures:

- Installing the SPE-DEX 5000 Extractor Module, Section 5.1, below
- Installing the SPE-DEX 5000 Software, Section 5.5, page 34
- Verifying the Initial System, Section 5.6, page 37

5.1 Installing the SPE-DEX 5000 Extractor Module

The SPE-DEX 5000 Extractor Module is capable of operating either on a benchtop (with the Solvent Vapor Exhaust Kit, P/N 49-5006-01) or within a laboratory fume hood. Using the Extractor Module on the benchtop saves valuable fume hood space.

5.2 Prerequisites

A workspace at least four feet wide is required to accommodate the Extractor Module, accessories and necessary supplies. The solvent bottles can be placed on top of the Extractor Module in the solvent container tray (similar to an HPLC) or next to the unit. This will save bench space and keep the solvent containers away from the working surface. The solvent recovery bottle can be placed close to the Extractor Module on the benchtop, while the water waste recovery bottle (possibly 20L capacity) should be placed below the system. If the Nitrogen Purge Kit (P/N 50-5085) is used, a suitable nitrogen source will be needed.

If the Solvent Vapor Exhaust Kit is used, ten feet of solvent vapor exhaust hose will require that the Extractor Module be located close to the laboratory's exhaust system.

5.3 Tools Required

The following tools are required for installation:

- Adjustable wrench
- Flat edge screw driver

5.4 Installation Procedure

- Step 1:** Carefully turn the Extractor Module so the backside of the module is facing you, as shown in Figure 4-3 on page 23.
- Step 2:** Locate the Solvent and Water Waste Line Kit (P/N 50-0225), which contains a total of six lines: three for the solvent waste and three for the water waste.
- Step 3:** Gently connect the three solvent waste lines to the bottom row of port fittings on the back, lower left side of the Extractor Module. Only finger-tighten these fittings. See Figure 5-1.
- Step 4:** Locate the 2.5 liter glass bottle, and manifold cap assembly. This bottle is used to collect the waste solvent. Direct the three solvent waste lines to the manifold cap assembly, and gently connect the caps to the manifold fittings. See Figure 5-1.

5: System Installation

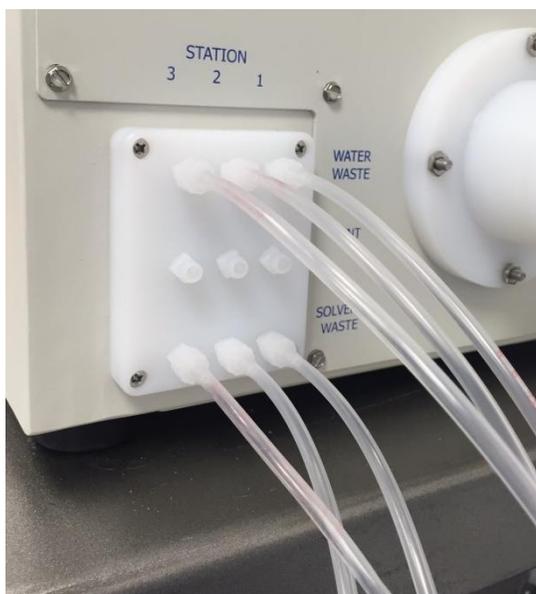


Solvent Waste Bottle



Figure 5-1. Three Solvent Waste Lines

- Step 5:** Gently connect the three water waste lines to the top row of port fittings on the back, lower left side of the Extractor Module. Only finger-tighten these compression fittings. See Figure 5-2.
- Step 6:** Locate the 20 Liter carboy (if purchased with the system) and the manifold cap assembly. Direct the three water waste lines to the manifold cap assembly, and gently connect the caps to the manifold fittings. See Figure 5-2.



Water Waste Bottle



Figure 5-2. Three Water Waste Lines

- Step 8:** Locate the Solvent Delivery Tubing Kit (P/N 49-5001, 49-5002, or 49-5003). This kit contains:

- A. One short piece of tubing with compression fittings on both ends. This line is approximately six inches (15.2 cm) in length.
- B. Seven 1/8-inch solvent lines. These lines are approximately three feet (91 cm) in length and have a number tag. For example:



Figure 5-3. Line Tag

- C. One 1/8-inch vent line with an attached in-line filter. This line is approximately one foot (30 cm) in length.
- Step 9:** The Solvent Inlet Valve is located on the back side of the Extractor Module, in the lower right corner. Remove the protective cap that covers the valve ports.
- Step 10:** Reference Step 8: item A. Just finger tighten the fitting part way into the center 1/4-28 port of the Solvent Inlet Valve. Make sure the tubing is pushed all the way into the port, then screw in the fitting until you feel an increase in resistance, which signifies that the fitting has contacted the bottom of the port. Refer to Figure 5-4 on page 29.
- Note: Extreme overtightening can result in loss of sealing capacity.**
- Step 11:** Just finger tighten the other end of this line to the Solvent Inlet port, which is located just to the left of the Solvent Inlet Valve.
- Note: Extreme overtightening can result in loss of sealing capacity.**
- Step 12:** Reference Step 8: item B. Locate the solvent line numbered with the “1” tag. Just finger tighten this line to port 1 on the Solvent Inlet Valve.
- Note: Extreme overtightening can result in loss of sealing capacity.**
- Step 13:** Connect the solvent line numbered “2” to port 2. Continue this procedure until all seven lines are connected. Refer to Figure 5-4.
- Step 14:** Reference Step 8: item C. Connect the vent line to port 8.
- Step 15:** Carefully feed all eight lines through the two Solvent Tubing Guides located on the right side of the back panel. For now, simply lay the lines in the Solvent Tray, located on the top of the Extractor Module. Refer to Figure 5-5.

5: System Installation

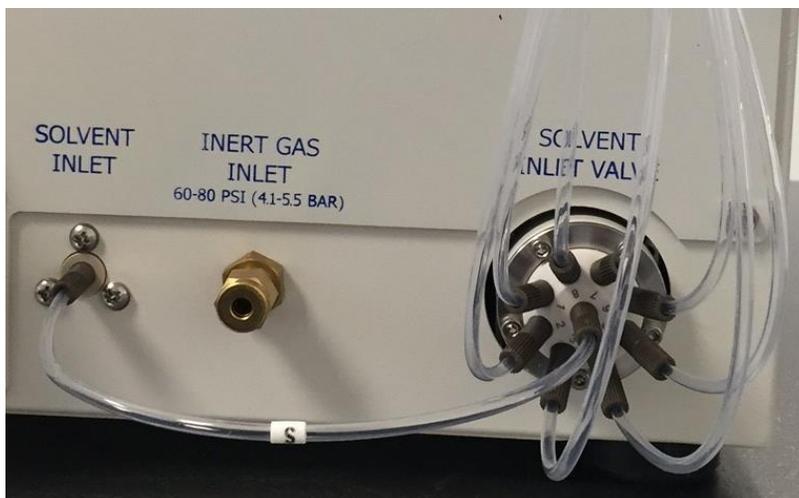


Figure 5-4. Connected Lines



Figure 5-5. Connected Lines

- Step 16:** If the optional Nitrogen Gas Purge Kit is to be used, connect the nitrogen gas line to the Inert Gas Inlet port. This port is located between the Solvent Inlet port and the Solvent Inlet Valve.
- Step 17:** Locate the solvent vapor exhaust hose. The standard length provided with the system is 10 feet. Center the hose and push it onto the exhaust port (refer to **Figure 5-6**). Use the hose clamp with the worm screw to secure the hose to the exhaust port. Extend the length of the hose to the appropriate hood or vent.



Figure 5-6. Exhaust Port at the Bottom Rear of the SPE-DEX 5000 System; Photo on the Right Shows the Exhaust Hose Installed

- Step 18:** Locate the module Power Supply. Connect the power cord to the 24V DC Power Adapter. Attach the 24 VDC cable to the backside of the module.

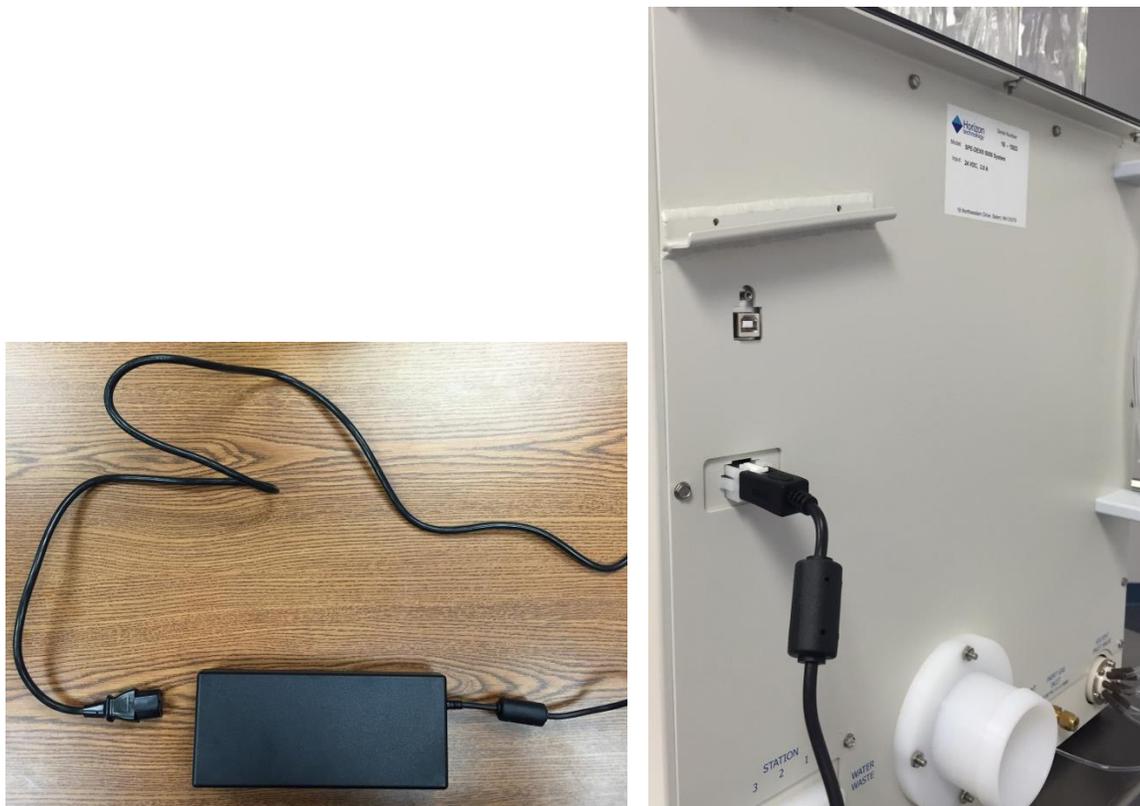


Figure 5-7. Power Supply Connected to the Power Supply Port

5: System Installation

Step 19: Attach the power cord to the 120 or 240 VAC outlet.

Step 20: Locate the PC Communication Cable. Connect the cable to the PC and to the PC Communication Port on the back side of the module. Tighten the screw on the cable connector.



Figure 5-8. PC Communication Cable Connected to the PC Communication Port

Step 21: Carefully turn the Extractor Module so the front side of the module is facing you.

Step 22: Locate the Elution Dispensing tubes (P/N 50-5010-01). Screw them carefully into the bottom of the collection adapter, finger tight, on each station as shown in Figure 5-9.

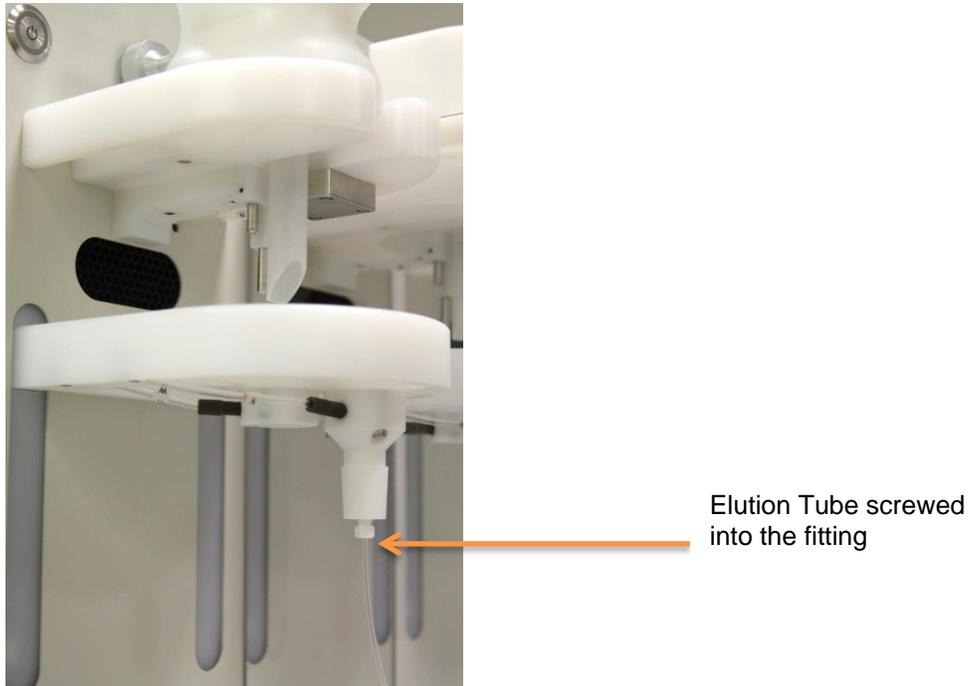


Figure 5-9. Elution tube installed on Station 1

Up to four SPE-DEX 5000 modules can be connected to the same controller. If additional SPE-DEX 5000 modules are to be installed, set each one up following the procedures in Steps 1-22 with the following exceptions:

In step 20, instead of connecting the PC communication cable directly to the PC, connect it to the USB Hub (P/N 63-2820-01) and then connect the USB Hub to the PC. For each additional SPE-DEX Module, also connect the communication cable from the Module to the USB Hub.

If the optional nitrogen kit is used (P/N 50-5085), the extra tubing will allow you to connect up to four SPE-DEX 5000 modules to one nitrogen source. See the kit for instructions.

This completes the SPE-DEX 5000 System installation. Continue by installing the SPE-DEX 5000 software, as described in the next section.

5.5 Installing the SPE-DEX 5000 Software/Establishing Communication with the Extractor Module

Step 1: Press the power button located on the upper left corner of the Extractor Module. A blue light will appear when the power is on.



Figure 5-9. Power Button; on the Right the Button is Lit Indicating the Power is ON

Step 2: Turn on the PC.

Step 3: After the PC has booted up, insert the software CD or USB memory stick into the appropriate drive on the PC.

Step 4: The installation software should launch automatically (CD only). If it does not, run the **Setup.exe** file located on the CD or USB memory stick.

Step 5: Follow the instructions on the screen. The software will install automatically. When completed, the Communication Port Manager screen will be displayed:

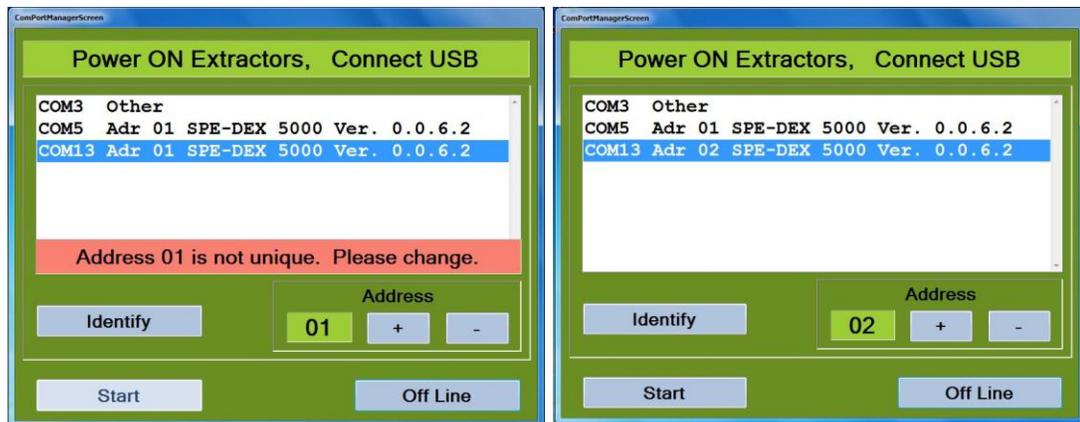


Figure 5-10. Communication Port Manager Screen with Conflicting Address and Resolution

5.5: Installing the SPE-DEX 5000 Software/Establishing Communication with the Extractor Module

Step 6: Review the Communication Port Manager screen. It should show at least one communication port with an address (Adr 1 in the example in Figure 5-10) and the module firmware version.

If more than one Module is connected, initially the addresses may have to be changed so they are unique. Clicking on the line with the conflicting address and using the plus and minus keys to choose a different address will resolve the situation and allow the **Start** button to be accessible.

If more than one Module is connected to the PC, select the first Module in the list, and click the **Identify** button. The Power Switch on the first Module will flash. Based on how the Modules are arranged on the bench top, you can assign each Module the proper address, so the Modules will be arranged in the correct order.

Step 7: Click **Start**. The Start Modules screen is displayed (in this case only one module is connected):

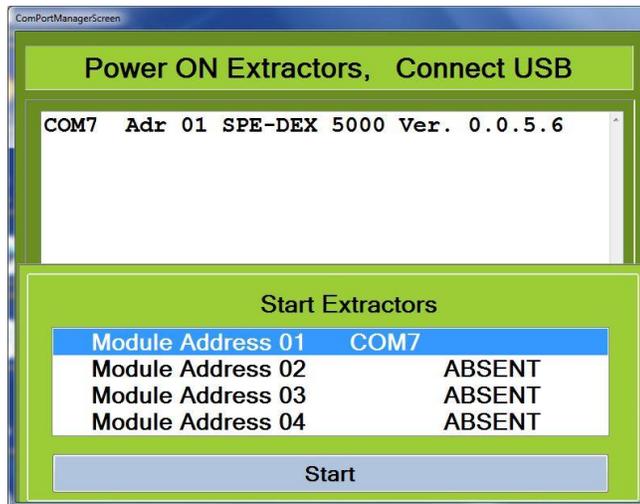


Figure 5-5. Start Modules Screen

Step 8: Click **Start**.

The SPE-DEX logo screen is displayed briefly, indicating the software version:

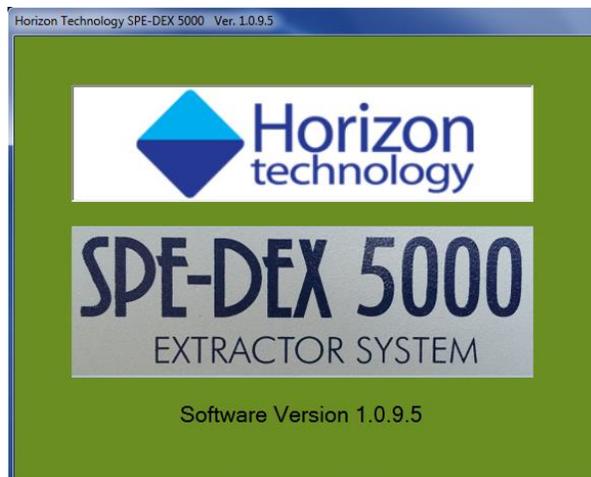


Figure 5-12. SPE-DEX 5000 Screen

5: System Installation

The Overview screen is displayed. In this example, (Figure 5-13) one module is connected:

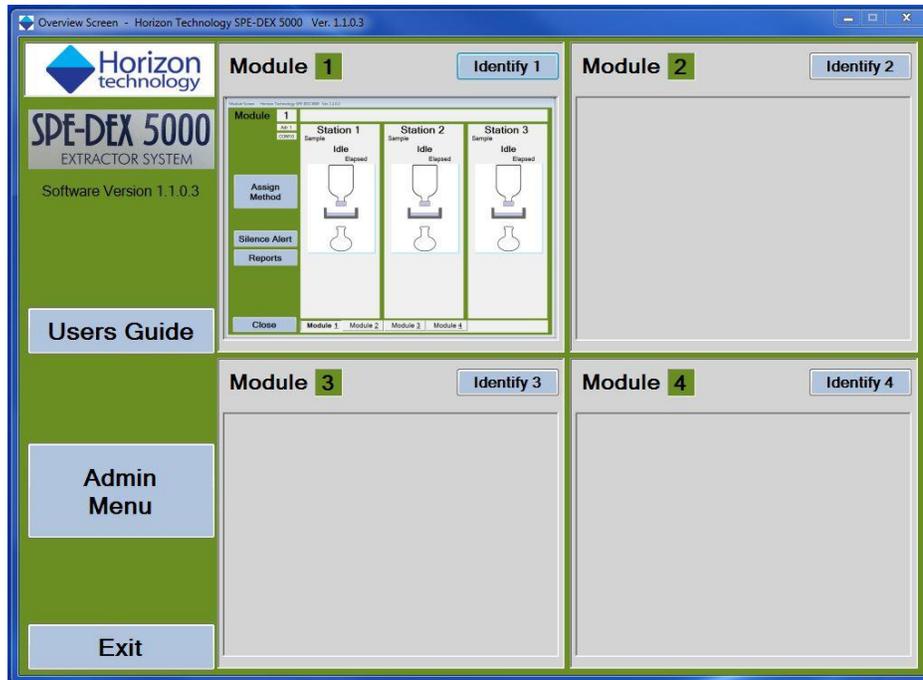


Figure 5-13. Overview Screen with One Module Connected

If more than one module is connected, each module will display on this screen. In this example, (Figure 5-14) three modules are connected.

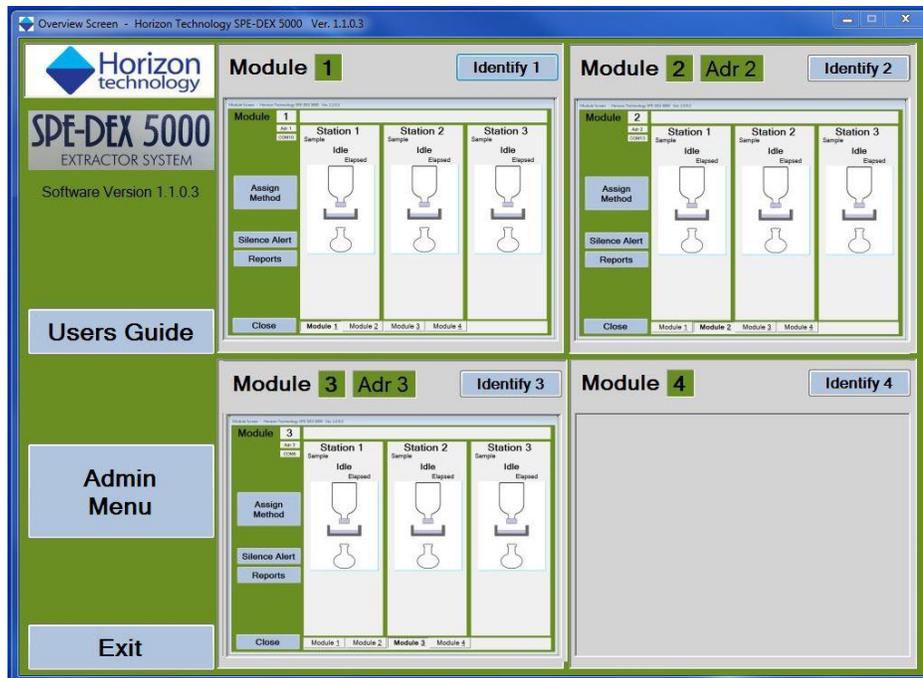


Figure 5-14. Overview Screen with Three Modules Connected

Once the Overview screen is displayed, you have established communication. Continue by verifying the initial system, as described in the next section.

5.6 Verifying the Initial System Connections

If you are uncertain about matching the module locations with their display on the Overview screen, you can click the **Identity** button. For example, you might click **Identity 2** (Figure 5-15) to determine the module to which the Module 2 Overview display is associated. The power button on Module 2 will flash so you can see which module is associated with entries in Module 2 software area.

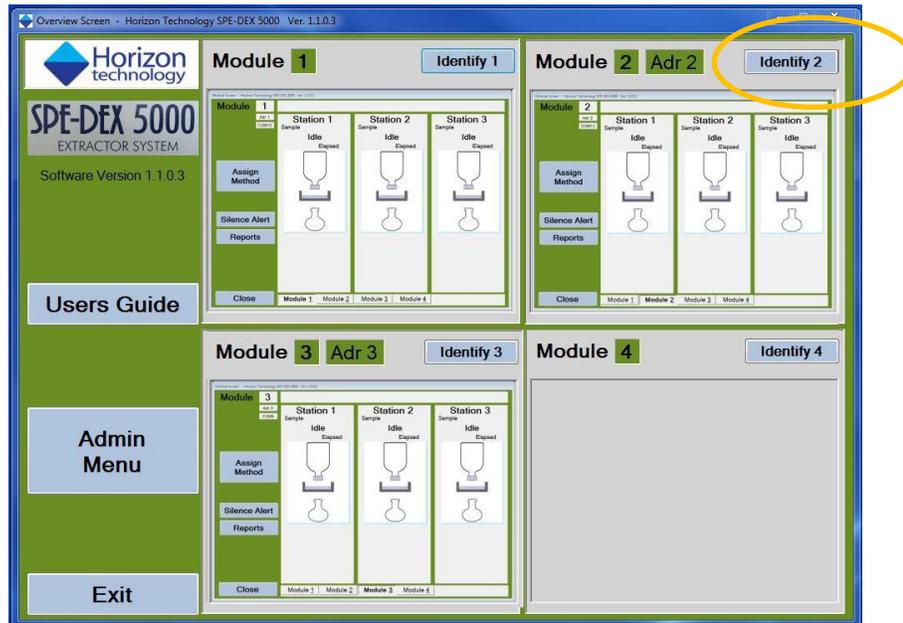


Figure 5-15. Identify Button

Each module needs a unique address. In Figure 5-15, the address of Module 1 is Adr 1, Module 2 is Adr 2, and Module 3 is Adr 3. If the modules have unique addresses, you have verified the initial system. If the addresses are not unique, complete the following procedure.

Step 1: Click **Admin Menu**. The Admin Menu screen is displayed in Figure 5-16:

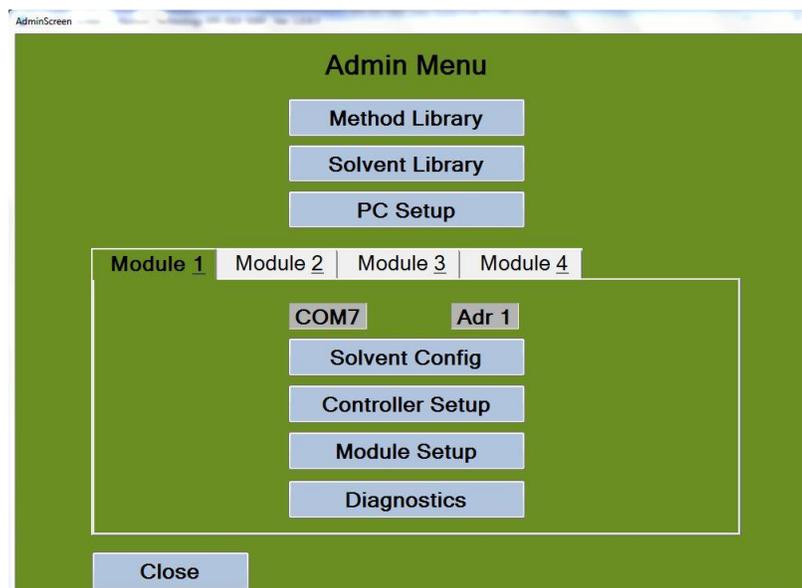


Figure 5-16. Admin Menu Screen

5: System Installation

- Step 2:** Click the module that you want to verify or change. In the example in Figure 5-16, Module 1 is selected. Notice that the module's Com port and Adr are indicated.
- Step 3:** Click **Module Setup**. The Module 1 Setup screen is displayed as shown in Figure 5-17:

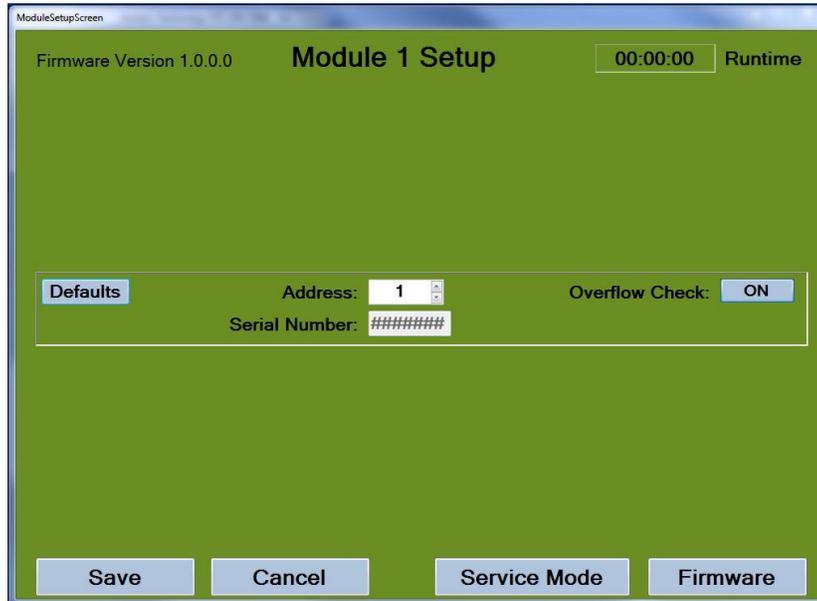


Figure 5-17. Module Setup Screen

- Step 4:** Using the Address up/down arrows, change the module address, as necessary.
- Step 5:** Click **Save**. Clicking Save will bring you back to the main Admin Menu screen.
- Step 6:** Then, check the other modules, as necessary. Setting each individual address, allows the modules to be sequentially identified once on the bench top.

Notes

6 Basic Operation

6.1 Setting Up the Hardware for Operation

The setup procedure is the same for all types of methods, with three exceptions: the content of the sample bottle, the disk holder assembly, and the SPE disk. These differences are noted within the procedure. Each method requires the use of a collection vessel.

Step 1: Be sure the system is set up, as described in Chapter 5, *System Installation*, starting on page 27. Ensure that:

- All solvent lines are connected. To ensure the solvent lines and sinkers are clean after the initial setup, but before the equipment is used, see section 8.1.1 for the cleaning procedure that should be used
- The Extractor Module is powered on.
- The PC is powered on with the SPE-DEX 5000 software (Overview screen) displayed.
- The system communication has been verified.
- The scroll wheel on the mouse will scroll some of the entry choices in the software, take care to ensure you are not making unintended changes in methods or solvent configurations.
- The overflow sensors are set to the appropriate height to match the disk holders in use. The setting will need to be re-set when different disk holders are installed in the future.

The Thermistor Sensor Height tool is shown in Figure 6-1, next to the supplied hex wrench.

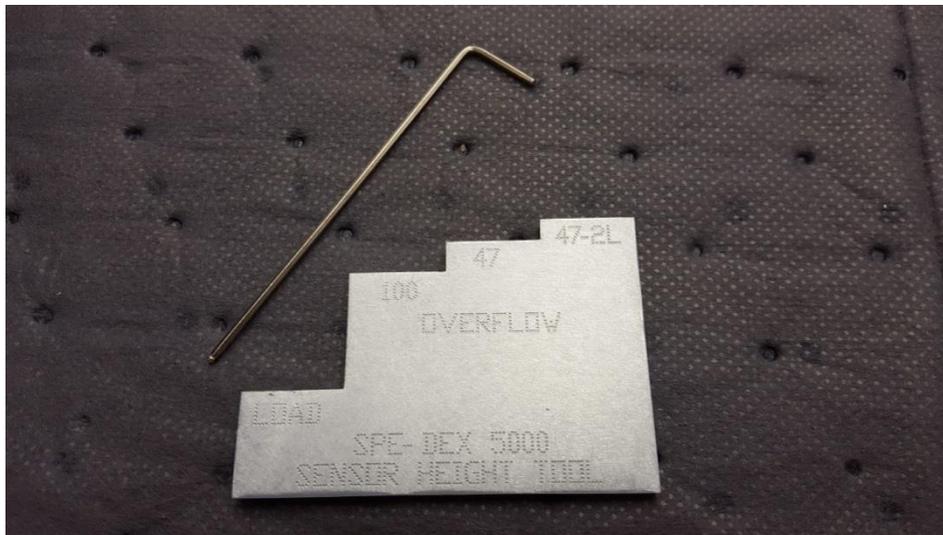


Figure 6-1. Thermistor Sensor Height Tool (P/N 02-3170-05) and Hex Wrench (P/N 99-0880)

To set the overflow sensors using the Thermistor Height tool (P/N 02-3170-05), place the tool on the disk holder platform as shown in Figure 6-2.

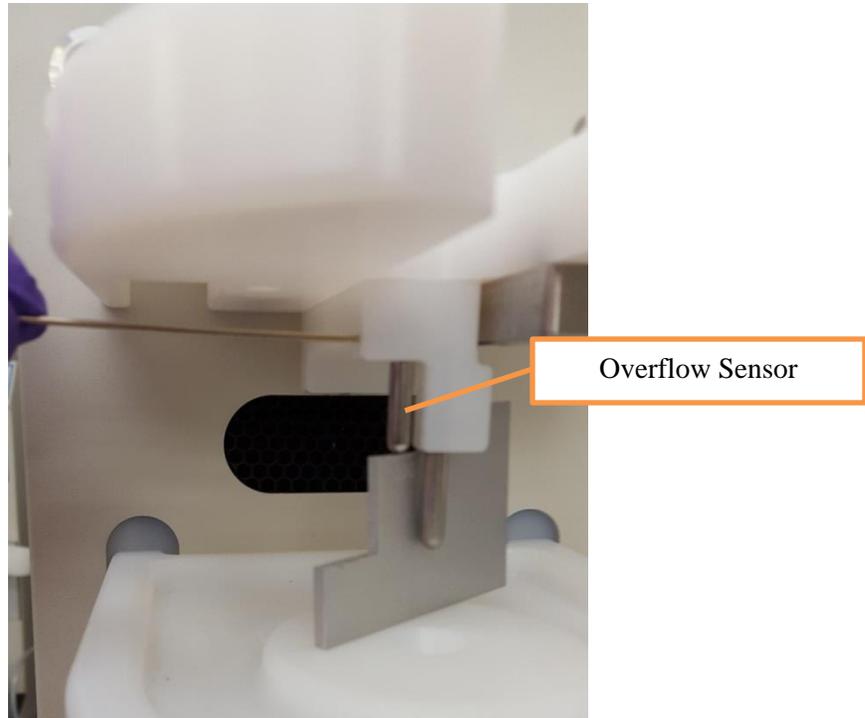


Figure 6-2. Thermistor Height Tool being used to set the Proper Height for the Overflow Sensor above the Disk Holder Platform

The Thermistor Height Tool has 3 positions indicated for the overflow sensor height: 100, 47, and 47-2L. The 100 setting is to be used for large disk holders (Fast Flow and 90 mm disk holders) and the 47 setting should be used for the small disk holders (47mm). If 2 L samples are to be used with 47mm disk holders, then use the highest setting (47-2L)

Using the appropriate level as labeled on the Thermistor Height Tool, check to see if the tool fits under the overflow thermistor with the platform in its highest position. The tool should fit snugly with a light interference or drag when sliding under the thermistor, while the platform remains at its highest position. If the platform moves down or if there is a gap between the thermistor and the tool, the thermistor height should be adjusted. To adjust the thermistor height, use the Hex Wrench (P/N 99-0880) to loosen (do not remove) the set screw on the overflow thermistor. After loosening the set screw, the thermistor can be adjusted to the desired height on the Thermistor Height Tool. With the Height Tool in place, re-tighten the set screw until resistance is felt (do not overtighten as this could damage the thermistor).

6.2 Setting Up the Solvent Configuration

Step 1: The first step is to configure the module with the solvents needed to run a specific SPE method. When the software opens, the Overview screen that appears is shown below.

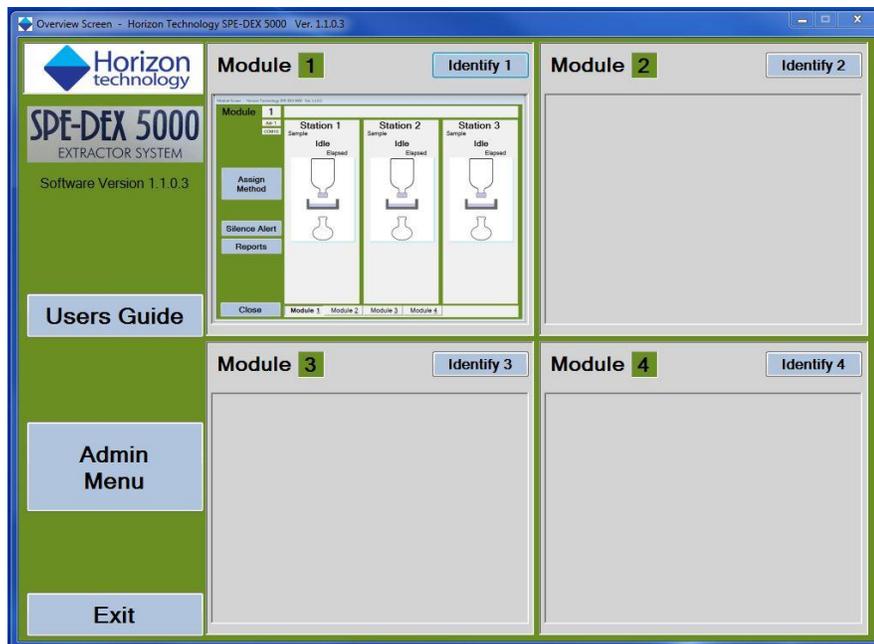


Figure 6-1. Overview Screen

Step 2: Click *Admin Menu* to open the Admin screen. The buttons for *Method Library*, *Solvent Library* and *PC Setup* will apply to all the modules connected to the controller. The buttons on the lower part of the page for *Solvent Configuration*, *Controller Setup*, *Module Setup* and *Diagnostics* can be set differently for each module connected, by choosing the appropriate tab for Module 1, Module 2, Module 3 or Module 4.

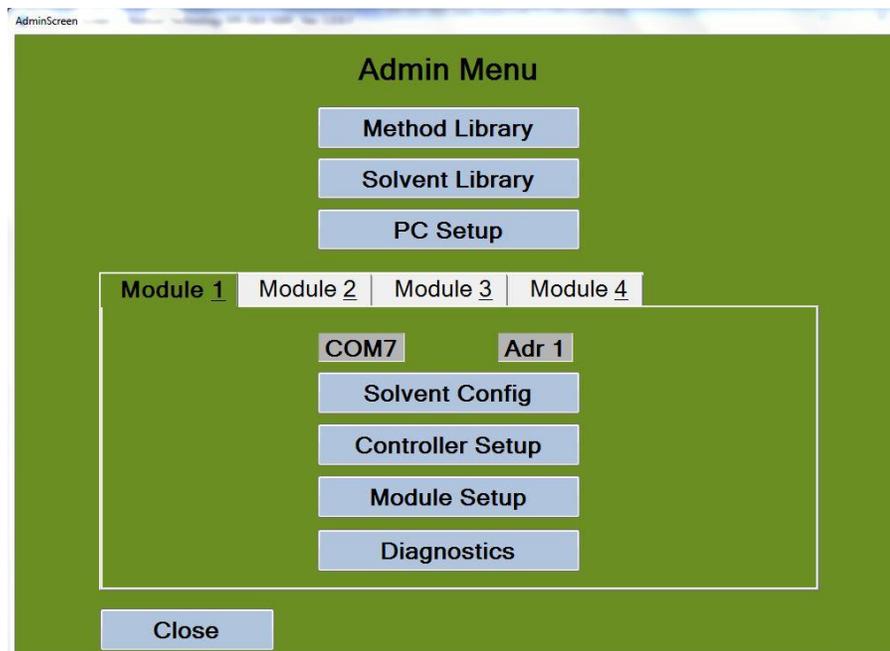


Figure 6-2. Admin Screen

Step 3: Click *Solvent Library* to open the Solvent Library screen – shown below.

The screenshot shows the Solvent Library interface. At the top, the title "Solvent Library" is centered. Below it is a table with four columns: Solvent Name, Density, Waste Dest., and Factory?. The table lists various solvents, with "Methylene Chloride" selected and highlighted in blue. Below the table, there are several buttons: "New Solvent", "Delete", "Duplicate", "Save", and "Cancel". To the right of these buttons is a form for editing the selected solvent. The form includes fields for "Solvent Name" (Methylene Chloride), "Density" (1.33), "Waste Destination" (Solvent Waste), and "ID Number" (10018).

| Solvent Name | Density | Waste Dest. | Factory? |
|---------------------------|-------------|----------------------|----------------|
| 10mM NH4Ac/MeOH | 0.90 | Solvent Waste | Factory |
| 1M HCl/MeOH | 0.90 | Solvent Waste | Factory |
| 1M NaOH | 1.04 | Water Waste | Factory |
| Acetone | 0.78 | Solvent Waste | Factory |
| Acetonitrile | 0.79 | Solvent Waste | Factory |
| Cond. Solution A | 1.00 | Water Waste | Factory |
| Cond. Solution B | 1.00 | Water Waste | Factory |
| Disk Eluting Solution | 1.00 | Water Waste | Factory |
| Ethyl Acetate | 0.90 | Solvent Waste | Factory |
| Hexane | 0.65 | Solvent Waste | Factory |
| Methanol | 0.79 | Solvent Waste | Factory |
| Methylene Chloride | 1.33 | Solvent Waste | Factory |
| MTBE | 0.74 | Solvent Waste | Factory |
| Pentane | 0.63 | Solvent Waste | Factory |
| Petroleum Ether (Ligroin) | 0.65 | Solvent Waste | Factory |

Buttons: New Solvent, Delete, Duplicate, Save, Cancel

Form fields:
 Solvent Name: Methylene Chloride
 Density: 1.33
 Waste Destination: Solvent Waste
 ID Number: 10018

Figure 6-3. Solvent Library Screen

Step 4: Review the solvent list and ensure that the solvents required for your method are available.

Step 5: If a needed solvent is not listed:

Step 6: Click *New Solvent*. A solvent titled “New Solvent” is added to the end of the Solvent Library.

The screenshot shows the Solvent Library interface after clicking "New Solvent". The table now includes "New Solvent" at the bottom, which is highlighted in blue. The form below the table is updated to reflect the new solvent: "Solvent Name" is "New Solvent", "Density" is "1.00", "Waste Destination" is "Solvent Waste", and "ID Number" is "0".

| Solvent Name | Density | Waste Dest. | Factory? |
|---------------------------|-------------|----------------------|----------------|
| Cond. Solution A | 1.00 | Water Waste | Factory |
| Cond. Solution B | 1.00 | Water Waste | Factory |
| Disk Eluting Solution | 1.00 | Water Waste | Factory |
| Ethyl Acetate | 0.90 | Solvent Waste | Factory |
| Hexane | 0.65 | Solvent Waste | Factory |
| Methanol | 0.79 | Solvent Waste | Factory |
| Methylene Chloride | 1.33 | Solvent Waste | Factory |
| MTBE | 0.74 | Solvent Waste | Factory |
| Pentane | 0.63 | Solvent Waste | Factory |
| Petroleum Ether (Ligroin) | 0.65 | Solvent Waste | Factory |
| Reagent Water | 1.00 | Water Waste | Factory |
| Toluene | 0.87 | Solvent Waste | Factory |
| Water (pH 12.0) | 1.00 | Water Waste | Factory |
| Water (pH 2.0) | 1.00 | Water Waste | Factory |
| New Solvent | 1.00 | Solvent Waste | Factory |

Buttons: New Solvent, Delete, Duplicate, Save, Cancel

Form fields:
 Solvent Name: New Solvent
 Density: 1.00
 Waste Destination: Solvent Waste
 ID Number: 0

Figure 6-4. Solvent Library Screen

a. In the bottom right of the screen, enter a unique solvent name.

6: Basic Operation

- b. Enter requested information: Density and Waste Destination (Solvent Waste or Water Waste).
By default, the new solvent will be user-defined and not listed as a “Factory” solvent.

If more solvents are to be added to the Library, repeat the steps above.

Step 7: If you did not need to add solvents, click *Cancel* to return to the Admin Menu screen. If you added solvents, click *Save* to return to the Admin Menu screen.

Step 8: To configure the solvents for each Extractor Module, click the *Module* (1 in this example).

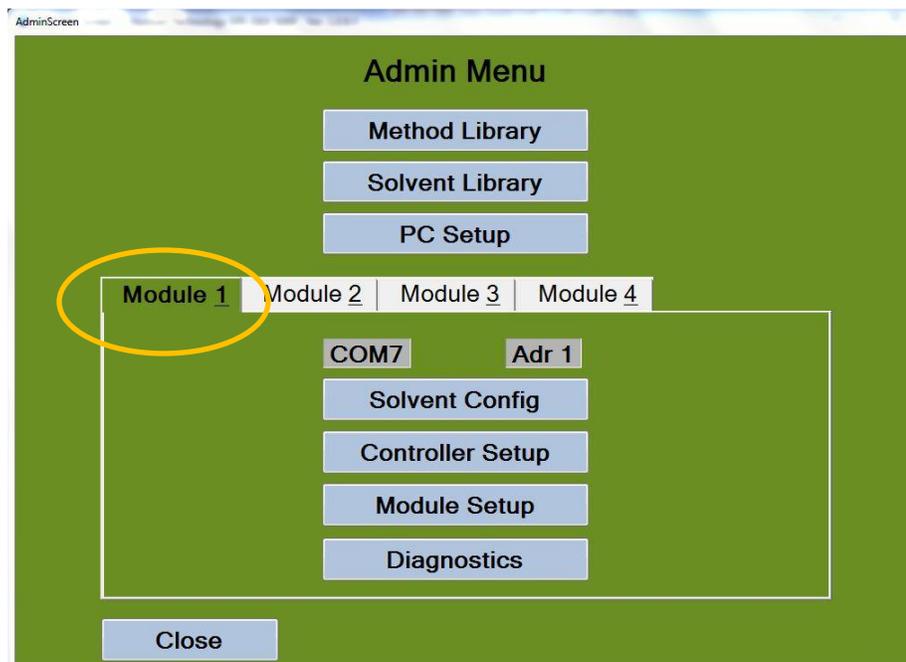


Figure 6-5. Module 1 Selected on the Admin Menu Screen (Circled)

Step 9: Click *Solvent Config* to display the Solvent Configuration screen (Figure 6-6).

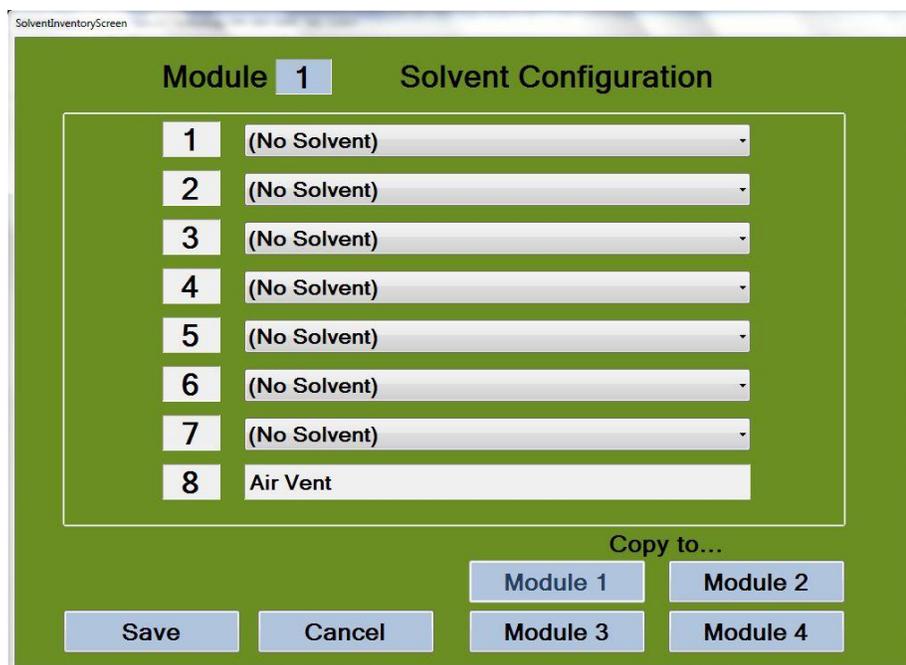


Figure 6-6. Solvent Inventory Screen

Step 10: Up to seven solvents can be run on each Extractor Module. Click in the solvent name drop-down box next to each number, and select the desired solvent from the solvent library. Repeat this sequence for all solvents to be used.

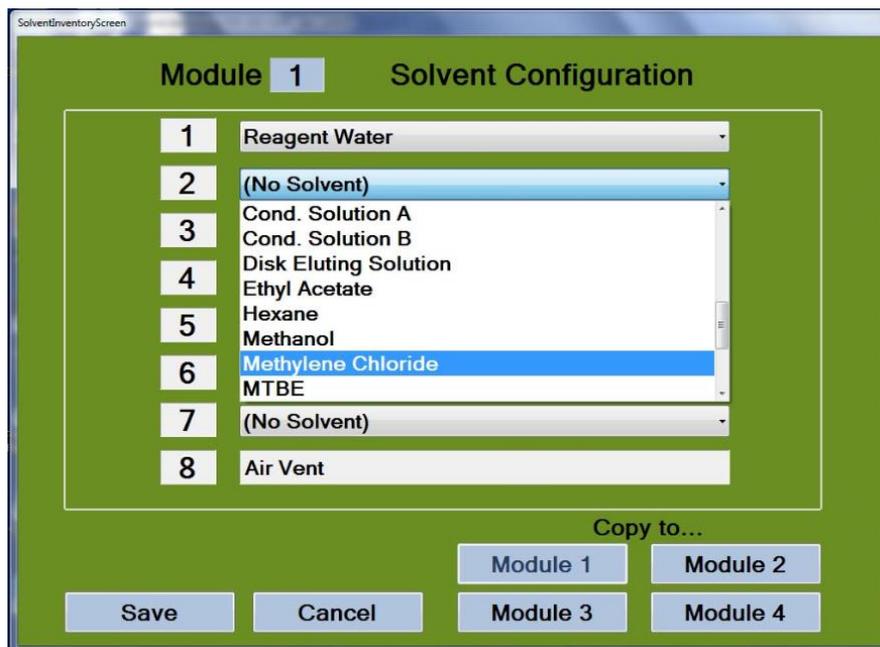


Figure 6-7. Selecting a Solvent

- Step 11:** Once all of the solvents have been identified, if more than one module is connected and multiple modules are to be run, you can use the *Copy to...* feature and use the same solvents for these modules. Click a Module button to copy the Solvent Configuration list to that module.
- Step 12:** Click *Save* when all solvents are properly configured.
- Step 13:** At this point, fill the solvent containers with the desired solvents and place the containers on the top of the Extractor Module in the solvent containment tray.
- Step 14:** Locate the individual solvent lines and place Solvent Line #1 into solvent container #1, Solvent Line #2 into solvent container #2, etc. Continue to do so for all of the solvents to be used. Carefully handle the lines so as not to introduce contamination. In addition, there is a cleaning method included in the factory method list to ensure the system is clean after installation and prior to analysis of samples.

6.3 Creating a New Method – A Prime Solvent Method

Before the module can be used to run actual samples, the solvent lines must be primed. Priming the solvent lines ensures that all air is removed from the lines. The next series of steps will show you how to create a Prime Method.

Step 1: From the *Admin Menu* screen, click *Method Library* button. The screen shown in Figure 6-8 will appear – note, this screen is only an example – your screen may be different.

6: Basic Operation

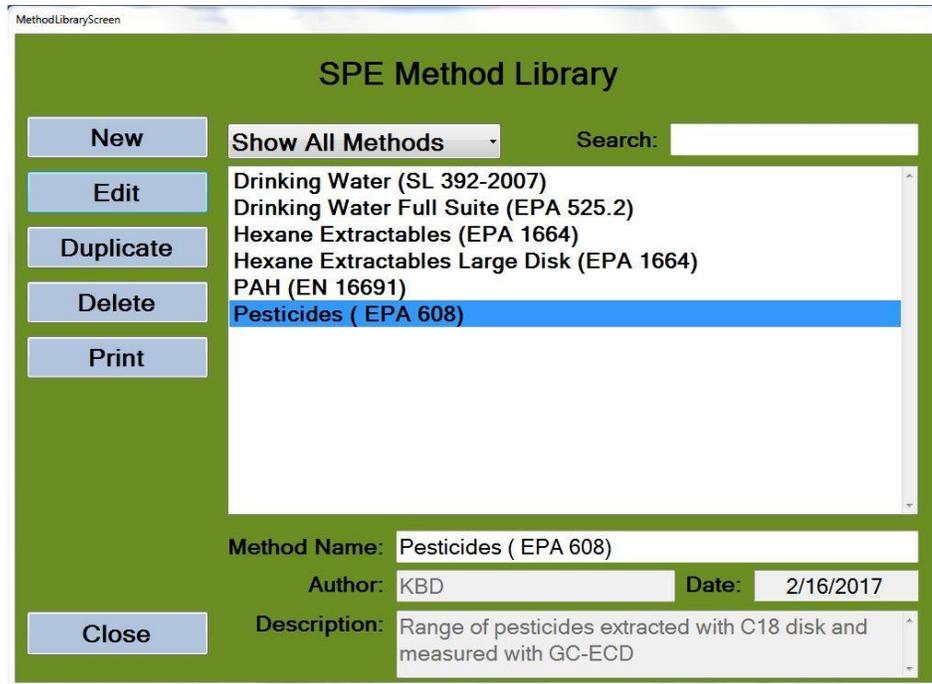


Figure 6-8. SPE Method Library Screen

Step 2: Click the *New* button. The SPE Method Editor screen is displayed without method steps defined (Figure 6-9).

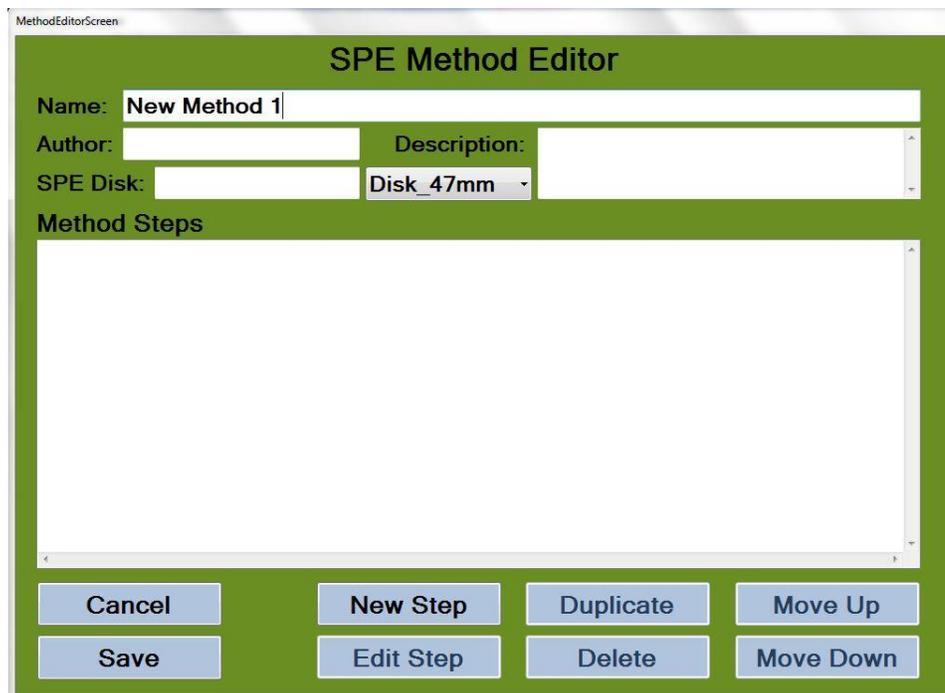


Figure 6-9. SPE Method Editor Screen for a New Method

Step 3: Now you can enter information for the method:

Name Unique name for the method. For example, delete the “New Method 1” name, and type in “*Prime Solvent Lines*”. (you cannot use <>?/:“\|*)

Author Name or initials of the person who created the method.

SPE Disk Name of the SPE disk (optional).

Disk Disk size selected from a drop-down menu.

Description Optional information to further describe the method.

Step 4: Once the above information has been entered, click the *New Step* button. Figure 6-10 will appear, as shown below. In this example, the Prime Solvent Lines step has been selected, as this is the operation that will be used to create a Prime Solvent Lines Method. Prime solvent lines will be used to flush air from each solvent line before operation starts.

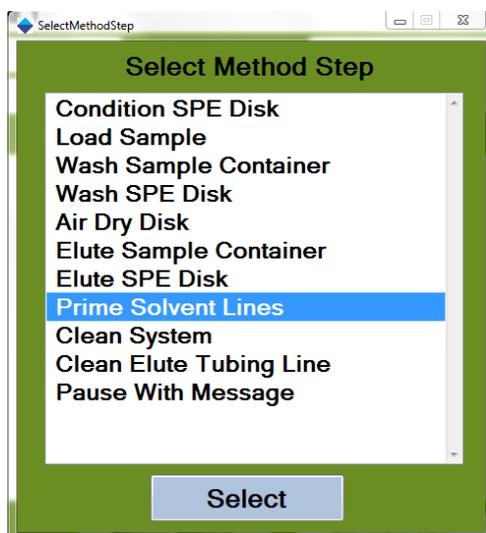


Figure 6-10. Select Method Step Screen

Steps are listed in the general priority of use. Once the Prime Solvent Lines step has been highlighted, click *Select*, or double click the step. The *Edit Step* screen is displayed with information applicable to the selected step (Figure 6-11).

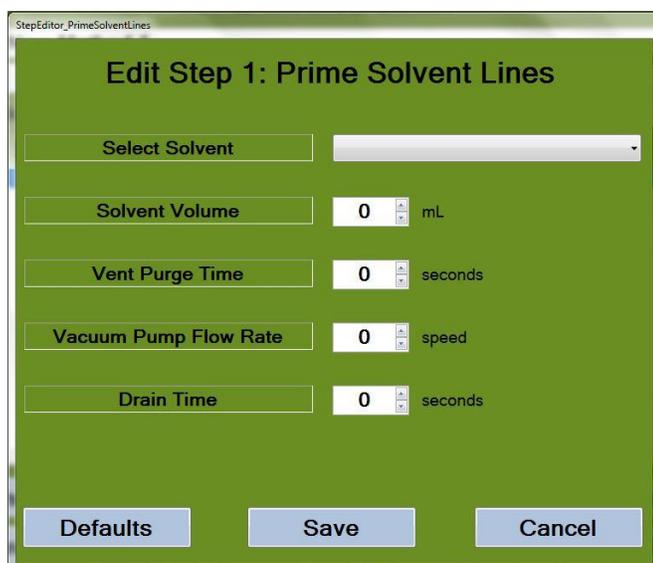


Figure 6-11. Edit Step screen for Prime Solvent Lines

6: Basic Operation

- Step 5:** Click the dropdown arrow in the Select Solvent box. The list of available solvents will appear. Find and select the first solvent to be primed. Once the solvent has been selected, it will now appear in the Select Solvent name box.
- Step 6:** Under the Solvent Volume, use either the up and down arrows to enter a volume, or click in the box, to enter a volume (0-300 mL). For priming the solvent lines, enter a volume of 10 mL.
- Step 7:** The Vent purge time clears the Vent line (0-300 sec). Enter 10 for this procedure.
- Step 8:** The Vacuum Pump Flow Rate is the rate at which the vacuum pump will remove the solvent from the Disk Holder (0-6). For priming, enter a value of 6.
- Step 9:** Next, enter a Drain Time. This is the time in seconds that the vacuum pump will run to pull the solvent out of the Disk Holder, and dispense the solvent into the Solvent waste container (0-300 sec). Enter a value of 15 seconds.
- Step 10:** Click *Save* to save the step information.
- Step 11:** The first Prime Solvent Line step will now appear in the Methods Step window. At this point, you have 2 options; you can repeat the above steps just followed, and click *New Step* and reenter all of the same information, but with a different solvent to prime, or as the first step is highlight (by the blue line) simply click the *Duplicate* button. A second line will appear, which a duplicate of the first line is. Double click the highlighted line, or click the Edit Step button. Change the name of the next solvent to prime.
- Step 12:** Continue to add new steps for all of the solvents to be primed.

While adding or editing steps, you can use the following features:

- Duplicate** Creates a duplicate of a selected step. This reduces the time needed to add information when you have similar steps.
- Delete** Removes a selected step from the method.
- Move Up** Moves a selected step up in order in the method.
- Move Down** Moves a selected step down in order in the method.

- Step 13:** Figure 6-12 shows a typical Prime Method. Complete your work by clicking *Save*.

MethodEditorScreen

SPE Method Editor

Name: Prime All Solvent Lines

Author: ADW Description: This method primes the solvent lines to remove air prior to operation

SPE Disk: No Disk Used Disk: Disk_47mm

Method Steps

| | |
|------------------------|--|
| 1: Prime Solvent Lines | Methylene Chloride:10mL Purge:10 Pump:6 Drain:15 |
| 2: Prime Solvent Lines | Ethyl Acetate:10mL Purge:10 Pump:6 Drain:15 |
| 3: Prime Solvent Lines | Methanol:10mL Purge:10 Pump:6 Drain:15 |
| 4: Prime Solvent Lines | Acetone:10mL Purge:10 Pump:6 Drain:15 |

Buttons: Cancel, New Step, Duplicate, Move Up, Save, Edit Step, Delete, Move Down

Figure 6-12. Example of a Prime Solvent Lines Screen

Step 14: Click the *Close* button to exit out of the SPE Method Library screen, and click *Close* again to exit out of the Admin Menu screen. You are now at the Overview screen.

6.4 Preparing to run the Prime Solvent Lines Method

- Step 1:** To run the prime method, a Disk Holder Assembly and a WIV must be in position.
- Step 2:** The height or position of the Disk Holder Platform can be adjusted. Gently press the Platform down. There are 2 stops which will hold the Platform in either one of the 3 positions. Position the Platform in the lower position.
- Step 3:** See section 6.7 for complete description of disk holder components. Locate the Basin of the Disk Holder assembly – only the Basin is needed for the Prime method.
- Step 4:** Gently press the Luer fitting of the Disk Holder into the center opening in the Disk Holder Platform. Gently seat the Luer fitting. Pressing too hard could deform the Luer fitting and cause a vacuum leak.
- Step 5:** Holding the Disk Holder Platform, gently raise the Platform to its highest position. An internal spring and pulley assembly will hold the Platform in position.
- Step 6:** Locate the WIV. As you look at the WIV, note the Guide Pins on the bottom side of the WIV housing. These pins will fit into the WIV Platform, and securely hold the WIV in place. Also note the Solvent Delivery Port. This port will connect to the bottom side of the WIV so solvents can be delivered to the Disk Holder, or to the sample bottle.

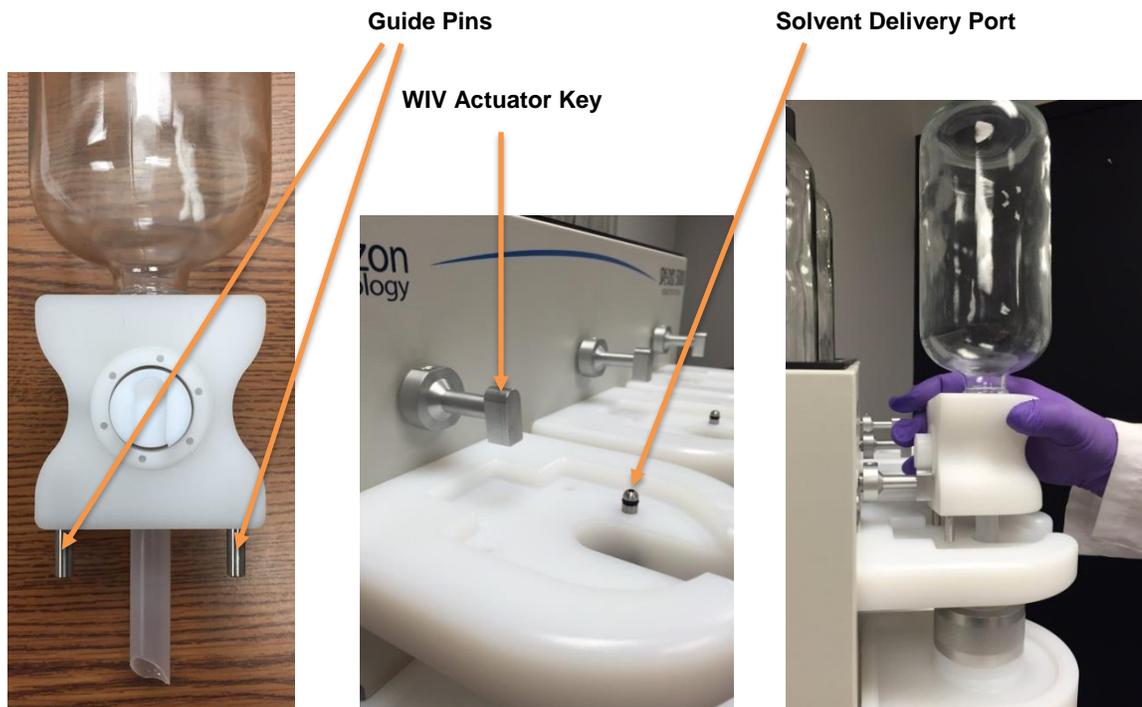


Figure 6-13. The WIV, Platform, and Loading the WIV

- Step 7:** Position the WIV over the WIV Platform, and while aligning the Actuator Key to the key on the backside of the WIV, gently, but firmly press the WIV into position. See the photos in Figure 6-13.
- Step 8:** Once the Disk Holder Assembly and the WIV with bottle are in place, click anywhere in the Module identification area (Module 1, circled in Figure 6-14).

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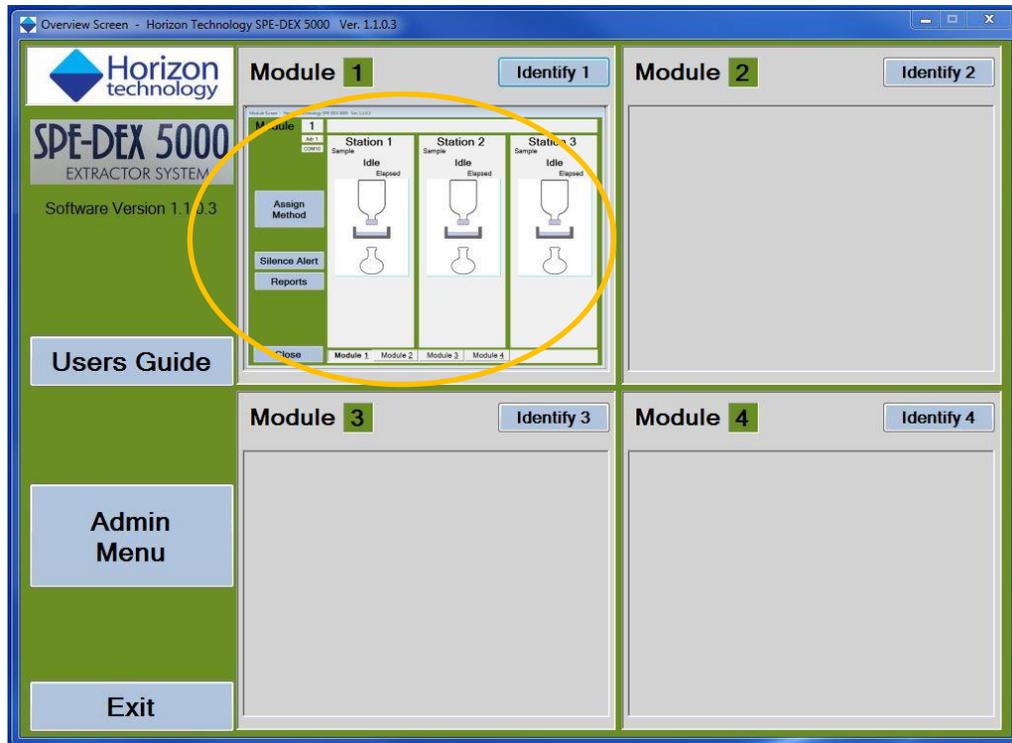


Figure 6-14. Selecting Module 1

Each module station will appear on the Module screen with an idle status because nothing is running as shown in Figure 6-15.

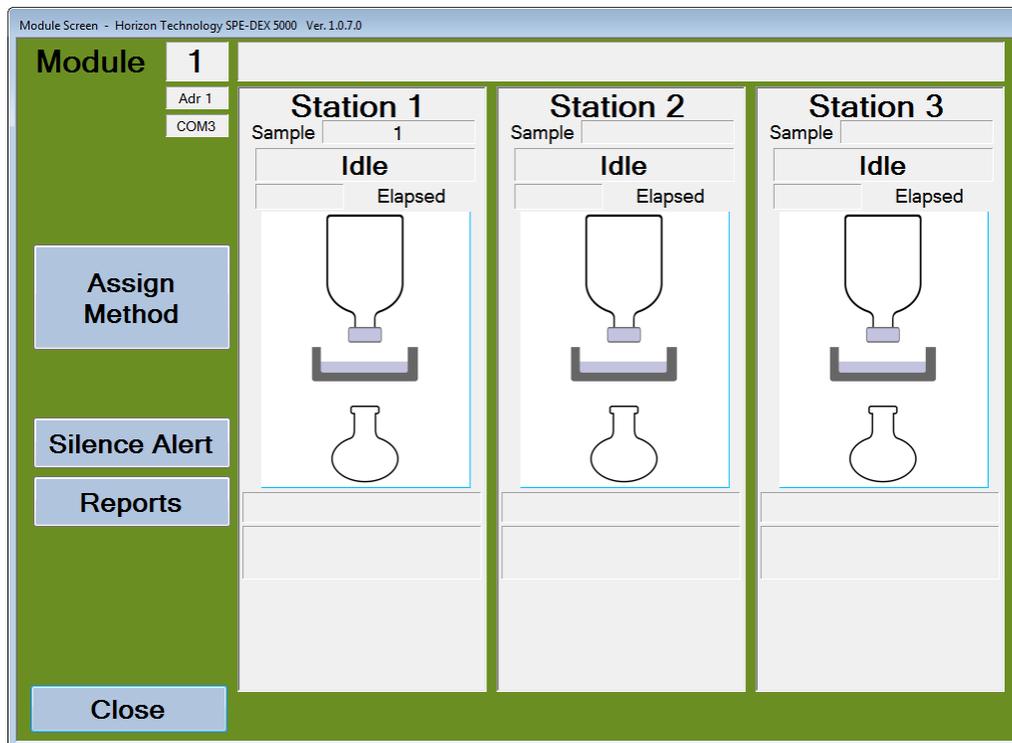


Figure 6-15. Module Screen

Step 9: Click *Assign Method* to open the Assign Method screen as shown in Figure 6-16.

The screenshot shows a software window titled "AssignMethodScreen". It has a green background and contains several sections:

- Select SPE Method:** A dropdown menu currently showing "Drinking Water (SL 392-2007)". To its left is a "Show All Methods" button and a "Search:" text box.
- Select Stations:** Three buttons labeled "Station 1", "Station 2", and "Station 3".
- Enter / Scan Sample ID:** Three empty input boxes.
- Operator:** A single large empty input box.
- Buttons:** "Assign" and "Cancel" buttons at the bottom.

Figure 6-16. Assign Method Screen

- Step 10:** Click in the box which shows the method list (currently displaying the first method *Drinking Water...*). (Figure 6-17) A drop-down box will appear and display the available methods which could be run. Select the newly created “Prime Solvent Lines method”. All methods stored in the SPE Method Library will be displayed. Remember, only a single method can be run on the Extractor Module.

This screenshot is similar to Figure 6-16 but with the dropdown menu open. The menu lists several methods:

- Drinking Water (SL 392-2007)
- Drinking Water Full Suite (EPA 525.2)
- Hexane Extractables (EPA 1664)
- Hexane Extractables Large Disk (EPA 1664)
- PAH (EN 16691)
- Pesticides (EPA 608)
- Prime All Solvent Lines** (highlighted in blue)

The "Operator" input box and "Assign/Cancel" buttons remain visible at the bottom.

Figure 6-8. SPE Methods

Once you select a method, it will appear in the box. For example, see Figure 6-18:

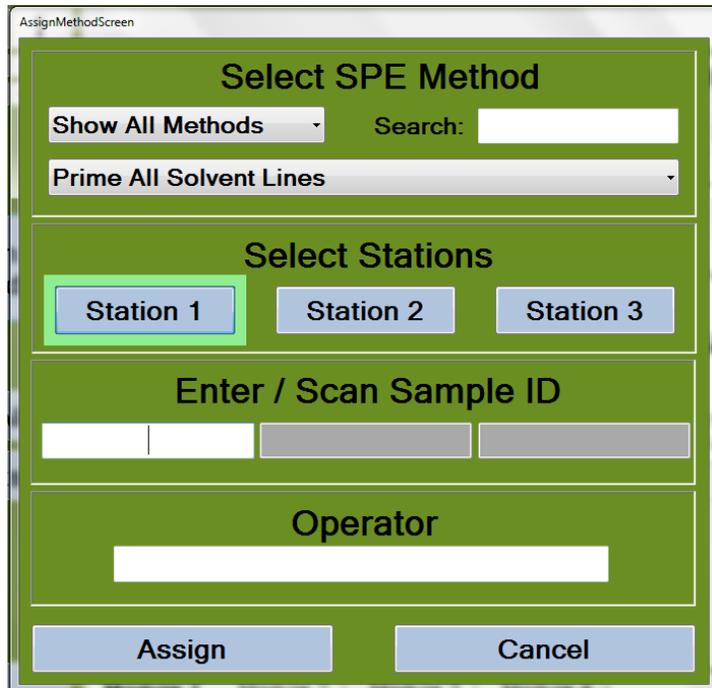


Figure 6-18. Selected SPE Method

Step 11: In the Select Stations area, click the station(s) on which to run the method. When you click a station button, the Station box will be highlighted and the cursor will appear in the box below where you can enter an ID (up to 14 characters) for the sample or scan a bar code serial number with an optional scanner.

Figure 6-19 shows an example of the screen when Prime Solvent Lines Method is to be run on Stations 1 and 2 with sample ID numbers entered. (Entering an operator name is optional.). Remember, in this example, 2 stations are going to be Primed. This requires 2 WIV's and 2 Disk Holder Basins to be installed on stations 1 and 2.

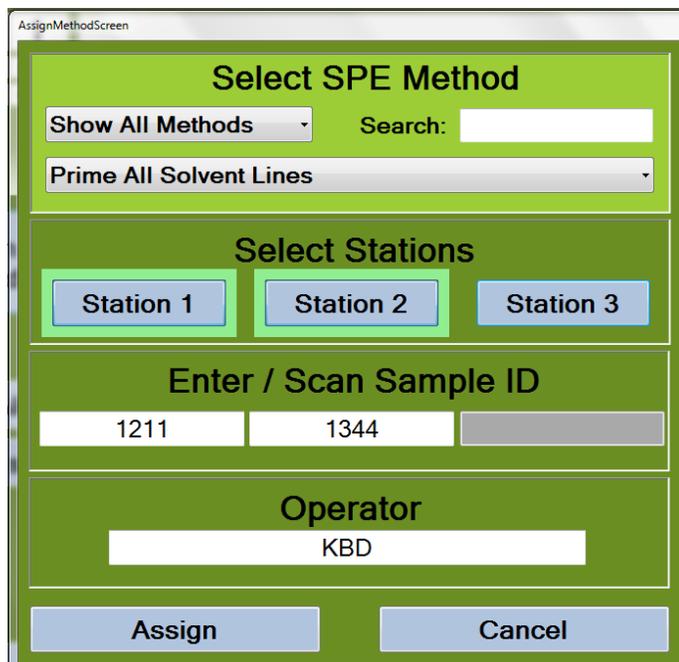


Figure 6-19. Selected SPE Method and Stations

Step 23: When all appropriate information is entered on the Assign Method screen, (Figure 6-20) click *Assign*. This action will assign the method to the station(s) and display a message as a reminder to prepare all stations. Click *OK*.



Figure 6-20. Station Preparation Reminder

If a solvent is missing from the solvent configuration for the Module, a message will indicate the required solvent. For example see Figure 6-21:

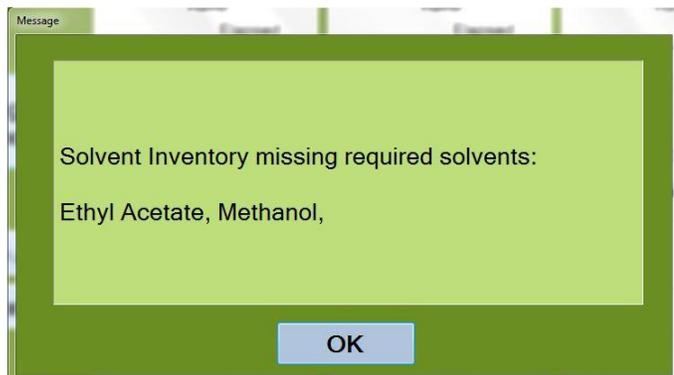


Figure 6-21. Missing Required Solvent

Step 24: In this case, click *OK* to close the screen.

Step 25: Click *Close* to exit the Module 1 screen.

Step 26: Click the *Admin Menu* button.

Step 27: Click the *Solvent Config* button.

Step 28: Click the drop down arrow next to the solvent port number, where the desired solvent is plumbed.

Step 29: Select the desired solvent.

Step 30: Click *Save*.

Step 31: Click *Close*.

Step 32: Click anywhere on the Module 1 icon and click *Assign Method*. When all appropriate information is entered on the Assign Method screen, click *Assign*. This action will assign the method to the station(s) and display a message as a reminder to prepare all stations.

Once a correctly configured method is assigned, the system will download the method as seen in Figure 6-22. The *Assign Method* button changes to a download indicator.

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Note: During the download sequence, the method is being transferred to the processor within the module chassis. Transferring the method directly to the module ensures a more reliable operation, as the module is truly running independently from the PC.

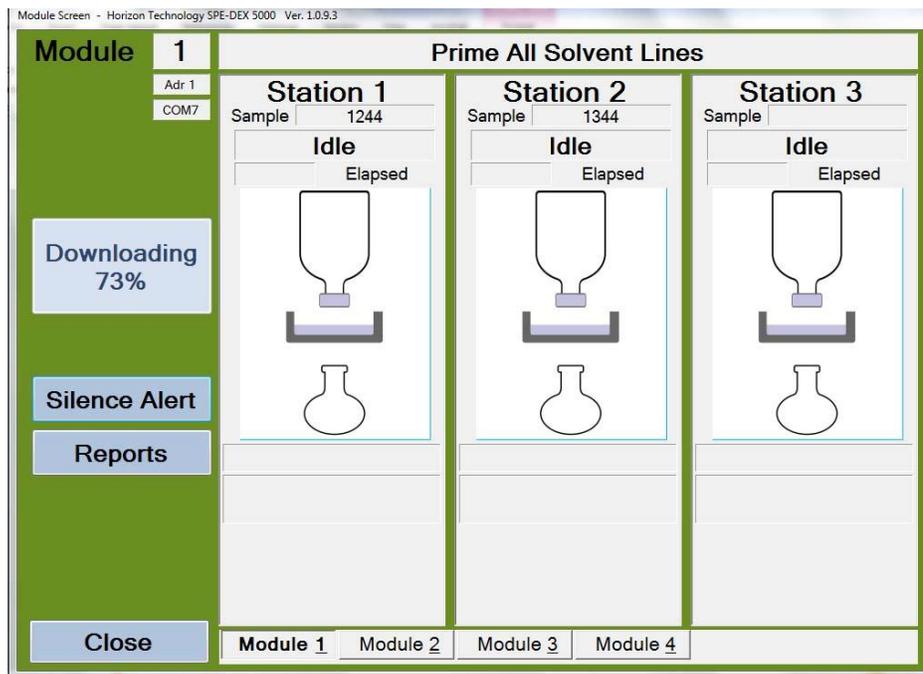


Figure 6-22. Downloading the Method

When the method is downloaded and ready to run, the button changes to **Start Run** (flashing). And the station to be run will turn yellow and indicate it is Ready as seen in Figure 6-23.

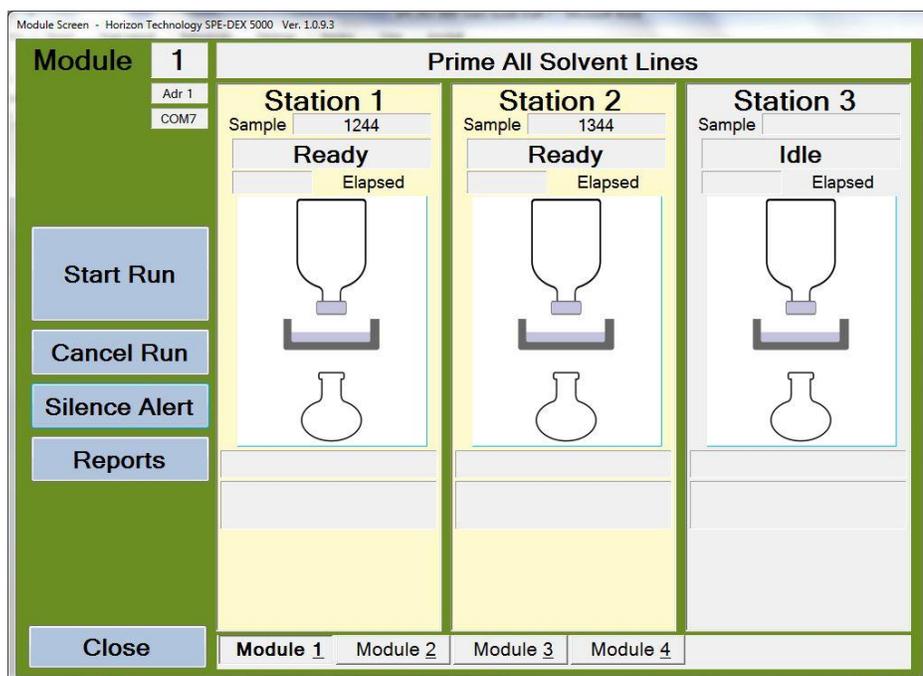


Figure 6-23. Method Ready to Run

Step 33: With the Disk Holder and the WIV in position, click **Start Run**. The Extractor Module will run the method and the Module screen (Figure 6-24) indicates the run status.

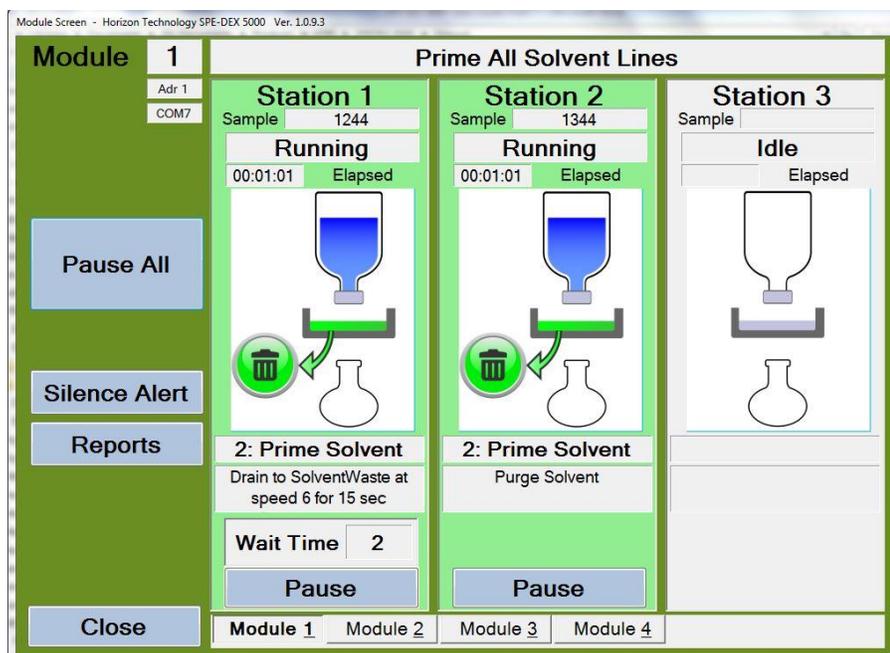


Figure 6-24. Example Showing the Status of Station 1 and 2

Notice that you can *Pause* the method running on a station (*Pause*) or *Pause All* stations.

You can click in the Station area to display additional details. The Station screen shows the method status as well as the method steps. Although you cannot change the method, you can scroll through the steps while the method is running.

If a running step includes a vacuum pump speed and/or wait time (as in the example below), you can adjust the speed or time for that station (Figure 6-25).

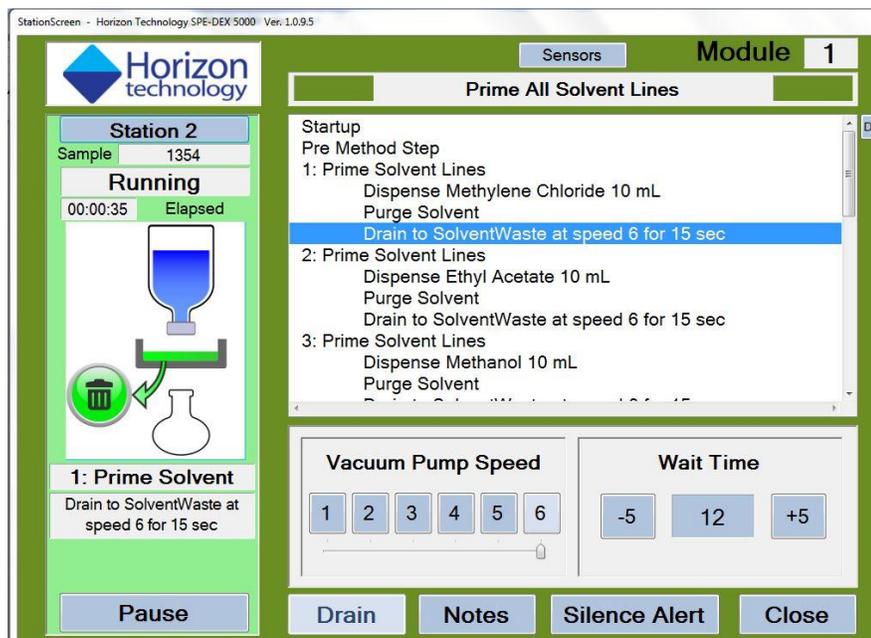


Figure 6-25. Details of the Method Running on Station 2

In addition, you can click:

Drain (Available only when the method is paused or the station is idle).
Manually drains the solvent waste, water waste, or eluate:

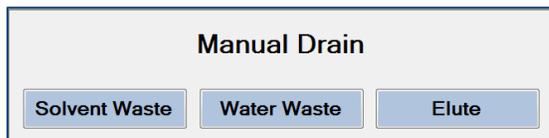


Figure 6-9. Manual Drain

Notes Enables you to enter notes that will be included in a method report:

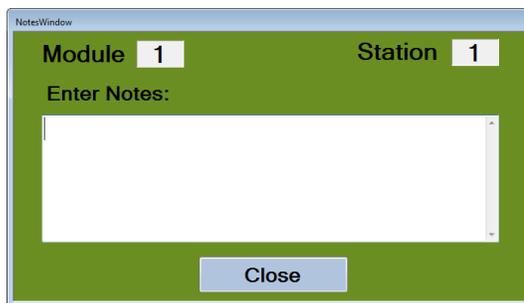


Figure 6-10. Method Notes

Silence Alert Turns off an audible alert.

A method could include a message that requires an operator action, such as shown in Figure 6-28. In this example, the operator must check the SPE disk and then click *Continue* for the method to continue:

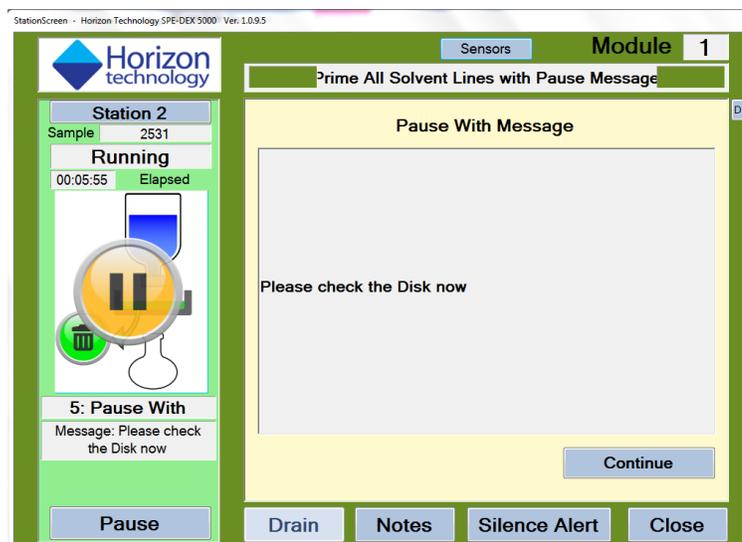


Figure 6-11. Example of a Message Requiring Operator Action (Station Screen)

During the method run, the solvents will be primed, in the sequence of the method. All solvents will be directed into the waste container configured. When the method run is complete, the station status screen will return to *Idle* and will be grey in color. The WIV and Disk Holder can now be removed from the module.

The module is now ready to run real samples.

6.5 Using a Factory Method

The SPE-DEX 5000 PC software comes preloaded with several “Factory” methods, shown in blue in the Method Library. These Factory methods allow rapid startup, and will help ensure the water samples are processed properly. The methods may need to be customized under certain circumstances or for certain matrices. If a Factory method exists for the samples you wish to process, you can simply download this method to the individual modules and stations, and run the method. Follow the steps in Section 6.4 (page 59) to load a method. Factory methods:

- Provide a series of methods designed to handle a full range of samples using a variety of disks, while maintaining a high level of recovery.
- Allow faster system startup by using proven methods that cannot be modified.
- Provide common “benchmark” methods that allow you to troubleshoot the system and ensure proper operation.
- Are helpful as a starting point for customized methods

See the chart in Appendix A for a listing of the factory preset method parameters.

You can modify existing methods and create new methods to handle unique samples and/or optimize the extraction parameters for a particular sample.

6.6 Creating a New Method

When a new method needs to be created, there are two ways this can be accomplished. These are as follows:

- **Duplicate** a Factory method, modify it, and save the method under a new name
- Create a new method.

The following section will discuss creating a new method, say EPA Method 525.2, for the analysis of semi-volatile compounds in drinking water. The steps shown here are exactly the same steps that would be used to create any new method.

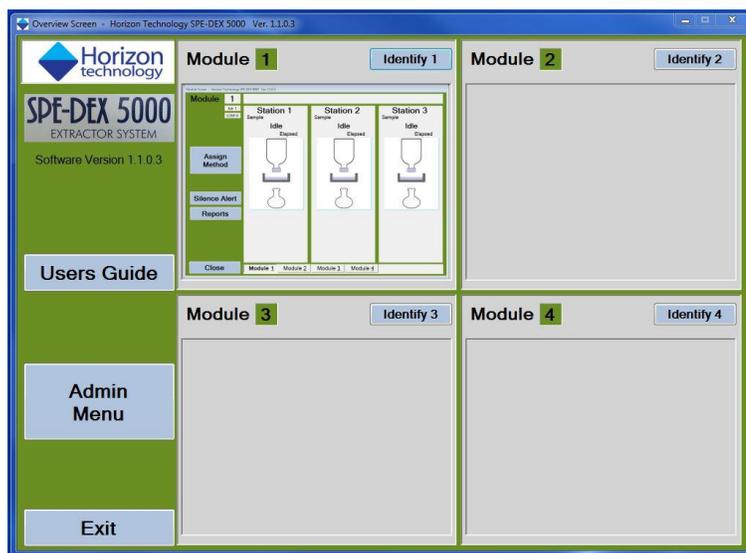


Figure 6-12. Overview Screen

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Step 1: From the main *Overview* screen, click the *Admin Menu* button. The screen in Figure 6-30 will appear.

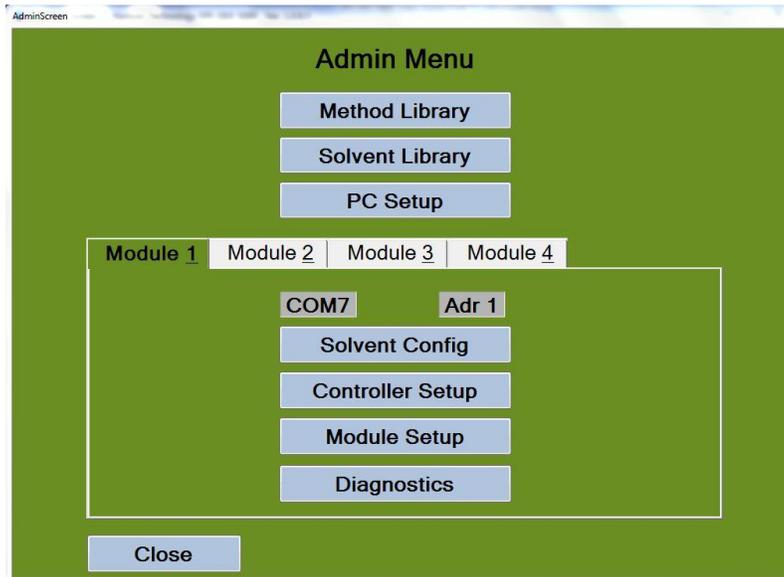


Figure 6-30. Admin Menu Screen

Step 2: Click the *Method Library* button. The screen in Figure 6-31 will appear (methods listed may be different).

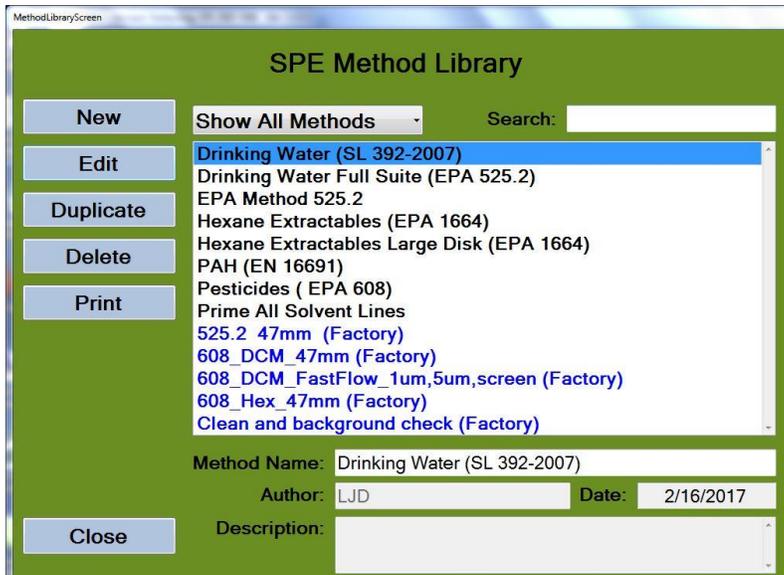


Figure 6-31. SPE Method Library Screen

Step 3: Click the *New* button. The screen in Figure 6-32 will appear.

The screenshot shows the 'SPE Method Editor' window. At the top, the title is 'SPE Method Editor'. Below the title, there are several input fields: 'Name:' with the text 'New Method 1', 'Author:' (empty), 'Description:' (empty), 'SPE Disk:' (empty), and a dropdown menu for 'Disk' currently showing 'Disk_47mm'. Below these fields is a large empty text area labeled 'Method Steps'. At the bottom of the window, there are eight buttons arranged in two rows: 'Cancel', 'New Step', 'Duplicate', 'Move Up' in the top row, and 'Save', 'Edit Step', 'Delete', 'Move Down' in the bottom row.

Figure 6-32. SPE Method Editor Screen

- Step 4:** Change the name of the method to something descriptive for the new method, and fill in any of the boxes that will provide more detailed information about the method. See the screen in Figure 6-33 as an example.

This screenshot shows the 'SPE Method Editor' window with the following information entered: 'Name:' is 'EPA Method 525.2', 'Author:' is 'RSJ', 'Description:' is 'EPA Method for semi-volatiles in Drinking Water', 'SPE Disk:' is 'C18', and the 'Disk' dropdown is still set to 'Disk_47mm'. The 'Method Steps' area remains empty. The same set of control buttons ('Cancel', 'New Step', 'Duplicate', 'Move Up', 'Save', 'Edit Step', 'Delete', 'Move Down') is visible at the bottom.

Figure 6-33. SPE Method Editor Screen with method information

- Step 5:** Near the bottom of the screen, click the *New Step* button. This will open the list of available SPE operations that can be used.

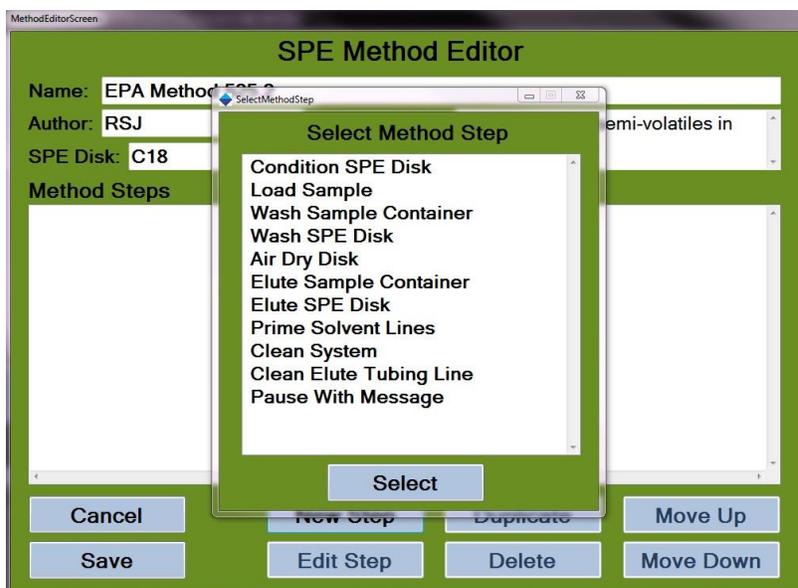


Figure 6-34. Select Method Step Screen

Step 6: Double click, or highlight the *Condition SPE Disk* operation, and click the Select button. This will open the screen shown in Figure 6-35.



Figure 6-35. Edit Step 1: Condition SPE Disk Screen

Step 7: Click the *Select Solvent* drop down box and select methylene chloride as the first solvent. Enter the values as shown in the screen in Figure 6-36.



Figure 6-36. Edit Step 1: Condition SPE Disk Screen with Values Entered

- Step 8:** Click the *Save* button. At this point, the fastest and easiest way to create a method is to use the *Duplicate* button. When the duplicate button is clicked, a duplicated line appears in the Method Editor window. Double click the highlighted step, or click the *Edit Step* button. Using the Select Solvent drop down box, change the solvent from methylene chloride to ethyl acetate. Leave the other values as they are. The screen should look like the one in Figure 6-37.

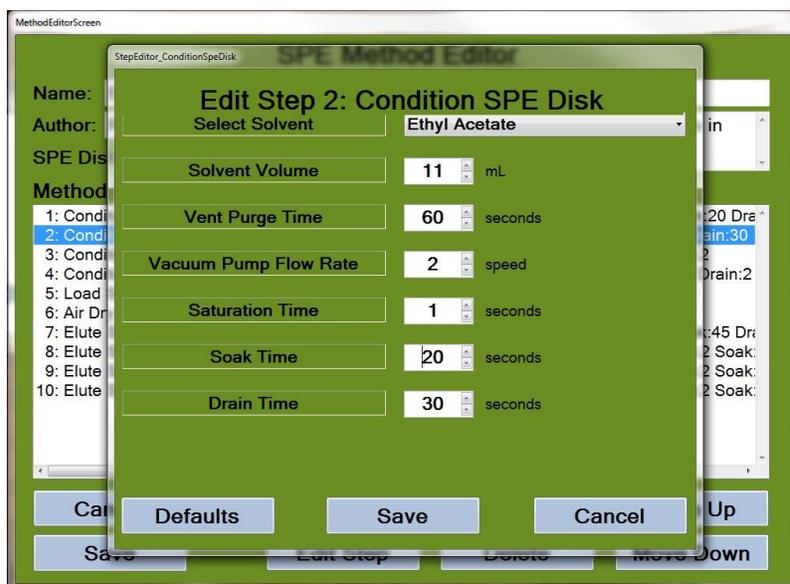


Figure 6-37. Edit Step 2: Condition SPE Disk Screen with Values Entered

- Step 9:** Click *Save*. You will now see 2 lines of the new method in the Method Editor window.
- Step 10:** The sequence of steps described above is how all SPE methods are created. To help with the completion of the 525.2 method, see the steps below. Use these values to complete the 525.5 method. Click *Save* when the method is complete.

EPA Method 525.2 – using the 47 mm Disk Holder

| | | |
|--------|----------------------------------|--------------------|
| Step 1 | <u>Condition SPE Disk</u> | |
| | Dispense Solvent | Methylene Chloride |
| | Solvent Volume | 15 mL |
| | Vent Purge Time | 60 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Saturate Time | 1 sec |
| | Soak Time | 20 sec |
| | Drain Time | 30 sec |
| Step 2 | <u>Condition SPE Disk</u> | |
| | Dispense Solvent | Ethyl Acetate |
| | Solvent Volume | 11 mL |
| | Vent Purge Time | 60 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Saturate Time | 1 sec |
| | Soak Time | 20 sec |
| | Drain Time | 30 sec |
| Step 3 | <u>Condition SPE Disk</u> | |
| | Dispense Solvent | Methanol |
| | Solvent Volume | 11 mL |
| | Vent Purge Time | 60 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Saturate Time | 1 sec |
| | Soak Time | 60 sec |
| | Drain Time | 2 sec |
| Step 4 | <u>Condition SPE Disk</u> | |
| | Dispense Solvent | Reagent Water |
| | Solvent Volume | 9 mL |
| | Vent Purge Time | 30 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Saturate Time | 1 sec |
| | Soak Time | 5 sec |
| | Drain Time | 5 sec |
| Step 5 | <u>Condition SPE Disk</u> | |
| | Dispense Solvent | Reagent Water |
| | Solvent Volume | 9 mL |
| | Vent Purge Time | 60 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Saturate Time | 1 sec |
| | Soak Time | 30 sec |
| | Drain Time | 0 sec |
| Step 6 | <u>Load Sample</u> | |
| | Sample Flow Rate | 2 |
| | Done Loading Sample Delay | 0 sec |
| Step 7 | <u>Air Dry Disk Timer</u> | |
| | Dry Time | 60sec |

| | | |
|---------|--------------------------------------|--------------------|
| | Vacuum Pump Flow Rate | 6 |
| | Nitrogen Blanket | Off |
| Step 8 | <u>Elute Sample Container</u> | |
| | Dispense Solvent | Ethyl Acetate |
| | Solvent Volume | 8 mL |
| | Vent Purge Time | 60 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Nitrogen Blanket | Off |
| | Saturate Time | 1 sec |
| | Soak Time | 30 sec |
| | Drain Time | 45 sec |
| Step 9 | <u>Elute Sample Container</u> | |
| | Dispense Solvent | Methylene Chloride |
| | Solvent Volume | 8 mL |
| | Vent Purge Time | 15 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Nitrogen Blanket | Off |
| | Saturate Time | 1 sec |
| | Soak Time | 30 sec |
| | Drain Time | 45 sec |
| Step 10 | <u>Elute Sample Container</u> | |
| | Dispense Solvent | Methylene Chloride |
| | Solvent Volume | 8 mL |
| | Vent Purge Time | 15 sec |
| | Vacuum Pump Flow Rate | 2 |
| | Nitrogen Blanket | Off |
| | Saturate Time | 1 sec |
| | Soak Time | 30 sec |
| | Drain Time | 45 sec |
| Step 11 | <u>Elute Sample Container</u> | |
| | Dispense Solvent | Methylene Chloride |
| | Solvent Volume | 8 mL |
| | Vent Purge Time | 15 sec |
| | Vacuum Pump Flow Rate | 6 |
| | Nitrogen Blanket | Off |
| | Saturate Time | 2 sec |
| | Soak Time | 30 sec |
| | Drain Time | 60 sec |

Note: Before this method is run, all solvent lines must be properly purged of air. A Purge Method can be used, such as created in Section 6.3. All Purge solvents are automatically discarded to waste.

Once the 525.5 Method has been completed, the method can be assigned to any one of the 3 stations, or assigned to all 3 stations. Whichever stations are to be run, the Disk Holders, WIV and sample bottles, and Collection Vessels must be in place. Follow the next section on how to properly load the SPE disk into the Disk Holder Assembly.

6.7 Loading the SPE Disk into the Disk Holder Assembly

Based on the accessories purchased with the 5000 System, there are several Disk Holder Assemblies that can be used. This section will explain how to use the multi piece Disk Holder Assembly. See Fig 6-38 below.

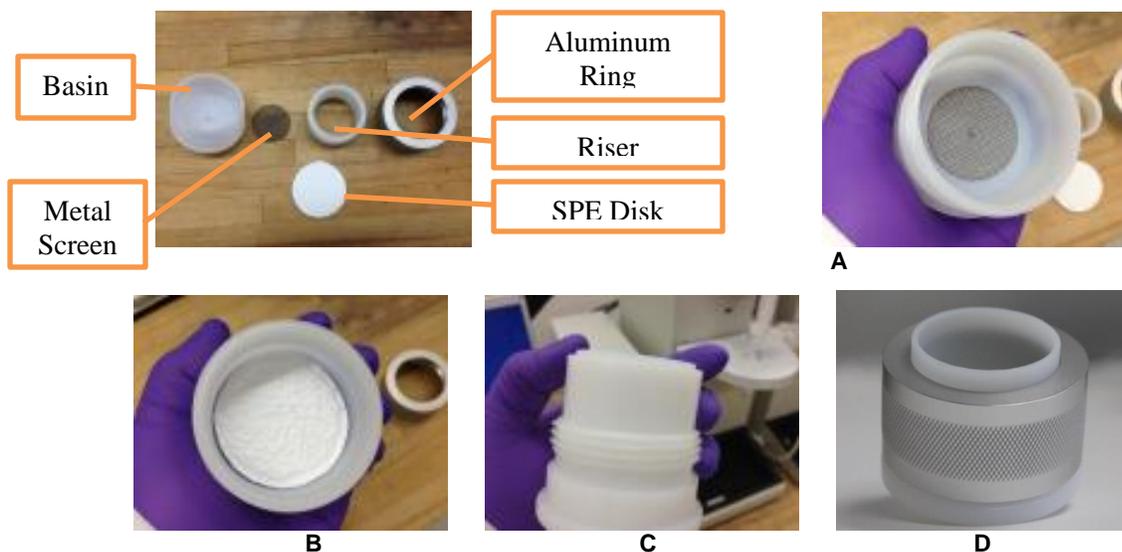


Figure 6-38. Multi Piece Disk Holder Assembly

- Step 1:** Locate the 47 mm Multi Piece Disk Holder Assembly. This should already be assembled. Unthread the Aluminum Ring from the Basin.
- Step 2:** Remove the Riser from the Basin, and put aside for now.
- Step 3:** Locate the small metal screen. This should be in the same bag the Disk Holder Assembly was in.
- Step 4:** Place the metal screen into the Basin (A), and make sure the screen is centered within the bottom recess of the Basin. *Note*; it is very important that the metal screen stay in this recess when the SPE Disk is placed in position. If the screen slides out from the recess, it is possible that an air leak will develop, which will significantly reduce the water sample flow rate.
- Step 5:** Locate the tube of SPE C18 Disks. While holding the Basin upright, and keeping the metal screen in the recess, gently place the SPE Disk over the metal screen (B). The SPE Disk will center itself in the Basin.
- Step 6:** Slide the Riser down on top of the SPE Disk, making sure the flat bottom side is inserted into the Basin, and the small ridge side is pointing up (C).
- Step 7:** Thread the Aluminum Ring Basin, making sure the Aluminum Ring is securely tight.
- Step 8:** On the Module, slide the Disk Holder Platform down to one of the lower detents, and gently place the Disk Holder Assembly into the center vacuum port on the Platform. Lift the Disk Holder Platform up to the highest position.
- Step 9:** Remove the cap from the sample bottle if one is in place. Inspect the condition of the bottle rim and threads and ensure that there are no chips or significant defects.
- Step 10:** Locate one of the WIV's. Look inside the top, threaded open, and confirm a sealing gasket is in place. (Figure 6-39) Also note the position of the stop cock portion of the WIV – it should be in the CLOSED position. Once the stop cock valve is closed, holding the WIV upside down, gently thread the WIV onto the sample bottle. The valve is designed to fit a 33x400-mm common round

bottle with narrow mouth (also known as a Boston round). Once the WIV is securely attached to the sample bottle, the entire assembly can be rotated upright, and loaded onto the WIV Platform. See the photo in Figure 6-40.

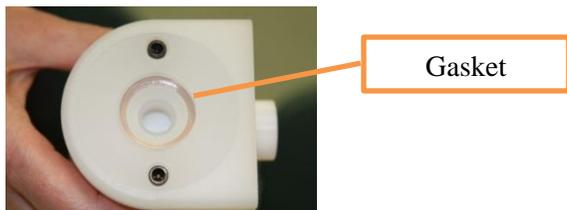


Figure 6-39. Gasket in Water Inlet Valve

Note: Cap adapters for various bottle sizes are available through Horizon Technology. To ensure that the appropriate cap adapters are obtained, determine either the dimensions of the threading or bottle manufacturer and catalogue number.

Note: Tighten the WIV onto the bottle with “moderate” force, as over-tightening could create leaks if the valve body is distorted, or the threads are stripped.

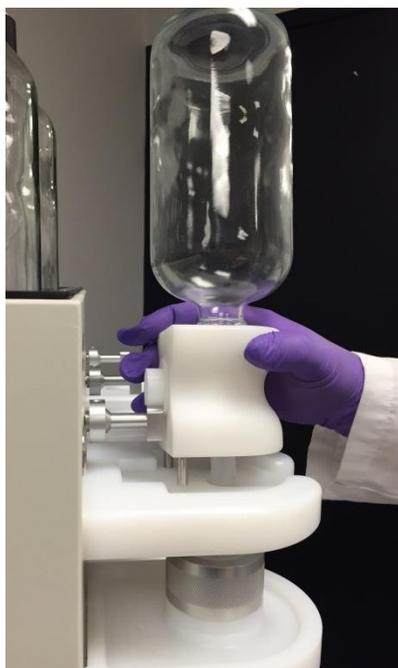


Figure 6-40. Loading the Sample Bottle and WIV onto the WIV Platform

Step 11: Next, locate the desired Collection Vessel. If purchased with the system, a convenient Collection Vessel is the 125 mL Erlenmeyer flask. See the photo in Figure 6-41.



Figure 6-41. Loading the Collection Vessel

- Step 12:** The Collection Vessel does not need a vacuum tight seal to work, as the Eluting solvent will be dispensed into the Collection Vessel. However, to ensure the Collection Vessel stays firmly attached, the Blue retaining clip can be used.
- Step 13:** Once the SPE Disk Holder, WIV and sample bottle, and the Collection Vessel are in place, the module is ready to run. In the previous section, the 525.2 method had been downloaded to any one of the 3 stations. When ready, press the **Start All** button to begin the run.
- Step 14:** The method will now run on the assigned stations. You can click on the station icon, and follow along as the method runs. You can also **Pause** and **Resume** operation anytime during the run. At the completion of the run, a report will be generated with all of the pertinent details of the run. This information can be exported out to a LIMS system if desired.

6.8 Editing an Existing Method

- Step 1:** On the Overview screen, click **Admin Menu** to open the Admin Menu screen.
- Step 2:** Click **Method Library** to open the SPE Method Library screen (Figure 6-42).

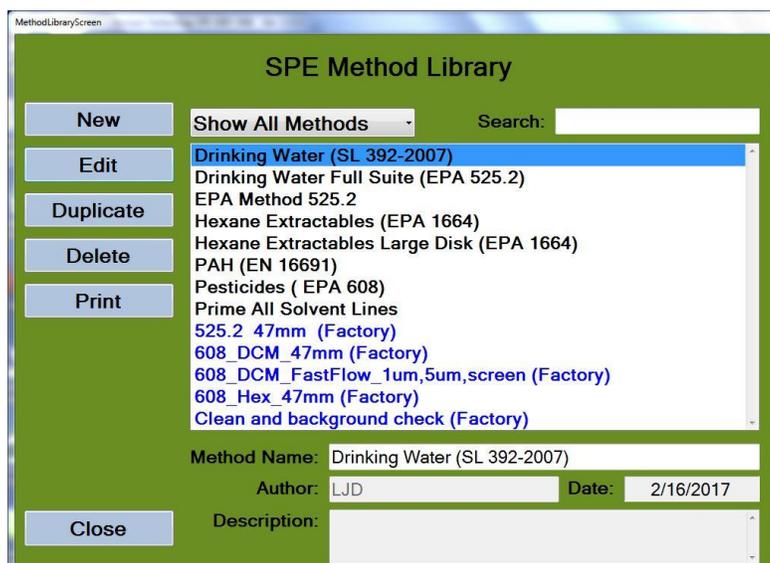


Figure 6-42. SPE Method Library Screen

- Step 3:** Click the *Show (Show All Methods)* drop-down and select an option to list only user-defined methods, all methods, or only factory-defined methods. By default, all methods are listed.
- Step 4:** To edit an existing method, select (highlight) the method name on the library list. (Note that you cannot edit a Factory method without first duplicating the method and then following this procedure.) Click *Edit*. The SPE Method Editor screen is displayed (Figure 6-43). You can modify any information on this screen, as described in Section 6.6.

SPE Method Editor

Name: Hexane Extractables (EPA 1664)

Author: CAD Description:

SPE Disk: Pacific Premium Disk: Disk_47mm

Method Steps

| | |
|-----------------------|---|
| 1: Condition SPE Disk | Acetone:8mL Purge:15 Pump:2 Saturate:2 Soak:30 Drain:30 |
| 2: Condition SPE Disk | Reagent Water:6mL Purge:20 Pump:2 Saturate:2 Soak:5 Drain:1 |
| 3: Condition SPE Disk | Reagent Water:6mL Purge:20 Pump:2 Saturate:2 Soak:5 Drain:0 |
| 4: Load Sample | VacuumPumpRate:4 DoneLoadingDelay:6 |
| 5: Air Dry Disk Timer | Dry:10sec PumpRate:5 N2:Off |

Buttons: Cancel, New Step, Duplicate, Move Up, Save, Edit Step, Delete, Move Down

Figure 6-43. SPE Method Editor Screen for an Existing Method

- Step 5:** Double click on the Method Step to edit. The step will open and allow changes to be made. When all changes have been made, click the *Save* button.
- Step 6:** Repeat these steps for all edits to be made.

6.9 Reviewing Methods and Functions

After the SPE-DEX 5000 System is set up for operation, you are ready to run methods. To help you more quickly understand the operation of the 5000 system, you can create simple methods which will help with the following:

- **Verification Method**

A Verification Method can be created to allow confirmation that everything is working on the system. A Verification Method could be as simple as Opening and Closing the WIV, or confirming that a specific solvent is being properly delivered.

- **Practice Method**

To gain familiarity with running the SPE-DEX 5000 Automated Extraction System with a water sample, you can create a Practice method. A Practice method will enable you to observe and understand the operation of the SPE-DEX 5000 System.

- **Standard Factory Method**

Factory Methods are pre-programmed into the software and cannot be edited, or changed. This is to ensure that “proven” methods are available for use. However, you can “duplicate” a

Factory Method, assign a different name, and then edit the copied method. This allows you to quickly create new methods which will have optimized parameters, already set. These copied methods can be deleted anytime, if they are not needed.

- **Drain Function**

This function drains liquids from the disk holder assembly, independent from running a method. There are three drain functions:

- **Water Waste** Drains any liquid in the disk holder assembly to the water waste container.
- **Solvent Waste** Drains any liquid in the disk holder assembly to an appropriate solvent waste container.
- **Elute** Elutes any liquid in the disk holder assembly into the collection vessel.

To run a Drain function, press the **Manual Drain** button on the Station screen (see Section 7.4, *Station Screen*).

- **Prime Solvent Line Methods**

The Prime Solvent Line methods are used predominantly for three reasons, to:

- Remove any air in the solvent lines prior to running any samples, typically performed after physically refilling the solvent canisters.
- Confirm that, during the initial system installation, all of the solvent solenoid valves are functioning properly.
- Verify system operation on a regular basis.

***Note:** It is recommended to run at least one Prime method every day before running actual samples to verify system operation. The need to run Prime methods will eventually be based on your own knowledge and experience with the system.*

***Note:** Be sure to load an empty Disk Holder Basin and a WIV with bottle on each station that is going to run the Prime Solvent Line method.*

- **Lockstep Operation**

During a Conditioning Step, if more than 1 station is to be used, the conditioning sequence steps are run simultaneously on all stations being run. This function is referred to as “Lockstep”. Lockstep operation ensures that all stations are treated identically, plus, it saves time. However, once the Load sample step begins, the stations will come out of Lockstep and now run independently from each other. This is important, as the water samples could be different, such as a clean blank compared to a dirty sample, so each station must be allowed to process the sample appropriately, individually from the other stations.

- **Air Pulse Time**

A single liquid dispensing pump is used to deliver solvents to each of the 3 stations. In order to ensure all of the solvent is removed from the lines before introducing the next solvent, an Air Pulse Time feature can be used, to use room air, to purge the solvents from the lines. The duration of the Air Pulse Time can be adjusted, based on the density or viscosity of the solvent.

- **Vapor Lock Air Pulse**

Based on the cleanliness of the water sample, at times it is possible that the WIV will open, but no water sample will flow down through the WIV, due to dirt or debris. When the water sample does not flow, it is referred to as a Vapor Lock. To automatically eliminate this poten-

tial Vapor Lock problem, a Vapor Lock Air Pulse is used. Upon opening the WIV, the liquid dispensing pump will send a burst of air into the WIV and dislodge the water Vapor Lock. This will ensure the water sample will flow.

6.10 Shutting Down the System

After processing your sample, the module system can be shut down. Follow the steps below to do this.

- Step 1:** Remove all disk holders, WIVs, and collection vessels from the Extractor Module(s).
- Step 2:** On the PC, close the SPE-DEX 5000 software program.
- Step 3:** Press the power button on the SPE-DEX 5000 Extractor Module(s) to turn the power off.

6.11 Cleaning/Disinfecting the System

The SPE-DEX 5000 may be wiped down with Isopropanol.



WARNING

Please note that the Overflow and Load sensors are warm. If preferred, the system can be turned off, before cleaning the sensors.

Notes:

7 SPE-DEX 5000 Software

7.1 Overview

The SPE-DEX 5000 Software running on the PC is the user interface to the SPE-DEX 5000 System Extractor Modules. This section provides an overview of the SPE-DEX 5000 software screens:

- Section 7.2, *Overview Screen*, page 74
- Section 7.3, *Module Screen*, page 75
- Section 7.4, *Station Screen*, page 76
- Section 7.5, *Assign Method Screen*, page 78
- Section 7.6, *Reports Screen*, page 79
- Section 7.7, *Admin Screen*, page 81
- Section 7.8, *SPE Method Library Screen*, page 82
- Section 7.9, *Solvent Library Screen*, page 83
- Section 7.10, *PC Setup Screen*, page 84
- Section 7.11, *Solvent Configuration Screen*, page 85
- Section 7.12, *Controller Setup Screen*, page 86
- Section 0, *Click Page 3* to display additional settings:

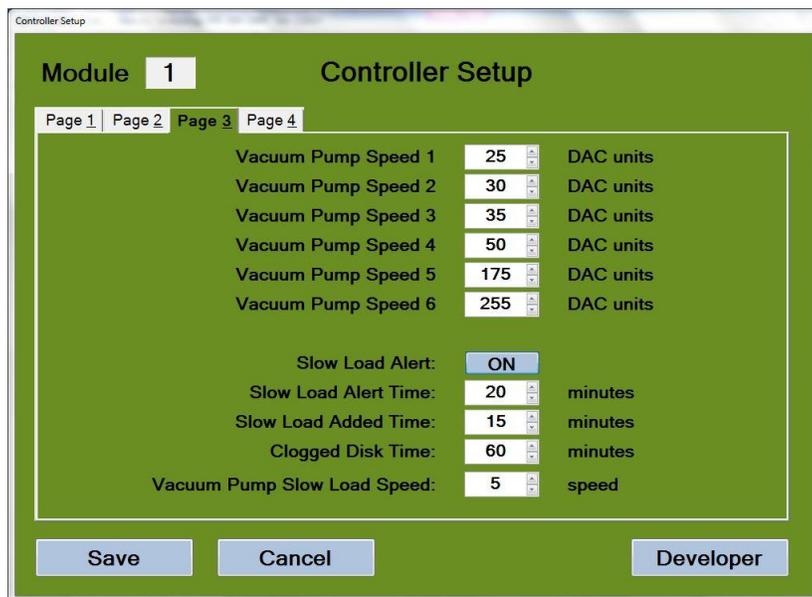


Figure 7-18. Controller Setup Screen (Page 3)

Vacuum Pump Speed 1-6 Not a linear scale and should not be touched unless a change is specified by the manufacturer

- Slow Load Alert** Indicates whether or not an alert occurs if the water sample load time exceeds the “Slow Load Alert Time” (below). It can be turned ON or OFF
- Slow Load Alert Time** Specifies the time before an alert occurs when the sample flow rate is slow.
- Slow Load Added Time** Automatically adds this time to Load Step, and before the method advances to the Air Dry Step. This ensures that the entire sample has been removed from the Disk Holder before advancing to the Air Dry Step.
- Clogged Disk Time** Allows the operator to input a time, such that if the sample takes longer than this time to filter through the SPE disk, to automatically stop the run, and display this warning to the operator. Long sample processing times typically indicate the SPE disk is clogged and the station needs attention.
- Vacuum Pump Slow Load Speed** The pump speed will automatically be increased to this number to try and clear the slow loading through the disk

Page 4 is currently unused and may have additional settings in future software versions

- Module Setup Screen, page 88
- Section 7.14, *Diagnostics Screen*, page 89

7.2 Overview Screen

The Overview screen displays the modules and provides access to the Admin Menu. If more than one module is connected, each will display on this screen. In this example, three modules are connected.

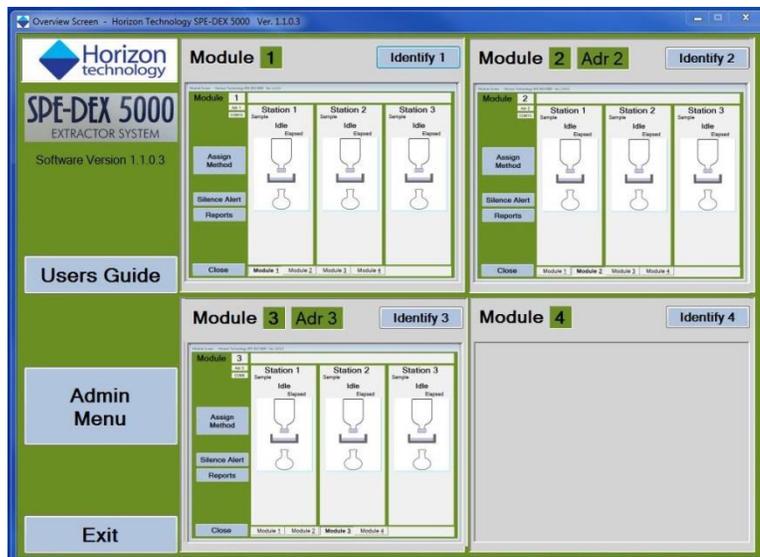


Figure 7-1. Overview Screen with Three Modules Connected

Features on the Overview screen are:

- Module** Displays each connected module in the system, identified with a Module number and address (Adr). You can click the **Identify** button to locate the associated module. The power button LED on the module will flash 10 times.

Click on an individual module to display more detail on the Module screen, as described in Section 7.3, *Module Screen*, page 75.
- Users Guide** The pdf (Acrobat Reader needed) is displayed and can be read or printed for reference
- Admin Menu** Launches the Admin Menu from which you can set up the system and methods. The Admin Menu also provides diagnostics, default parameters, and new software revision uploading. Refer to Section 7.7, page 81.
- Exit** Closes the SPE-DEX 5000 software.

7.3 Module Screen

The Module screen displays the three stations and the methods currently loaded into each station.

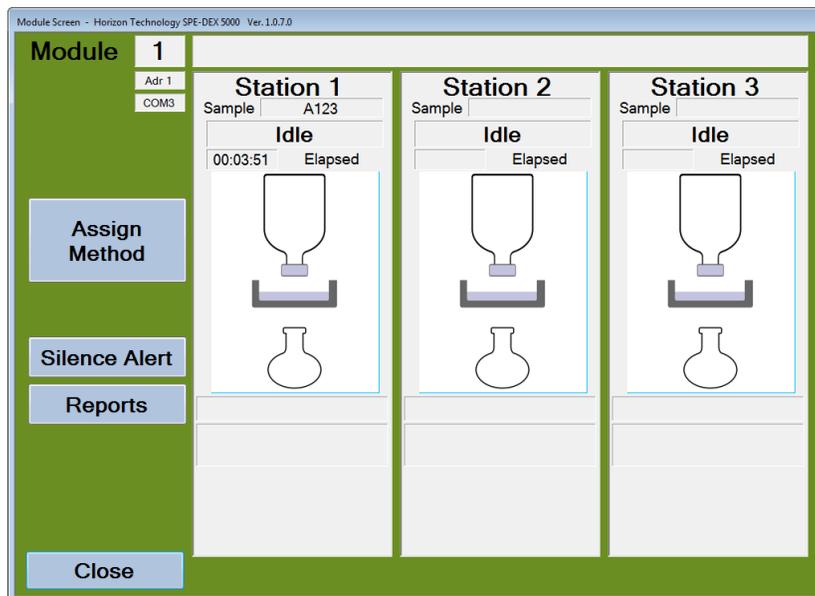


Figure 7-2. SPE-DEX 5000 Software Module Screen

The Module screen showing 3 stations is displayed, by default, and allows viewing of the current operational state of the extractor station(s) simultaneously. This is the main operating screen and it includes:

Station Status Displays the status of the currently running method step. For example, see Figure 7-3:

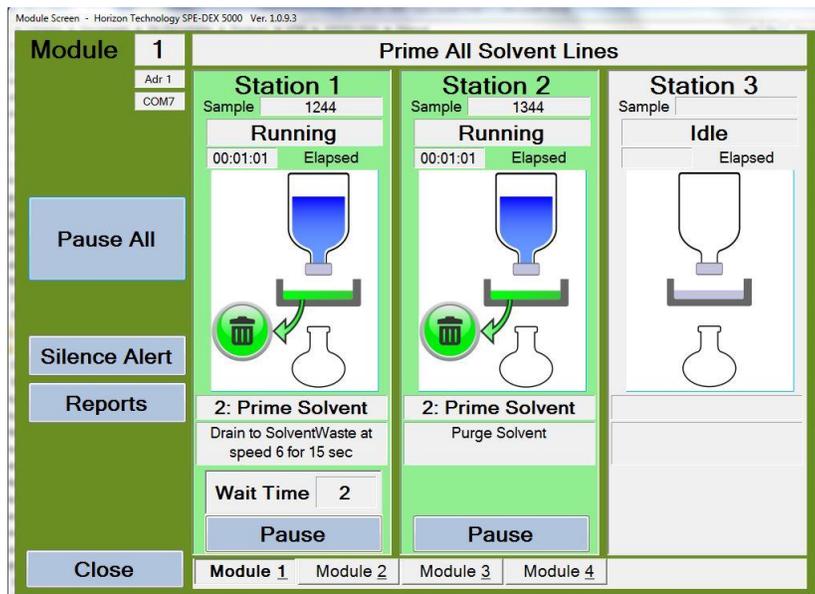


Figure 7-3. Example of a Running Method

Sample Identification of the sample. This can be entered or scanned (bar code) on the Assign Method screen (see Section 7.5, page 78).

| | |
|--|---|
| Status | Displays text indicating the method status, such as Idle or Running. |
| Elapsed | Displays the length of time a method has been running. |
| Icon | Shows a graphical representation of the step that is running. |
| Step Information | Describes the step that is running and includes additional information such as the wait time. Click Pause if you want to pause the method. After clicking Pause , you can Resume the run or Stop to cancel the method run. |
| Assign Method/ Start Run/ Pause All/ Resume All | Enables you to select a method to run. This button then changes and enables you to start running the method. Once running, this button again changes and enables you to pause all running methods. |
| Cancel Run | Once a Method(s) has been assigned, but before the system is started, clicking the Cancel Run button will return each station to the Idle state. This button appears only when the method is ready to start running (Start Run). |
| Silence Alert | Turns off an audible alert. |
| Reports | Displays the Reports feature. |
| Close | Closes this screen and returns to the Overview screen. |

7.4 Station Screen

Click anywhere in the Station area on the Module screen to access the Station screen shown in Figure 7-4.

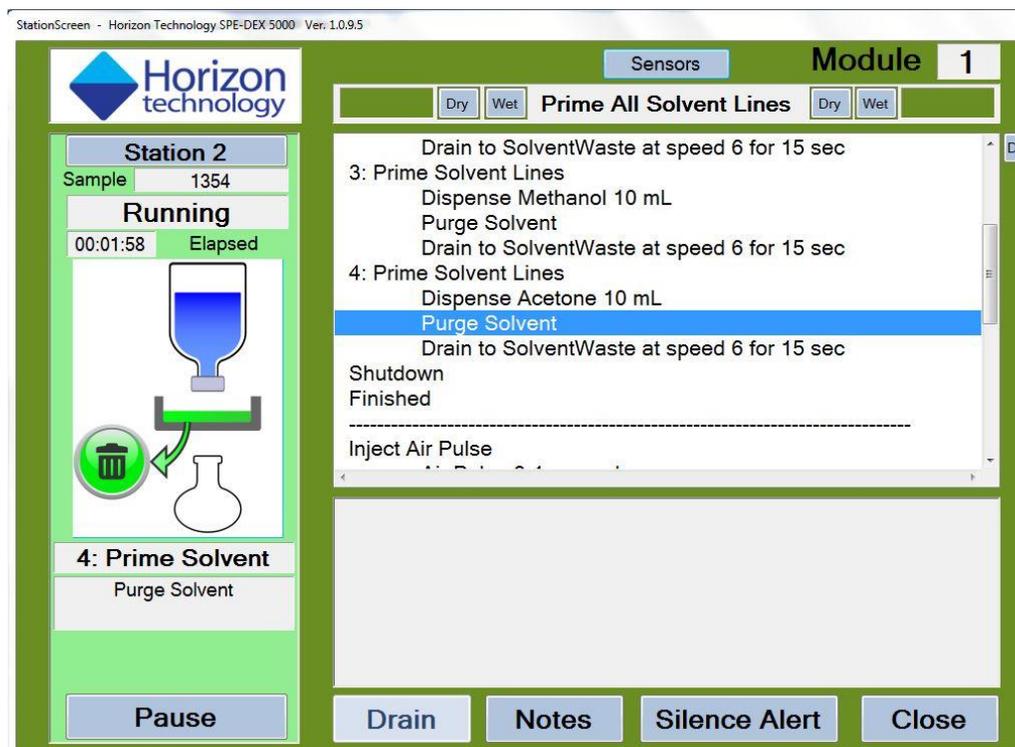


Figure 7-4. Station Screen

The Station screen is used to review the method for a station. This screen includes:

Station Icon Displays the status information (Sample, Status, Elapsed, Diagram, and Step Information), as described for the Module screen. Click **Pause** if you want to pause the method. After clicking **Pause**, you can **Resume** the run or **Stop** to cancel the method run.

Method Steps Displays the steps in the method. Although you cannot change the method, you can scroll through the steps while the method is running.

Vacuum Pump Speed or Wait Time

Displays if a running step includes a vacuum pump speed and/or wait time (as in the example above). You can adjust the speed or time while the method is running.

Drain (Available only when the method is paused or the station is idle). Manually drains the solvent waste, water waste, or elute:

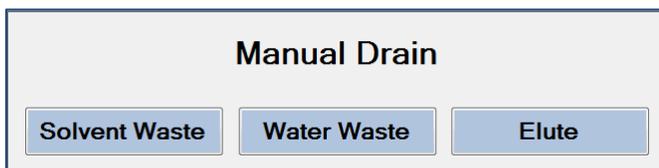


Figure 7-5. Manual Drain

Notes Enables you to enter notes that will be included in a method report:

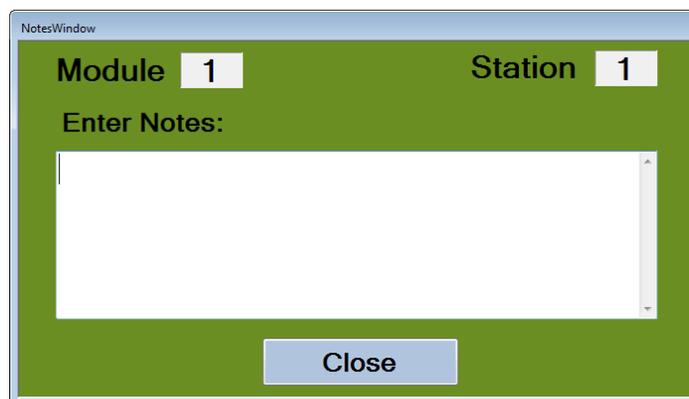


Figure 7-6. Method Notes

Silence Alert Turns off an audible alert.

7.5 Assign Method Screen

Click *Assign Method* on the Module screen to access the Assign Method screen (Figure 7-7).

Figure 7-7. Assign Method Screen

The Assign Method screen is used to assign a method to a station(s). This screen includes:

- | | |
|------------------------|---|
| SPE Method | Provides a list of the available SPE methods from which you can select. |
| Select Stations | Enables you to select a station(s) and then enter (up to 14 characters) or scan a sample ID. When you click a station button, the Station box is highlighted and the cursor will appear in the box below where you can enter an ID for the sample or scan a bar code serial number. <i>Note: All information associated with the samples to be run can be later exported to the lab's LIMS system.</i> |
| Operator | Enables you to enter an operator name or identification (optional). |
| Assign | Assigns the method to the station(s). |
| Cancel | Cancels any entries made, and returns to the Module screen. |

7.6 Reports Screen

Click the **Reports** button on the Module screen to access the Reports screen (Figure 7-8).

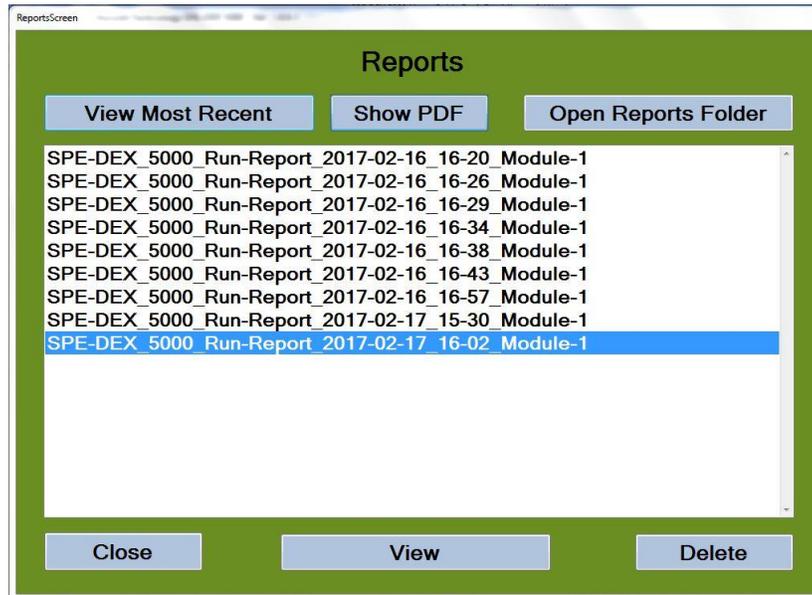


Figure 7-8. Reports Screen

This screen lists reports for the methods run on the modules. You can use the options to open and view reports.

- | | |
|----------------------------|---|
| View Most Recent | Opens a PDF or Excel (CSV) of the most recently recorded report. The format of the report depends on the toggle of the Show PDF/Show CSV button. |
| Show PDF/Show CSV | Enables you to indicate that the report will be displayed as a PDF or CSV (Excel) file. |
| Open Reports Folder | Opens the folder in Windows Explorer where reports are saved: C:\PC User Name\AppData\Local\Horizon\SPE-DEX 5000\data\Reports |
| View | Opens a PDF or Excel (CSV) of the report highlighted on the list. The format of the report depends on the toggle of the Show PDF/Show CSV button. (See Figures 7-9 and 7-10) |
| Delete | Deletes the report highlighted on the list. Note that the report is deleted as soon as you click Delete . |

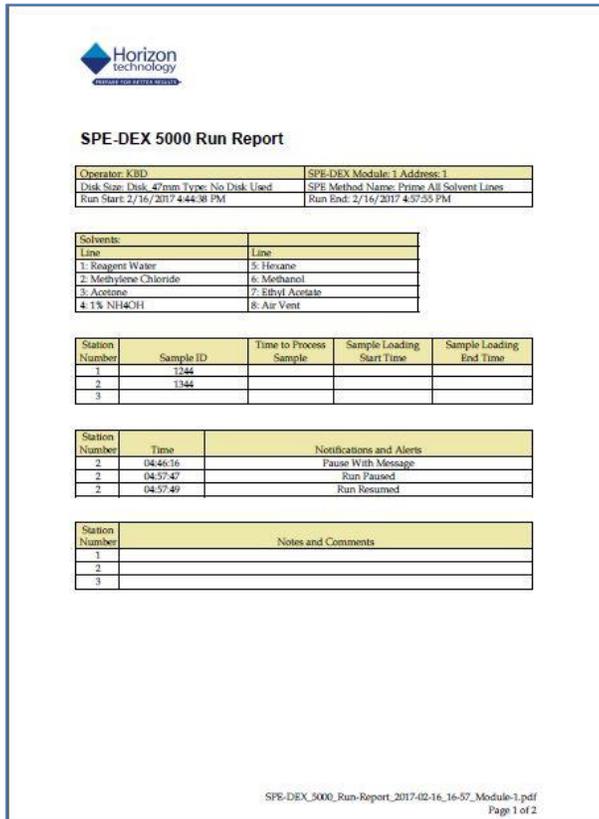


Figure 7-9. Example of a Report—PDF Format

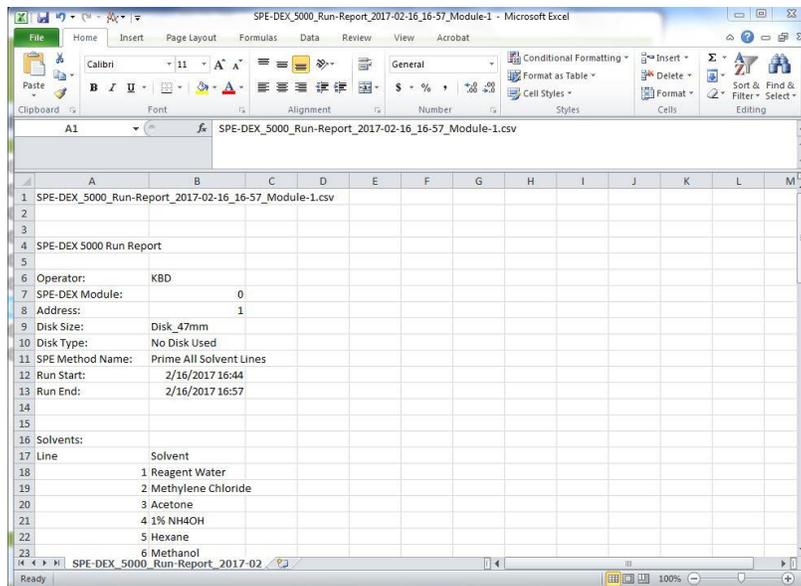


Figure 7-10. Example of a Report—CSV Format

7.7 Admin Screen

Click *Admin Menu* on the Overview screen to access the Admin Menu screen (Figure 7-11).

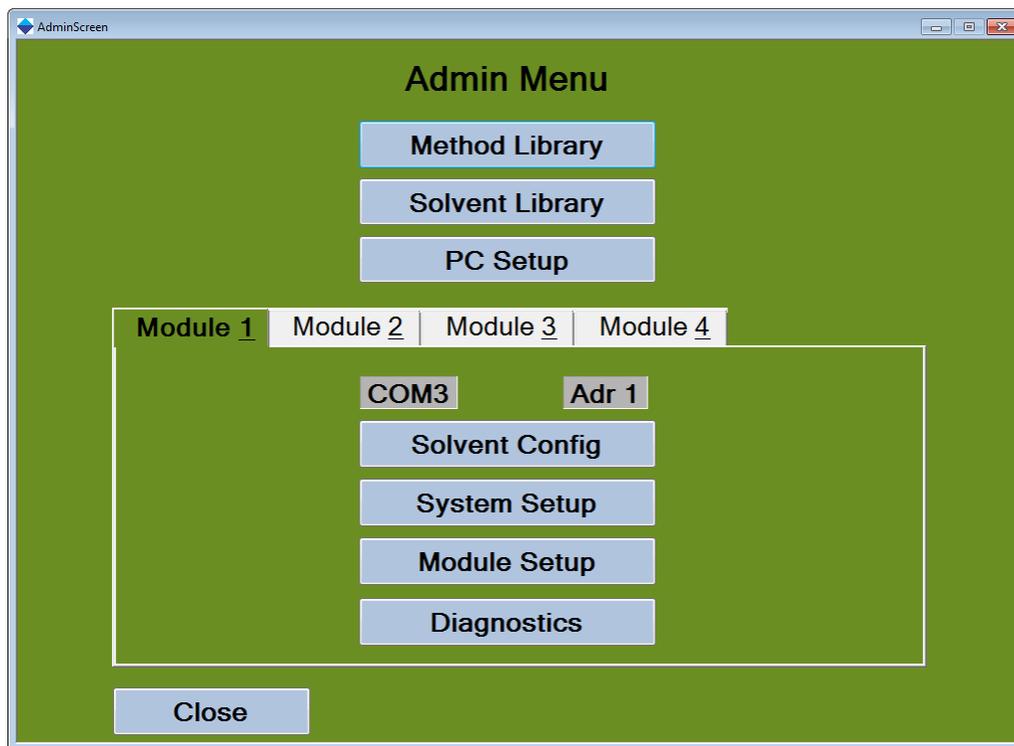


Figure 7-11. Admin Screen

The Admin Menu screen provides access to:

- Method Library
- Solvent Library
- PC Setup

For each of up to four modules, the Admin Menu screen also provides access to:

- Solvent Configuration
- System Setup
- Module Setup
- Diagnostics

7.8 SPE Method Library Screen

Click *Method Library* on the Admin Menu screen to access the SPE Method Library screen (Figure 7-12).

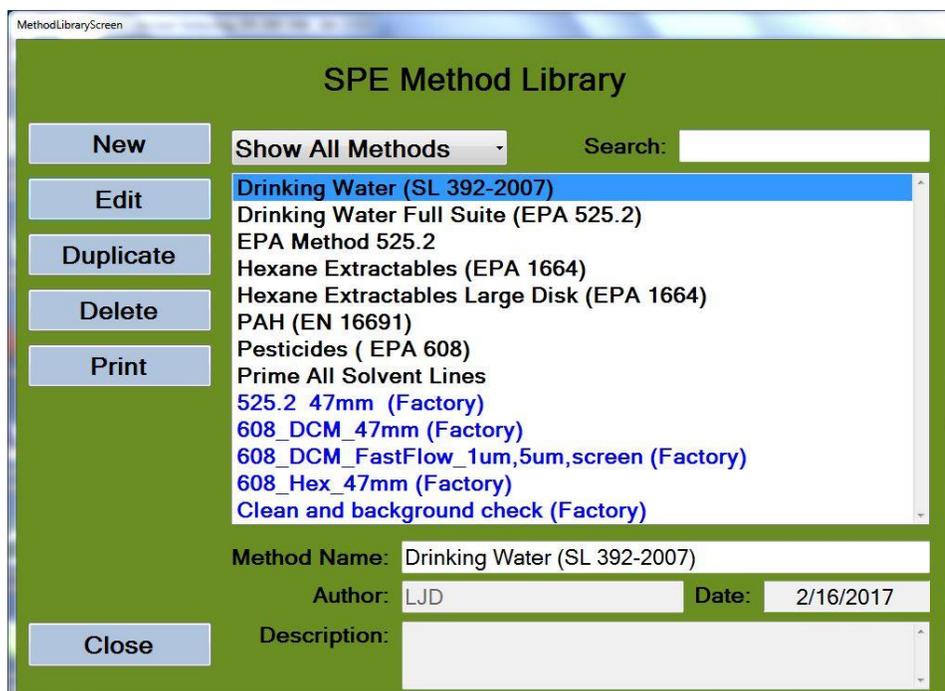


Figure 7-12. SPE Method Library Screen

This screen displays a list of available methods. When you highlight a method on the list, the following is displayed in the bottom right of the screen:

| | |
|--------------------|---|
| Method Name | Identifies the method. You can change this name. |
| Author | Identifies the person who created the method. |
| Date | Indicates the date the method was created. |
| Description | Displays additional information that was entered when the method was created. |

The Method Library screen also offers features to:

- Display specific methods. Click the Show drop-down (above the list of methods) to:
 - Show User Methods
 - Show All Methods
 - Show Factory Methods
- Search for a method. As you key in text, the matching methods are displayed.

Use the buttons on the Method Library screen to add, edit, delete, and print methods:

| | |
|-------------|---|
| New | Creates a new method. Click <i>New</i> to display the SPE Method Editor screen, which enables you to define a new method. |
| Edit | Edits the method selected on the library list. Click <i>Edit</i> to display the SPE Method Editor screen, which enables you to modify the method. |

- Duplicate** Duplicates the method selected on the list. Click **Duplicate** to copy this method as a new method that you can then edit to customize. The duplicate method is displayed with the same name as the original followed by the word “copy.” You can change the name in the Method Name field on this screen or when you edit to customize the method.
- Delete** Deletes a method. Select (highlight) a method in the list. Be sure you want to delete this method because it will be removed as soon as you click the **Delete** button.
- Print** Opens a PDF of a report for the selected method.

7.9 Solvent Library Screen

Click the **Solvent Library** button on the Admin Menu screen to access the Solvent Library screen (Figure 7-13).

The screenshot shows the Solvent Library screen with a table of solvents and a form for editing a selected solvent. The table has columns for Solvent Name, Density, Waste Dest., and Factory?. The selected solvent is Methylene Chloride, with a density of 1.33, waste destination of Solvent Waste, and factory status of Factory. The form below the table has fields for Solvent Name (Methylene Chloride), Density (1.33), Waste Destination (Solvent Waste), and ID Number (10018). There are buttons for New Solvent, Delete, Duplicate, Save, and Cancel.

| Solvent Name | Density | Waste Dest. | Factory? |
|---------------------------|---------|---------------|----------|
| 10mM NH4Ac/MeOH | 0.90 | Solvent Waste | Factory |
| 1M HCl/MeOH | 0.90 | Solvent Waste | Factory |
| 1M NaOH | 1.04 | Water Waste | Factory |
| Acetone | 0.78 | Solvent Waste | Factory |
| Acetonitrile | 0.79 | Solvent Waste | Factory |
| Cond. Solution A | 1.00 | Water Waste | Factory |
| Cond. Solution B | 1.00 | Water Waste | Factory |
| Disk Eluting Solution | 1.00 | Water Waste | Factory |
| Ethyl Acetate | 0.90 | Solvent Waste | Factory |
| Hexane | 0.65 | Solvent Waste | Factory |
| Methanol | 0.79 | Solvent Waste | Factory |
| Methylene Chloride | 1.33 | Solvent Waste | Factory |
| MTBE | 0.74 | Solvent Waste | Factory |
| Pentane | 0.63 | Solvent Waste | Factory |
| Petroleum Ether (Ligroin) | 0.65 | Solvent Waste | Factory |

New Solvent Solvent Name:
 Density:
 Waste Destination:
 ID Number:

Figure 7-13. Solvent Library Screen

This screen displays a list of available solvents, including the solvent name, density, waste destination, and indication that it is factory or user defined. When you select a method on the list, the Solvent Library screen displays the following in the bottom right of the screen:

- Solvent Name
- Density
- Waste Destination (either Solvent Waste or Water Waste)
- ID Number

You can change this information for a solvent that is not factory-defined. If you do so, click **Save** to save the information you enter.

Use the buttons on the Solvent Library screen to edit or delete solvents:

- New Solvent** Creates a new solvent titled “New Solvent” and adds it to the end of the Solvent Library. Customize the solvent (including its name) by entering information in

the bottom right of the screen. By default, the new solvent will be user-defined (not factory). Click **Save** to save the information you enter.

- Delete** Deletes a selected user-defined solvent. Be sure you want to delete this solvent because it will be removed as soon as you click **Delete**.
- Duplicate** Duplicates a selected solvent as a new solvent that you can then edit to customize by modifying information in the bottom right of the screen. The duplicate solvent is displayed with the same name as the original followed by an asterisk (*). Change/edit information and then click **Save**.
- Save** Saves all information that you entered/changed.

7.10 PC Setup Screen

Click **PC Setup** on the Admin Menu screen to access the PC Setup screen as shown in Figure 7-14.

Figure 7-14. PC Setup Screen

This screen enables you to customize the following PC settings:

- Administration Password** Indicates the password required for access to the Admin Menu screen. If this field is blank, a password is not required.
- Create Run Report** Specifies whether or not a report is created when a method is run.
- Enable Station Notes Button** Specifies whether or not the Note option is available on the Station screen.
- Background Color** Defines the color displayed for the screen background. You can see the color chosen in the box to the right of **Pick Color**.
- Button Color** Defines the color displayed for buttons. The color chosen is shown on the buttons and in the box to the right of **Pick Color**.
- Restore Colors** Restores background and button colors to the previously stored colors.

| | |
|-----------------------------|--|
| Factory Colors | Restores background and button colors to the original colors received from the factory. |
| PDF Viewer | The PDF viewer is needed to read pdf-formatted reports. This shows the PDF viewer version installed on the system. |
| PDF Viewer Arguments | This entry ensures the PDF viewer can close as well as open pdf-formatted reports. |
| Factory Setpoints | Available for service only. Click the <i>Cancel</i> button to return to the PC Setup screen. |

7.11 Solvent Configuration Screen

Click *Solvent Config* on the Admin Menu screen to access the Solvent Configuration screen (Figure 7-15).

The screenshot shows a window titled "SolventInventoryScreen" with a green background. At the top, it says "Module 1 Solvent Configuration". Below this is a list of 8 modules, each with a number in a box and a solvent name in a dropdown menu:

| | |
|---|--------------------|
| 1 | Reagent Water |
| 2 | Acetone |
| 3 | 10% H2SO4/MeOH |
| 4 | Hexane |
| 5 | Methylene Chloride |
| 6 | Toluene |
| 7 | Methanol |
| 8 | Air Vent |

At the bottom right, there is a "Copy to..." section with four buttons: "Module 1", "Module 2", "Module 3", and "Module 4". At the bottom left, there are "Save" and "Cancel" buttons.

Figure 7-15. Solvent Configuration Screen

For each line 1-7, you can select a solvent for the Module indicated at the top of the screen. In addition, you can copy the configuration to any of the other connected modules.

7.12 Controller Setup Screen

Click **Controller Setup** on the Admin Menu screen to access the Controller Setup screen. There are three pages of settings that can be customized. The default values will be sufficient for most methods and you will not have to adjust these settings unless there is a problem.



Figure 7-16. System Setup Screen (Page 1)

This screen enables you to customize the following system settings:

Pump Loading Wait Time

The Pump Load Wait Time (in seconds) is a delay time from when the WIV opens, to when the vacuum pump is turned on to drain the sample. This wait time allows the Load Sensor time to equilibrate in the water sample. Typically a time of 5 to 10 seconds is sufficient for the sensor equilibration.

Vacuum Pump Manual Drain Speed

Sets the speed of the vacuum pump when the manual Drain feature is used.

Post Purge Delay Time Indicates the time between when rinse solvent dispensing is done and the vacuum pump is turned on. This allows sufficient time for the rinse solvent to drain off the sample bottle wall and onto the SPE disk.

Overflow Drain Time If an Overflow condition is detected, a Warning message is displayed on the PC screen. The operator has the option to Stop the run, or Continue. If the run is to Continued, the vacuum pump will run for this specified Overflow Drain Time. The Overflow Sensor will be monitored again, to confirm if the sample level has dropped below the Overflow sensor. If it has, the run will continue. If not, the warning message will be displayed again, and the drain function run again.

Save Saves the system settings.

Click **Page 2** to display additional settings:

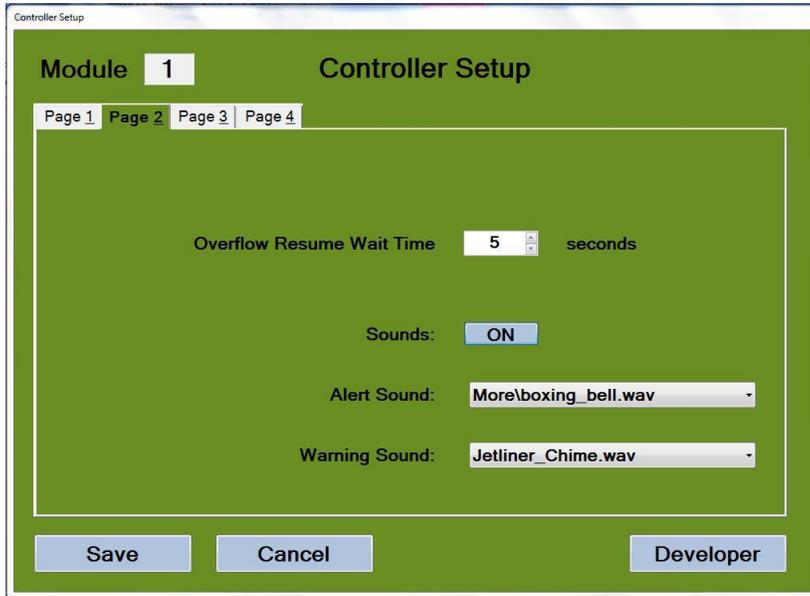


Figure 7-17. Controller Setup Screen (Page 2)

This screen enables you to customize the following system settings:

Overflow Resume Wait Time The Overflow Resume Wait Time (in seconds) is a timer which allows the Overflow Sensor to re-equilibrate and confirm the overflow condition has actually been cleared.

Sounds Indicates whether or not sounds will be issued for an alert or warning.

Alert Sound Issues this sound when an alert occurs.

Warning Sound Issues this sound when a warning occurs.

Click *Page 3* to display additional settings:

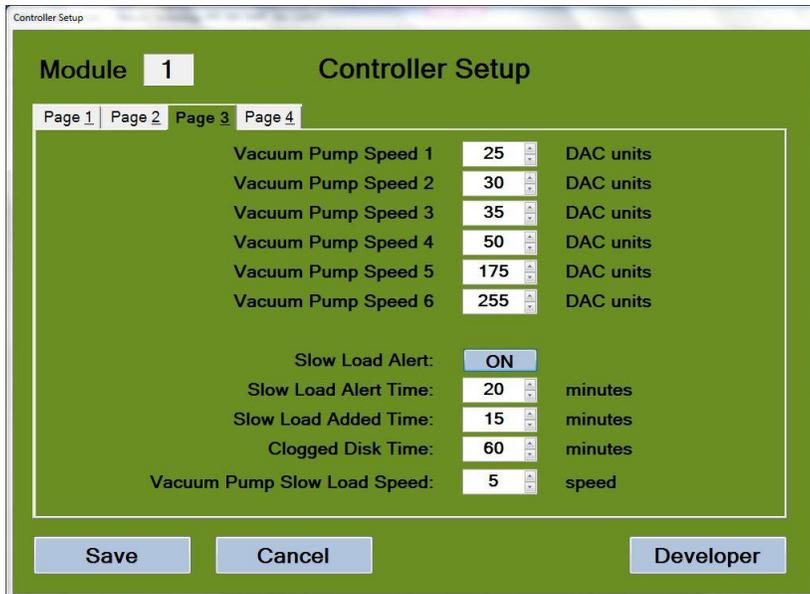


Figure 7-18. Controller Setup Screen (Page 3)

Vacuum Pump Speed 1-6 Not a linear scale and should not be touched unless a change is specified by the manufacturer

| | |
|------------------------------------|--|
| Slow Load Alert | Indicates whether or not an alert occurs if the water sample load time exceeds the “Slow Load Alert Time” (below). It can be turned ON or OFF |
| Slow Load Alert Time | Specifies the time before an alert occurs when the sample flow rate is slow. |
| Slow Load Added Time | Automatically adds this time to Load Step, and before the method advances to the Air Dry Step. This ensures that the entire sample has been removed from the Disk Holder before advancing to the Air Dry Step. |
| Clogged Disk Time | Allows the operator to input a time, such that if the sample takes longer than this time to filter through the SPE disk, to automatically stop the run, and display this warning to the operator. Long sample processing times typically indicate the SPE disk is clogged and the station needs attention. |
| Vacuum Pump Slow Load Speed | The pump speed will automatically be increased to this number to try and clear the slow loading through the disk |

Page 4 is currently unused and may have additional settings in future software versions

7.13 Module Setup Screen

Click *Module Setup* on the Admin Menu screen to access the Module Setup screen, shown in Figure 7-19.

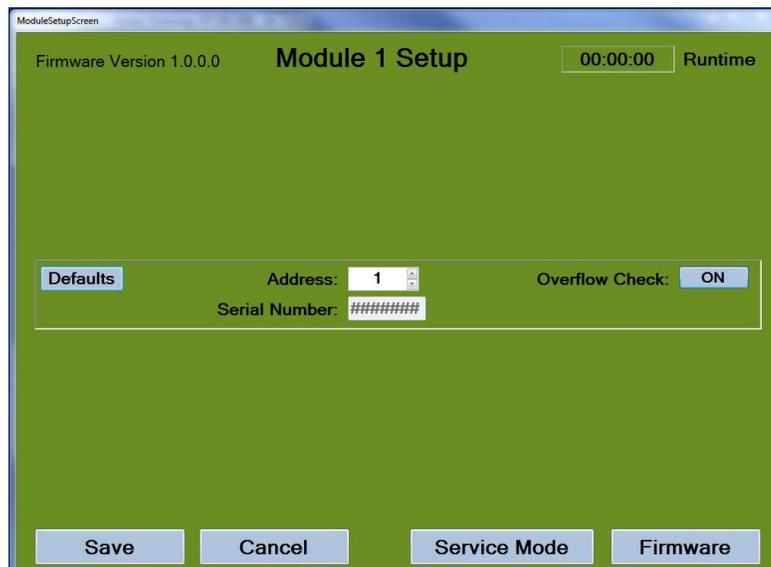


Figure 7-18. Module Setup Screen

This screen enables you to customize the following module settings:

| | |
|-----------------------|---|
| Defaults | Restores all factory settings for the module |
| Address | Indicates an address unique to this module. |
| Serial Number | Displays the module serial number sensed when a Module is connected |
| Overflow Check | Indicates whether or not Overflow Check is enabled so that a warning is issued when a sample overflow occurs. |

| | |
|---------------------|--|
| Fan | Allows the internal solvent vapor exhaust fan to be turned ON and OFF. The fan should remain on, even in a hood, because it also provides cooling to the electrical components inside the unit. If the fan is off in a warm environment, this could lead to overheating and damage to the unit. Changing this setting will take effect at the next power up of the system. |
| Save | Saves the module settings. |
| Service Mode | Available for service only. Press the Esc key to return to the PC Setup screen. |
| Firmware | Allows firmware code updates to be implemented. |

7.14 Diagnostics Screen

The Diagnostics screen is intended for service only.

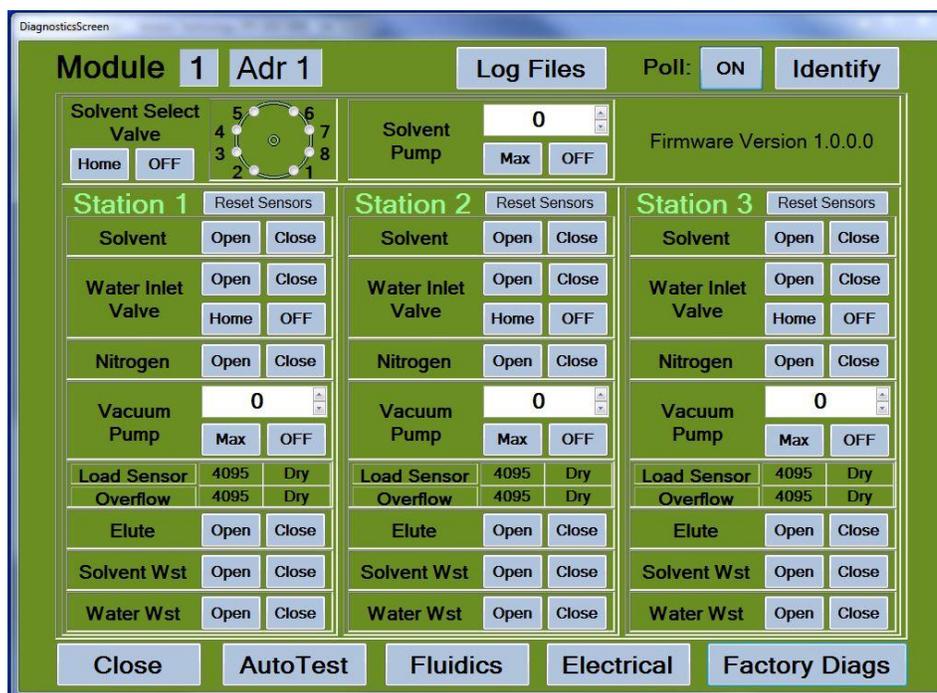


Figure 7-20. Diagnostic Screen

Allows control over all pumps, both Liquid Dispensing and Vacuum Pumps, and all solenoid valves. It should only be used for troubleshooting the system.

Notes:

Part II: Maintenance and Troubleshooting

8 Maintenance

The following information is designed to assist with the general maintenance and replacement of standard components on the SPE-DEX 5000 System. If any questions arise, contact Horizon Technology, Inc. for assistance.

8.1 Daily Maintenance

8.1.1 Cleaning the Water Inlet Valve (WIV):

The WIVs should be cleaned after each use to reduce the risk of cross-contamination between runs. To clean the WIV, remove from the extractor and rinse with warm water in the normal flow direction (top to bottom as installed on the extractor). The valve will be in the closed position when removed from the extractor. While under running warm water, rotate the valve stem slowly through a full 360° of rotation until it returns to the normal (valve closed) position. Valve rotation may be difficult by hand, but the WIV Wrench (Horizon P/N 02-5792) can be used for additional leverage as shown in Figure 8-1. Figure 8-2 shows the full rotation procedure.



Figure 8-1. Using the supplied WIV to Rotate the Valve

Note – WIVs should be stored off of the unit, not installed on the WIV platforms. This helps to prolong the O-ring life on the WIV platforms.

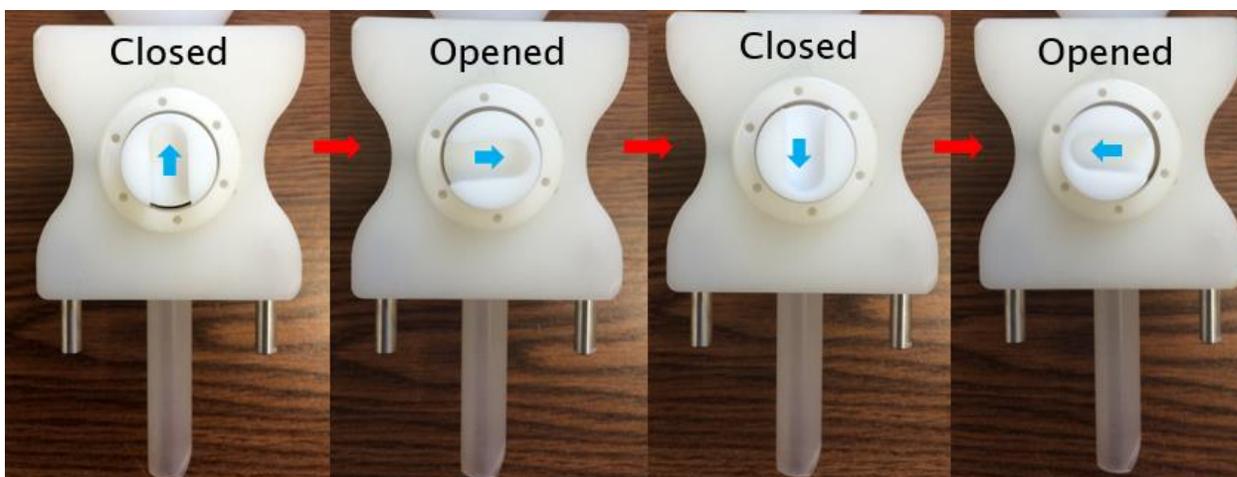


Figure 8-2. Full Rotation of the WIV Shown

8.1.2 Cleaning/Inspecting Solvent Lines

Solvent lines and solvent inlet filters may have dust and particles sticking to them, especially after opening the solvent containers for filling and removal/re-installation of the lines. Before re-installing the lines, run some methanol or other appropriate solvent down the lines to knock off dirt, dust and residual contaminants from handling. Place the solvent inlet filters in a large beaker (Figure 8-3) and elevate the solvent lines to collect the methanol for disposal. For routine maintenance, the lines may be dried and returned to service.

However, for initial system startup after installation (and anytime a more thorough decontamination is desired), it is recommended to further clean the solvent inlet filters. Soak the solvent inlet filters in a beaker of clean solvent (cover the solvent inlet filters completely with acetone or DCM). After soaking for several minutes, remove the lines and place into a clean, empty beaker. Next, run the solvent delivery pump while alternately selecting ports 1 through 7 on the Solvent Select Valve. This can be accomplished from the Diagnostic screen, as shown in Figure 8-4.

- Step 7:** Place WIV with sample bottle on STATION 1
- Step 8:** Place Empty disk holder on STATION 1
- Step 9:** On the Diagnostic screen, select Station 1 Solvent OPEN
- Step 10:** Station 1 Water Inlet Valve CLOSE
- Step 11:** Station 1 Vacuum Pump MAX
- Step 12:** Station 1 Solvent Waste OPEN
- Step 13:** SELECT SOLVENT PORT (e.g., 1)
- Step 14:** SOLVENT PUMP MAX (Let pump run until there are no droplets in the solvent line).
- Step 15:** With pump still running SELECT NEXT SOLVENT PORT. Let the pump run until there are no droplets in the solvent line
- Step 16:** Repeat steps 8 and 9 until all solvents 1 through 7 (or as many lines are connected) are completed.

Step 17: Turn off Vacuum Pump, Solvent Pump, and close Solvent Waste and Solvent valves on the Diagnostic screen.

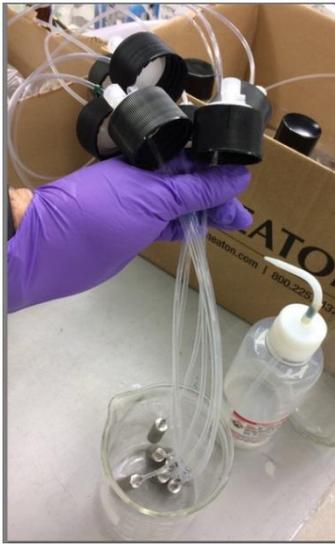


Figure 8-3. Solvent Line Sinkers being Cleaned

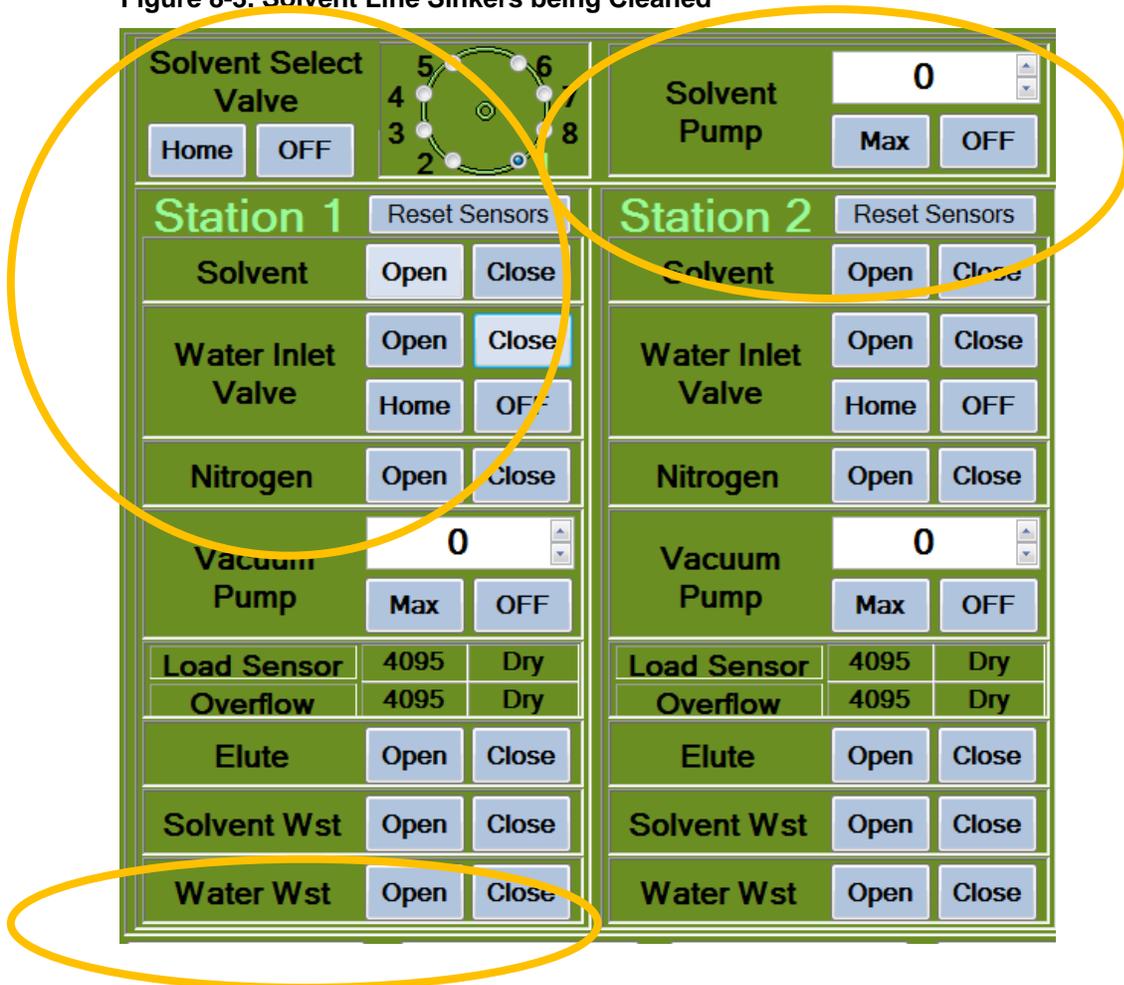


Figure 8-4. Diagnostic Screen Functions that Would be Used in Solvent Line Cleaning

Use care when handling solvent lines, as improper handling can lead to kinked lines. Inspect the solvent lines for any signs of kinking. Kinks are most likely to occur either near the solvent bottle caps or near the 8-port valve at the rear of the extractor. If any kinks are found, squeeze the solvent line gently across the kinked area using two fingers to smooth out the kink. Slightly kinked lines will not affect system performance.

8.1.3 Cleaning Thermistor Sensors

Rinse the level sensors with acetone. Collect the waste acetone with a beaker or cup placed on the disk holder platform.

8.1.4 Cleaning the 19/22 Elution Collection Adapter and Extension Line

With the disk holder platform in the raised position, rinse the collection adapter and the outside of the extension line with acetone. Use a large beaker on the lower platform to collect the solvent.

8.1.5 Cleaning the Extractor System – Exterior

The extractor should be wiped down daily with a soft cloth soaked in warm water. For stain removal, isopropyl alcohol (IPA) may be used on any external surface.

8.1.6 Running the Cleaning Method

The method “Clean and Background Check” can be run anytime to help ensure that the Extractor’s internal tubing and components are clean and free from contamination. The method contains instructions on how to set up the system with empty disk holders, solvent selections, and fraction collection for background contamination checks.

8.2 Weekly Maintenance/Inspection

8.2.1 Running the Cleaning Method

The method “Clean and Background Check” can be run anytime (weekly is recommended) to help ensure that the Extractor’s internal tubing and components are clean and free from contamination. The method contains instructions on how to set up the system with empty disk holders, solvent selections, and fraction collection for background contamination checks.

8.2.2 Inspect WIV Gasket

The WIV utilizes a gasket to seal to the sample bottle or cap adapter (Horizon P/N 02-0560-02). This gasket should be inspected weekly for tears. If any are found, or if the gasket fails to seal during operation (this would most likely show up as small air bubbles during the loading step), then replace the gasket with a new one. To remove the gasket, it may be necessary to rotate it counter-clockwise to work it out of the threaded portion of the WIV.

8.3 Monthly Maintenance/Inspection

8.3.1 Re-sealing the Luer Fitting on Disk Holders

The Luer fitting on the underside of the disk holders relies upon Teflon® tape to develop a tight seal to the disk holder base. Over time, this area may develop minor vacuum leaks which could slow the rate of processing samples (the risk of water leaks is very minimal). If slow sample processing is suspected, or otherwise on a monthly schedule, it is recommended to remove the Luer fitting and service. To repair the seal, remove all of the old Teflon® tape and re-wrap (in a clockwise direction) about 2 to 3 times around the threaded portion of the fitting. Ensure that no tape extends past the end of the fitting such that it could be exposed inside the disk holder. Re-install the Luer fitting, do not overtighten.

8.3.2 Inspect WIV Platform O-ring

The Solvent Dispensing Nozzles utilize special chemically-resistant O-rings and should not require regular service or replacement. However, if the O-rings become damaged it could compromise the sealing capability of the solvent delivery system. Inspect the O-rings monthly for any signs of rips or tears.

8.4 Annual Maintenance/Inspection

8.4.1 Inspecting the Solvent Waste, Water Waste, and Vent Lines

Inspect the solvent waste, water waste, and vent lines for signs of kinking, leaking, cracking or loose fittings at either the extractor or the waste containers. These lines can be replaced if necessary by ordering Horizon P/N 50-0225 (kit contains two lines, a total of 9 lines are used on the SPE-DEX 5000).

The Frequency of the maintenance items described in this section are summarized in Table 8-1.

Table 8-1. Maintenance Summary for the SPE-DEX 5000

| | Daily | Weekly | Monthly | Annually |
|---|-------|--------|---------|----------|
| Cleaning WIV | X | | | |
| Clean/Inspect Solvent Lines | X | | | |
| Clean Level Sensors | X | | | |
| Clean Collection Adapter and Elution Line | X | | | |
| Clean Extractor – Exterior | X | | | |
| Run the Clean and Background Check Method | | X | | |
| Inspect WIV Gasket | | X | | |
| Inspect/Service Luer Fitting on Disk Holders | | | X | |
| Inspect WIV Platform O-ring (Solvent Dispensing Nozzle) | | | X | |
| Inspect Solvent Waste, Water Waste, and Vent Lines | | | | X |

8: Maintenance

Notes:

9 Troubleshooting

The SPE-DEX 5000 System is engineered to perform in a demanding environment. This system is a mixture of electrical, mechanical, and pneumatic subsystems. Unfamiliarity with the operation of the system, especially with new system start-ups, can be intimidating. Before calling for assistance, attempt to rectify any issues by referring to the troubleshooting tips below. It is important to keep a record of all conditions used and all the various remediation attempts. This will assist a Horizon Technology technical representative in pinpointing the problem quickly.

Note: No matter what the problem is, remember that opening the cover of the Extractor Module(s) voids the warranty, unless instructed to do so by a Horizon Technology technical representative.

Product quality, reliability, and customer satisfaction are important to us. If the problems cannot be resolved, please contact a Horizon Technology technical representative without delay.

9.1 Troubleshooting Guide

Logging Files

If problems occur during operation of the Extractor, it may be helpful to turn on the logging function to assist Horizon Technology service personnel in diagnosing the issue. The logging feature can be accessed on the diagnostic panel (admin menu, diagnostics). To turn on logging, press the button at the top of the screen titled *LOG FILES*. Select both COMM LOG and EVENT LOG (the buttons will display current ON/OFF state). It is recommended to turn off logging after it is no longer required, since the files will continue to take up more space on the hard drive of the controller computer.

Troubleshooting Table

| Symptom | Probable Cause | Solution |
|--|---|--|
| The SPE-DEX 5000 Software or Extractor is not responding | The USB cable from the equipment is not connected | Power off the Extractor and the Close the SPE-DEX 5000 Software on the PC. Make sure the USB cable is securely connected at both ends. Restart the Extractor and then the SPE-DEX 5000 Software. |
| Solvents are not dispensed | Volume is not correctly programmed in the method | Verify the dispense volume programmed for the method. |
| Solvents are not dispensed | Solvent bottles are empty or solvent inlet filters are above the level of solvent | Verify that the solvent bottles are filled and that the solvent inlet filters are submerged. |
| Solvents are not dispensed | Crimped solvent lines prevent solvent flow | Check for crimped or pinched solvent lines that could prevent solvent flow. Also verify all solvent line connections at the rear of the extractor. |
| Solvents are not dispensed | Solvents may be leaking from the system prior to the disk holder destination | Check for liquid leaks. |
| Conditioning or rinse solvent overflows | Too much solvent is programmed for the condition- | Verify the dispense volume programmed for the method. |

9: Troubleshooting

| | | |
|--|---|--|
| | ing or rinse step | |
| Conditioning or rinse solvent overflows | Not enough drain time is allowed before the step advances to the next | Verify that the conditioning steps have sufficient drain times. |
| Conditioning or rinse solvent overflows | There may be a crimp or clog in the drain system | Verify that the solvent is draining to the solvent waste container during the drain steps. |
| Conditioning or rinse solvents do not drain | Not enough time was programmed for the drain step | Verify the method used has sufficient drain time programmed. |
| Conditioning or rinse solvents do not drain | There may be a crimp or clog in the drain system | Examine the solvent to waste line for any crimps, clogs or damage that may impede the flow. |
| Conditioning or rinse solvents do not drain | The Luer fitting on the bottom of the disk holder may be drawing in air | Inspect the disk holder Luer fitting, remove and replace Teflon® tape seal with 2-3 turns of Teflon® tape on threaded portion (see section 8, Maintenance). |
| Water Inlet Valve does not open to dispense sample | The valve may be too tight | Remove Water Inlet Valve and try to turn the valve stem to exercise the valve. The Valve should be tight but still turn by hand. If necessary, use a large screwdriver as leverage in the slot on the end of the valve stem. |
| Sample Overflow sensor is triggered | The sample bottle is not feeding into the disk holder smoothly | Examine the sample container being used as small defects in the glassware may cause air leaks. These defects may take the form of chips, cracks, or waves in the sealing surface of the rim of the sample bottle. Bottles with these types of defects should not be used. |
| Sample Overflow sensor is triggered | The thermistor sensor is set incorrectly | Check the distance of the thermistor from the platform. Refer to section 6 for proper overflow sensor settings. |
| Sample Overflow sensor is triggered | One of the gaskets has failed | Examine the gasket on the water inlet valve and cap adapters being used. This is the gasket that forms a seal between the sample bottle and Inlet valve or, if using a cap adapter, between the cap adapter and inlet valve. Verify that all gaskets are correctly in place and that they are not worn or damaged. |
| Sample Overflow sensor is triggered | Possibly a false alarm | Observe the method that caused the issue while performing a test run. The conditioning solvent level should be low enough such that it does not cover the opening of the WIV down tube at the end of each conditioning step. If it does cover the opening of the down tube, the vent purge portion of each dispensing step may cause splashing |

| | | |
|---|---|---|
| | | of solvent onto the overflow sensor, which can cause a false overflow alarm. |
| Sample Overflow sensor is triggered | Possibly a false alarm | False overflow alarms may be caused by changes in air currents during operation. While a method is running, avoid turning on or off a ventilation hood, and avoid excessive personnel motion or foot traffic in very close proximity to the disk holders. In cases where air current changes cannot be avoided during operation, the optional Vapor Shield (Horizon P/N 50-5012, 3 required per extractor) may be used to ensure reliable level sensor operation. |
| Overflow false alarm | Airflow changes during loading step cause the overflow sensor to read wet | Select the station screen for the station that is in overflow. Verify that the overflow is a false alarm (overflow sensor is actually dry). Select the <i>Sensors</i> button at the top center of the station screen. Select the <i>Dry</i> button next to the “Overflow” displayed in red. This will reset the overflow sensor to the dry state. Select <i>Continue</i> at the bottom of the screen in the Overflow Warning section. |
| Sample is not draining to waste or draining very slowly | Drain program may not be set-up as expected | Verify the drain time programmed in the method if using a method other than factory-supplied methods |
| Sample is not draining to waste or draining very slowly | Possible pump failure | Verify that the vacuum pump is on and working. While running a method, increase the vacuum pump speed (during a drain step) to 6 and listen for the sound of the pump to change to confirm operation. |
| Sample is not draining to waste or draining very slowly | Particulates may be clogging disk | If running a dirty sample the disk may be clogged. Use the Fast Flow disk holder with pre-filters and a screen for samples with particulates. |
| Sample is not draining to waste or draining very slowly | A crimp or kink may be slowing liquid flow | Verify that all waste lines (water to waste and solvent to waste) are free of kinks and securely connected. |
| Sample is not draining to waste or draining very slowly | There may be an air leak compromising the vacuum strength | The disk holder assembly components need to be tight to form a good seal. If the components are not tight, air will flow through the parts when vacuum is pulling thus reducing efficiency. Remove, inspect, and re-seal the Luer fitting on the bottom of the disk holders (see section 8, Maintenance). |

9: Troubleshooting

| | | |
|---|--|---|
| Sample is not draining to waste or draining very slowly | Dirt may be clogging the drainage system | Dirt may be trapped in the water to waste valve. Flush the valve by filling a disk holder cup with warm water and using the Drain and then Water Waste buttons for the station in question. |
| No rinse spray into the sample bottle | Solvent bottle may be empty | Verify that the solvent bottles are filled at least enough to completely cover the solvent inlet filters. |
| No rinse spray into the sample bottle | Crimps or kinks in the solvent delivery lines will impede the flow of solvents | Examine the solvent delivery lines and ensure that no kinks are present. Kinks usually appear at the fittings on either the solvent bottles or the connection to the extractor. Straighten the kinks if possible and replace any damaged lines if necessary. |
| No rinse spray into the sample bottle | Rinse spray line is clogged | The water inlet valves have an opening through which the rinses are sprayed. The rinse opening is the metallic insert located on the inlet valve, below the valve stem. Make sure that there is no blockage of the opening. If the rinse opening is blocked with debris, the rinse spray may be weak and/or off center. The valves can be sonicated in a warm water bath to dislodge the debris from the rinse opening. Alternatively, a fine needle-like tool or syringe cleaner can be used to clear the opening. |

Notes:

Part III: Appendices

A Preprogrammed Methods (Factory)

The following methods are included in with SPE-DEX 5000 software.

| US EPA 525.2 47 mm Disk Holder | | Dry Time (s) | Dis-pense Solvent (mL) | Vent Purge Time (s) | Vacu-um Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|-----------------------------------|--|---|------------------------|---------------------|-------------------|---------------------------|-------------------|---------------|----------------|
| Step 1: | Condition 1 Methylene Chloride | | 15 | 60 | 2 | OFF | 1 | 20 | 30 |
| Step 2: | Condition 2 Ethyl Acetate | | 11 | 60 | 2 | OFF | 1 | 20 | 30 |
| Step 3: | Condition 3 Methanol | | 11 | 60 | 2 | OFF | 1 | 60 | 2 |
| Step 4: | Condition 4 Reagent Water | | 9 | 30 | 2 | OFF | 1 | 5 | 5 |
| Step 5: | Condition 5 Reagent Water | | 9 | 60 | 2 | OFF | 1 | 30 | 0 |
| Step 6: | Load Sample | Sample Flow Rate 2, Done Loading Sample Delay 0 | | | | | | | |
| Step 7: | Air Dry | 60 | | | 6 | OFF | | | |
| Step 8: | Elute Sample Container Ethyl Acetate | | 8 | 60 | 2 | OFF | 1 | 30 | 45 |
| Step 9: | Elute Sample Container Methylene Chloride | | 8 | 15 | 2 | OFF | 1 | 30 | 45 |
| Step 10: | Elute Sample Container Methylene Chloride | | 8 | 15 | 2 | OFF | 1 | 30 | 45 |
| Step 11: | Elute Sample Container Methylene Chloride | | 8 | 15 | 6 | OFF | 2 | 30 | 60 |

| US EPA 608 (DCM) 47 mm Disk Holder | | Dry Time (s) | Dis-pense Solvent (mL) | Vent Purge Time (s) | Vacu-um Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|---------------------------------------|-----------------------------------|---|------------------------|---------------------|-------------------|---------------------------|-------------------|---------------|----------------|
| Step 1: | Condition 1 Methylene Chloride | | 20 | 30 | 6 | OFF | 2 | 60 | 90 |
| Step 2: | Condition 2 Methanol | | 20 | 60 | 3 | OFF | 3 | 60 | 15 |
| Step 3: | Condition 3 Methanol | | 10 | 60 | 3 | OFF | 1 | 30 | 15 |
| Step 4: | Condition 4 Reagent Water | | 10 | 60 | 2 | OFF | 2 | 5 | 15 |
| Step 5: | Load Sample | Sample Flow Rate 2, Done Loading Sample Delay 5 | | | | | | | |

A: Preprogrammed Methods (Factory)

| | | | | | | | | | |
|----------|--|-----|----|----|---|-----|---|----|----|
| Step 6: | Wash Sample Container Reagent Water | | 10 | 30 | 6 | OFF | 1 | 10 | 15 |
| Step 7: | Air Dry | 240 | | | 6 | OFF | | | |
| Step 8: | Elute Sample Container Acetone | | 10 | 20 | 2 | OFF | 1 | 30 | 45 |
| Step 9: | Elute Sample Container Methylene Chloride | | 10 | 15 | 2 | OFF | 1 | 30 | 45 |
| Step 10: | Elute Sample Container Methylene Chloride | | 10 | 15 | 2 | OFF | 1 | 30 | 45 |
| Step 11: | Elute Sample Container Methylene Chloride | | 10 | 15 | 6 | OFF | 1 | 30 | 60 |

| US EPA 608 (DCM) Fast Flow Disk Holder 1 µm, 5µm, screen | | Dry Time (s) | Dispense Solvent (mL) | Vent Purge Time (s) | Vacuum Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|--|--|--|-----------------------|---------------------|------------------|---------------------------|-------------------|---------------|----------------|
| Step 1: | Condition 1 Methylene Chloride | | 40 | 30 | 6 | OFF | 3 | 60 | 120 |
| Step 2: | Condition 2 Methanol | | 40 | 60 | 3 | OFF | 3 | 60 | 30 |
| Step 3: | Condition 3 Methanol | | 20 | 60 | 3 | OFF | 3 | 30 | 30 |
| Step 4: | Condition 4 Reagent Water | | 20 | 60 | 3 | OFF | 3 | 15 | 30 |
| Step 5: | Load Sample | Sample Flow Rate 3, Done Loading Sample Delay 45 | | | | | | | |
| Step 6: | Wash Sample Container Reagent Water | | 20 | 30 | 6 | OFF | 3 | 20 | 30 |
| Step 7: | Air Dry | 360 | | | 6 | OFF | | | |
| Step 8: | Elute Sample Container Acetone | | 40 | 20 | 3 | OFF | 3 | 60 | 90 |
| Step 9: | Elute Sample Container Methylene Chloride | | 40 | 15 | 3 | OFF | 3 | 60 | 90 |
| Step 10: | Elute Sample Container Methylene Chloride | | 40 | 15 | 3 | OFF | 3 | 60 | 90 |
| Step 11: | Elute Sample Container Methylene Chloride | | 40 | 15 | 6 | OFF | 3 | 60 | 120 |

A: Preprogrammed Methods (Factory)

| US EPA 608 (Hexane) 47 mm Disk Holder | | Dry Time (s) | Dis- pense Solvent (mL) | Vent Purge Time (s) | Vacu- um Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|--|--|--|--|------------------------------------|---------------------------------------|--|------------------------------|--------------------------|-------------------------------|
| Step 1: | Condition 1 Hexane | | 20 | 70 | 6 | OFF | 2 | 60 | 90 |
| Step 2: | Condition 2 Acetone | | 15 | 40 | 6 | OFF | 2 | 60 | 60 |
| Step 3: | Condition 3 Methanol | | 20 | 60 | 3 | OFF | 3 | 60 | 15 |
| Step 4: | Condition 3 Methanol | | 10 | 60 | 3 | OFF | 1 | 30 | 15 |
| Step 5: | Condition 4 Reagent Water | | 10 | 60 | 2 | OFF | 2 | 5 | 15 |
| Step 6: | Load Sample | Sample Flow Rate 2, Done Loading Sample Delay 5 | | | | | | | |
| Step 7: | Wash Sample Container Reagent Water | | 10 | 30 | 6 | OFF | 1 | 10 | 15 |
| Step 8: | Air Dry | 240 | | | 6 | OFF | | | |
| Step 9: | Elute Sample Container Acetone | | 10 | 20 | 2 | OFF | 1 | 30 | 45 |
| Step 10: | Elute Sample Container Hexane | | 10 | 35 | 2 | OFF | 1 | 30 | 45 |
| Step 11: | Elute Sample Container Hexane | | 10 | 35 | 2 | OFF | 1 | 30 | 45 |
| Step 12: | Elute Sample Container Hexane | | 10 | 35 | 6 | OFF | 1 | 30 | 60 |

| Clean and Back- ground Check, Empty Disk Holder | | Dry Time (s) | Dis- pense Solvent (mL) | Vent Purge Time (s) | Vacu- um Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|--|---|-----------------------------|--|------------------------------------|---------------------------------------|--|------------------------------|--------------------------|-------------------------------|
| Step 1: | Condition 1 Ac- etone | | 15 | 0 | 6 | OFF | 0 | 60 | 30 |
| Step 2: | Condition 2 Acetone | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |
| Step 3: | Condition 3 Methylene Chlo- ride | | 15 | 0 | 6 | OFF | 0 | 60 | 30 |
| Step 4: | Condition 3 Methylene Chlo- ride | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |
| Step 5: | Condition 4 Acetone | | 15 | 0 | 6 | OFF | 0 | 60 | 30 |
| Step 6: | Wash Sample Container Acetone | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |
| Step 7: | Wash Sample Container | | 15 | 0 | 6 | OFF | 0 | 60 | 30 |

A: Preprogrammed Methods (Factory)

| | | | | | | | | | |
|-----------------|--|---|----|----|---|-----|---|---|----|
| | Methylene Chloride | | | | | | | | |
| Step 8: | Wash Sample Container Methylene Chloride | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |
| Step 9: | Elute SPE Disk Acetone | | 15 | 0 | 6 | OFF | 2 | 0 | 30 |
| Step 10: | Elute SPE Disk Acetone | | 15 | 30 | 6 | OFF | 2 | 0 | 30 |
| Step 11: | Elute SPE Disk Methylene Chloride | | 15 | 0 | 6 | OFF | 2 | 0 | 30 |
| Step 12: | Elute SPE Disk Methylene Chloride | | 15 | 30 | 6 | OFF | 2 | 0 | 30 |
| Step 13: | Pause with Message | Empty solvent elute rinse to solvent waste, then clean collection flask before returning it to the elute port of the station. Press resume. This fraction to be concentrated and analyzed for background. | | | | | | | |
| Step 14: | Elute Sample Container Acetone | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |
| Step 15: | Elute Sample Container Methylene Chloride | | 15 | 30 | 6 | OFF | 0 | 0 | 30 |

0:



Method Worksheet

16 Northwestern Drive, Salem, NH 03079
 Telephone: (603) 893-3663
 Toll-Free: (800) 997-2997 USA only
 Website: www.horizontechinc.com

Method Name: _____

Name: _____ **Phone:** _____

Company: _____

| Disk Holder | | Dry Time (s) | Dis-pense Solvent (mL) | Vent Purge Time (s) | Vacu-um Pump Flow | Nitrogen Blanket (ON/OFF) | Saturate Time (s) | Soak Time (s) | Drain Time (s) |
|-------------|------------------------|---|------------------------|---------------------|-------------------|---------------------------|-------------------|---------------|----------------|
| Step 1: | Condition 1 | | | | | OFF | | | |
| Step 2: | Condition 2 | | | | | OFF | | | |
| Step 3: | Condition 3 | | | | | OFF | | | |
| Step 4: | Condition 4 | | | | | OFF | | | |
| Step 5: | Condition 5 | | | | | OFF | | | |
| Step 6: | Load Sample | Sample Flow Rate 2, Done Loading Sample Delay 0 | | | | | | | |
| Step 7: | Air Dry | | | | | OFF | | | |
| Step 8: | Elute Sample Container | | | | | OFF | | | |
| Step 9: | Elute Sample Container | | | | | OFF | | | |
| Step 10: | Elute Sample Container | | | | | OFF | | | |
| Step 11: | Elute Sample Container | | | | | OFF | | | |

B Parts List

The following items are necessary to run the SPE-DEX 5000 System:

- Water Inlet Valves (WIV) (3)
- SPE Disk Holder Assemblies: 47 mm, 50 mm, 100 mm, or Fast Flow Disk Holder
- SPE Disks (47 mm for SVOC or O&G or 90 mm for O&G)
- SPE Prefilters (100 mm)
- Solvent containers for the needed solvents
- Solvents to run the desired methods
- A Solvent Waste container (of sufficient capacity)
- A Water Waste container (of sufficient capacity)
- Optional - One 10 foot section of Solvent Exhaust Hose, for benchtop operation

Please Contact the Sales Department at Horizon Technology for part numbers and current pricing, or visit our website at www.horizontechnic.com.

B.1 SPE Disk Holder Assemblies

The Disk Holder Assembly allows the use of the SPE disk, Prefilters, and Fine Mesh Screens.

| Part Number | Description |
|--------------|--|
| 50-5018-01 | 47 mm Holder. Includes metal collar, 6-mm riser, cup and support screen |
| 50-2767 | Fast Flow Holder Assembly, 100 mm wide, uses standard 47-mm disks, Includes metal collar, riser, cup, support screen, fine mesh screen, 5 µm prefilter sample pack, 1 µm prefilter sample pack |
| 50-0809-01 | 90 mm Holder. Includes metal collar, riser, cup and support screen (used for Oil & Grease analysis) |
| 31-0454 | Dirty Sample Fine Mesh Screen - Fits 47-mm Disk Holder Assembly |
| 31-0457 | Dirty Sample Fine Mesh Screen - Fits 90-mm Disk Holder Assembly |
| 49-0665 | Dirty Sample Fine Mesh Screen - 100 mm for use with 50-2767 disk holder |
| FFAP-47-HT | Atlantic Fast Flow Prefilter, 47 mm, coarse 5.0 µm (Pkg of 50) |
| FFP-47-HT | Pacific Oil & Grease Fast Flow Pre-filter 47 mm (Pkg of 50) |
| FFP-90-HT | Pacific Oil & Grease Fast Flow Pre-filter 100 mm (Pkg of 50) |
| FFAP-100-HS1 | Atlantic Fast Flow Prefilter 100 mm Fine 1.0 µm (Pkg of 50) For use with 50-2767 |
| FFAP-100-HS5 | Atlantic Fast Flow Prefilter 100 mm Coarse 5.0 µm (Pkg of 50) For use with 50-2767 |
| 49-2620-01 | Carbon Cartridge Max Detect 20CC, Kit 16 per pack |
| 49-2620 | Carbon Cartridge 20 CC, Kit 16 per pack |

B.2 SPE Disks

| Part Number | Description |
|--------------|---|
| 47-2346-02 | Atlantic® C-18 Disks, 47 mm (Pkg of 24) |
| 47-2346-13 | Atlantic High Capacity C18 Disks, 47 mm (Pkg of 24) |
| 47-2346-06 | Atlantic DVB-D Disks, 47 mm (Pkg of 24) |
| 47-2346-08 | Atlantic HLB-L Disks, 47 mm (Pkg of 40) |
| 47-2346-09 | Atlantic HLB-M Disks, 47 mm (Pkg of 30) |
| 47-2346-10 | Atlantic HLB-H Disks, 47 mm (Pkg of 24) |
| 47-2346-11 | Atlantic One Pass Disks, 47 mm (Pkg of 24) |
| 1664-100-PHT | Pacific® Disk Premium 100 mm -50/box |
| 1664-47-PHT | Pacific Disk Premium 47 mm -50/box |
| 1664-90-HT | Pacific Disk 1664A 100 mm -50/box |
| 1664-47-HT | Pacific Disk 1664A 47 mm -50/box |

B.3 Collection Vessels

| Part Number | Description |
|-------------|--|
| 27-0476-01 | 125-mL Erlenmeyer with stopper, 19/22 neck |
| 03-5000-01 | 250-mL Erlenmeyer with stopper, 19/22 neck |
| 160-0001-02 | VOA Vial Adapter |
| 22-0687 | Clamp, Blue #19 to secure vessel to SPE-DEX 5000 |

B.4 Bottle Cap Adapters for the SPE-DEX 5000 Extractor Module

The Water Inlet Valves (WIV) for the SPE-DEX 5000 System are designed to fit a 33x400 narrow-mouth bottle (Boston round bottle). If a larger sample bottle will be used, select the bottle cap adapter that will fit your sample bottles. For custom bottle adapters, contact Horizon Technology.

| Part Number | Description |
|-------------|--|
| 150-0005-01 | 70x400 adapter for 1 L jar |
| 150-0006-01 | 89x400 adapter for 1 L jar |
| 150-0009-04 | 53x400 adapter for 1-L wide mouth bottle |
| 150-0010-01 | 58x400 adapter for 1-L French Square |
| 150-0012-01 | 48x400 adapter |
| 150-0014-01 | 63x400 adapter |
| 150-0015-01 | 83x400 adapter |
| 150-0025 | 33x430 adapter |

Notes

C Warranty

Limited Warranty

Horizon Technology, Inc. (“Horizon”) warrants the *SPE-DEX 5000 Automated Extraction System* (Product) against defects in material or workmanship as follows:

1. **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.
2. **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.

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-(603)-893-3663

1-(800)-997-2997 (Toll-free, US-only)

Or write to:

Horizon Technology, Inc.

16 Northwestern Drive

Salem, NH 03079

Email: info@horizontechinc.com

Website: www.horizontechinc.com

Notes: