

# PICARRO



SDM Users Guide  
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Part 30010  
Rev. B

# PICARRO

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This manual assumes the user is familiar with the operation of the Windows XP computer system, the Picarro L1115-i including use of the coordinator for liquid injections, and that the L1115-i with vaporizer is fully installed and running. The autosampler should be disconnected from the analyzer, please see the L1115-i for analyzer and vaporizer instructions.

## PreInstallation Requirements

Picarro L1115-i analyzer and liquid sampler vaporizer running host software version 3.5.7-3 or higher. The host software version can be checked in the analyzer GUI software under Help, About GUI. If the host software does not meet requirements Picarro can remotely update the software if the analyzer is connected to the internet, please contact Picarro (<http://www.picarro.com/resources/support.php> or +1-408-962-3900) for further details on software updates.

Dry air supply with a moisture level of <200ppmv, 350sccm, ambient pressure. The SDM comes with an air pump that draws in room air and supplies the correct pressure and flow when connected to a gas drying unit such as the recommended model 27070 from Drierite® Company. The recommended model holds 50 g of water and is suitable for field use. The user must supply the gas drying unit (or equivalent) along with the necessary tubing and adapters to connect the ¼” Swagelok compression style fitting at the outlet of the pump and the male 1/8” Swagelok compression style fitting at the injector assembly to the gas drying unit. Recommend parts are SS-400-6 (1/4” Swagelok Union, SS-400-6-1 (1/4” to 1/8” Swagelok Reducing Union), SS-201-PC (1/8” Swagelok port connector) and ¼” OD Bev-A-Line IV tubing .

## Installation

Picarro strongly recommends users first set up the Standards Delivery Module (SDM) in a laboratory to familiarize themselves with its operation before a field deployment.

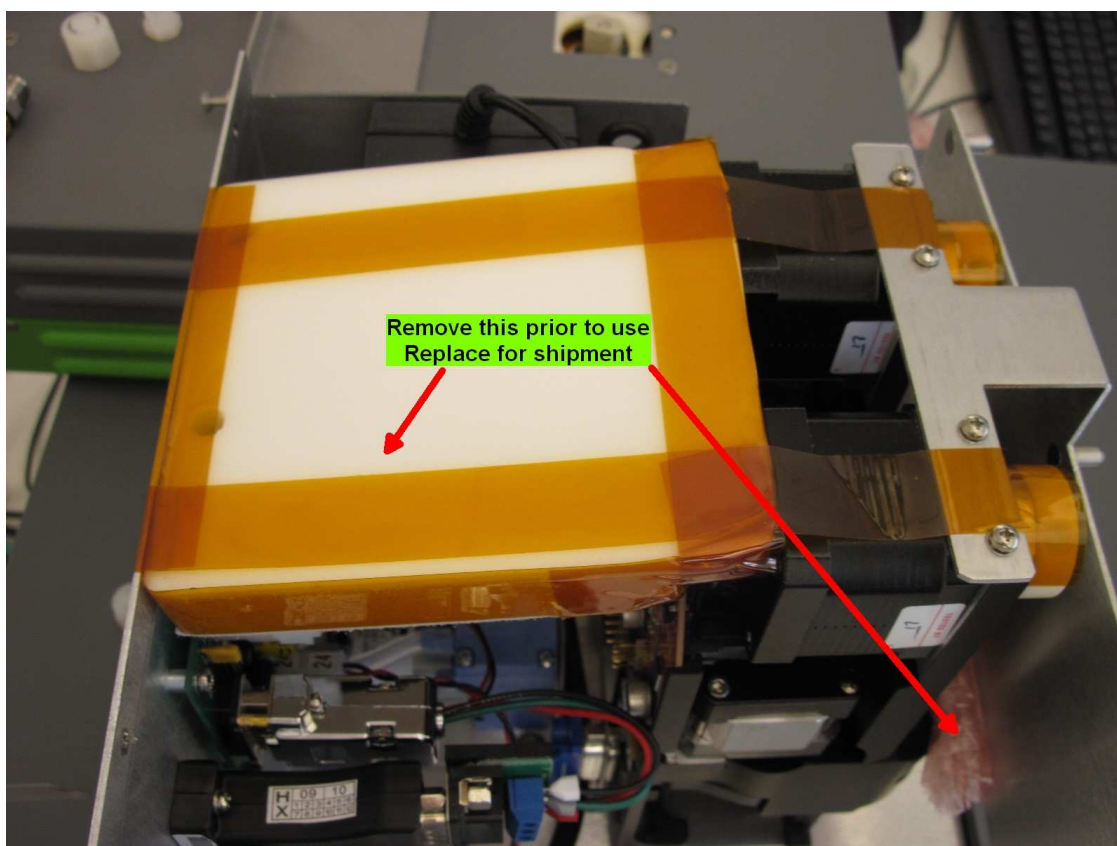
1. Remove contents from the shipping box.
2. Place main unit on top of Picarro analyzer DAS to the left of the vaporizer, front edge even with the front of the vaporizer as shown.

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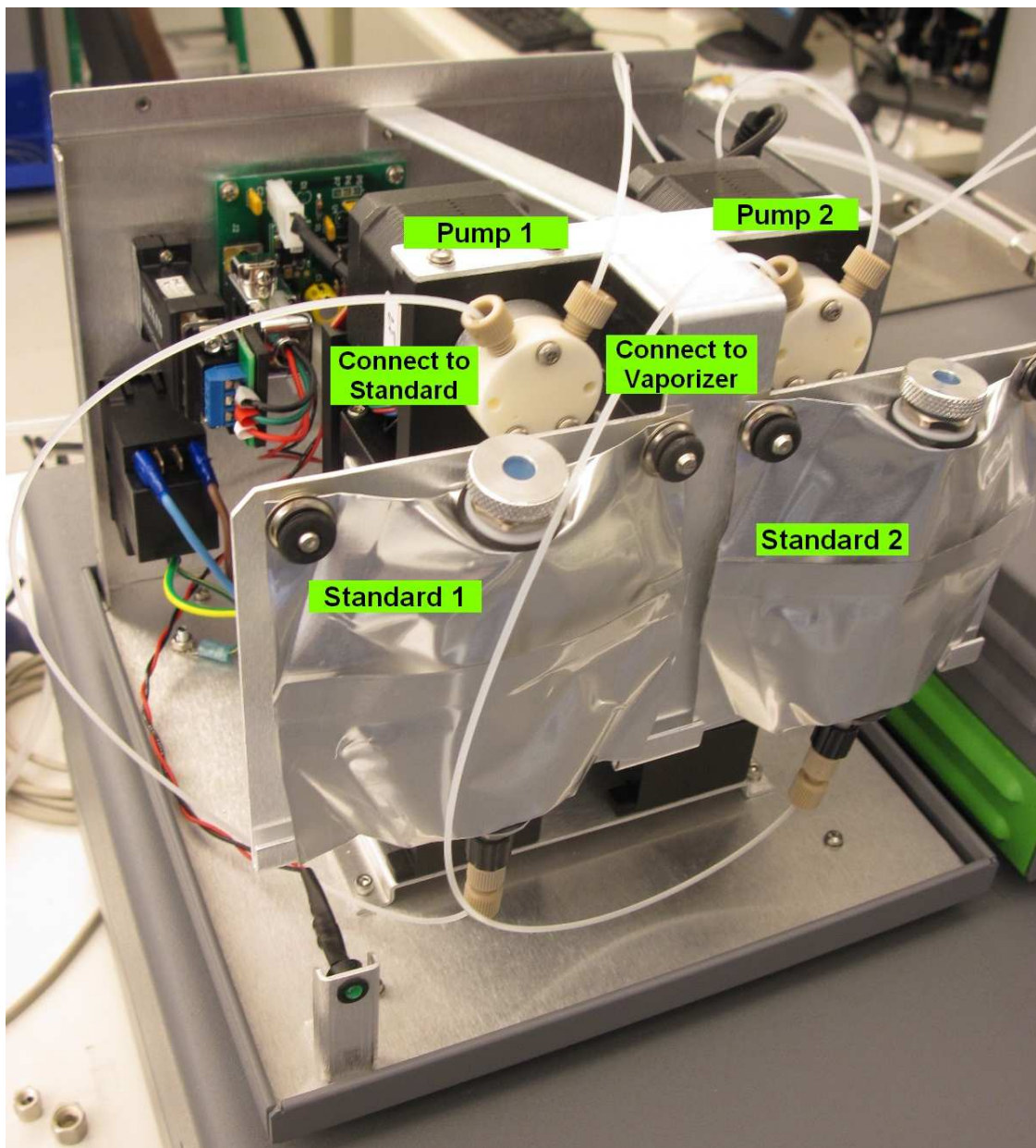
3. Plug in the power cord to the back of the SDM but do not turn it on. Connect the serial cable to the SDM and the Picarro analyzer CPVU Com 1 (directly above the monitor cable).
4. Remove cover of the SDM by loosening the 4 screws in the back and sides of the unit. No tools are required for the screws which are designed to remain attached to the cover after loosening. Remove the foam and bubble wrap pieces in the SDM; these are for protection during shipment. Remove Kapton tape on ceramic valves of the pump.

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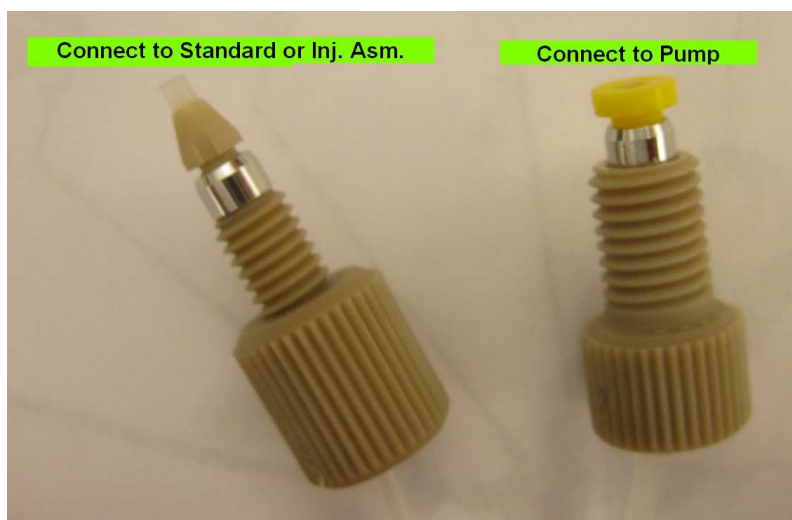
5. Connect liquid tubing to the left port on both pumps. The  $\frac{1}{4}$ -28 nut with yellow ferrule attaches to the left port. Tighten snugly using your fingers. Tools are not required.

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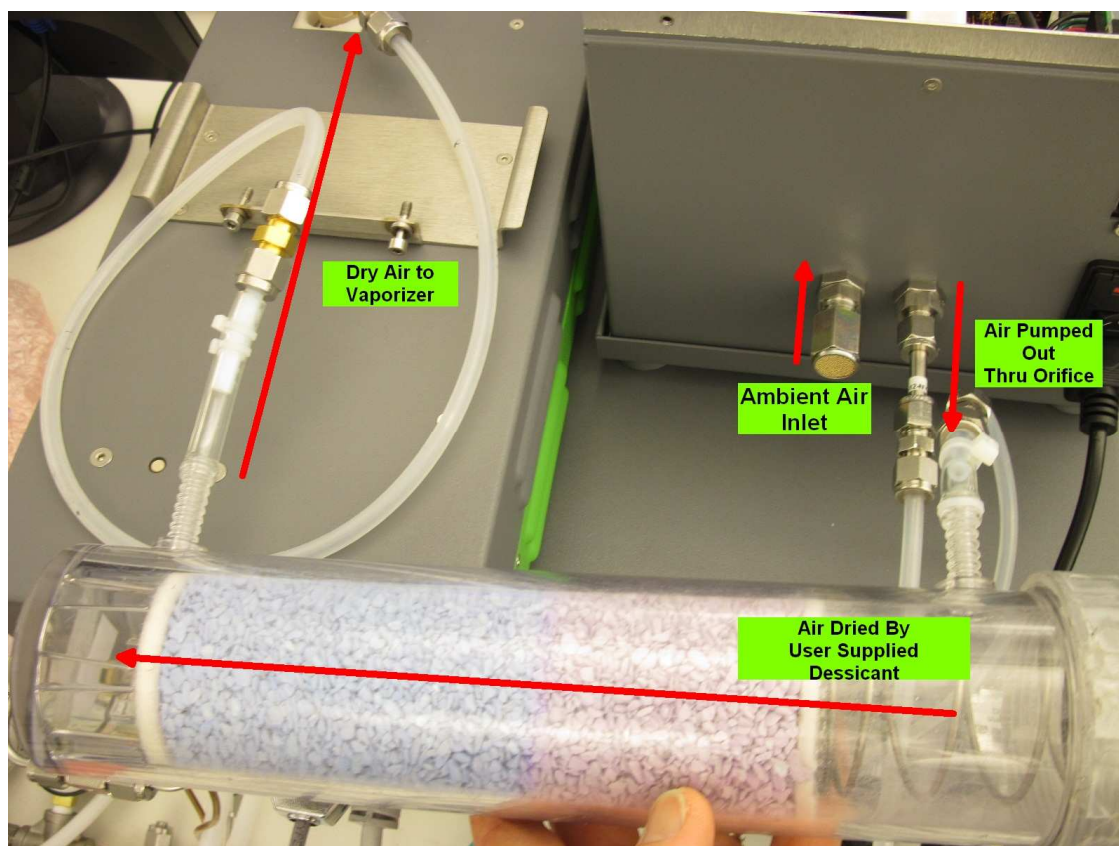
6. Connect liquid tubing to the right port on both pumps. Use the tubing segment with yellow and beige connectors. The  $\frac{1}{4}$ -28 nut with yellow ferrule attaches to the right port. Tighten snugly using your fingers. Tools are not required.

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7. Fill the liquid bags with standards water according to the section “Standards Choice and Bag Filling” of this manual. Label each bag appropriately.
8. Lay the liquid bag down on table top. Hold with black stopper pointing up, remove stopper, gently press until bead of water comes out of connector and connect to the appropriate pump with the beige ferrule end of the tubing. The pump on left is #1, right is #2. The left port of each pump connects to the bag.
9. Hang the bag by sliding the corner holes on the bag over the posts on the SDM. Secure the bags by pressing the black grommets over the posts. Pull on bag edges and tap with finger around the bottom outlet to force any trapped air bubbles up.
10. Connect air output of SDM to supplied ¼” tubing segment with flow restrictor orifice (orifice side closest to SDM) then to desiccant canister using ¼” tubing and Swagelok compression style fittings. Connect output of desiccant canister to segment of ¼” tubing long enough to reach injector port of vaporizer. The end of the segment should be reduced to 1/8” male Swagelok compression style fitting.

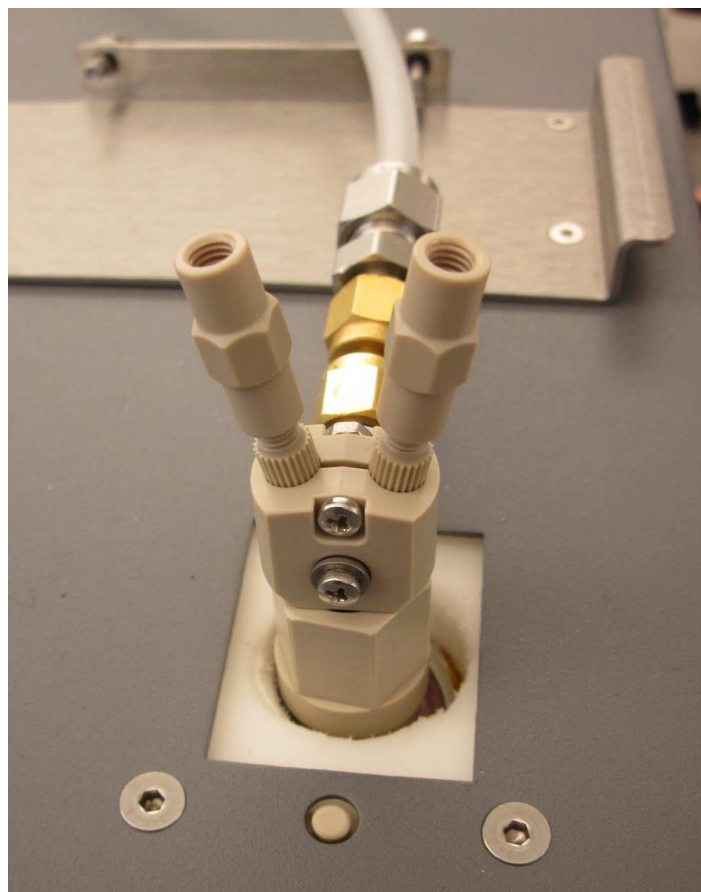
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11. Remove the original injector port cap and septum from the vaporizer (Caution HOT). Screw on lower portion of SDM injector port assembly.
12. Press and twist in upper portion of SDM injector port assembly into the lower portion. It will gently snap into place. It is designed to rotate and should be rotated such that the desiccant output tubing puts minimal strain on it.



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13. Do not connect the remaining hardware.
14. Turn on the power switch in the back of the SDM. The green LED in the front will light.
15. To install software create the directories “C:\SyringePump” and “C:\SyringePumpData” and “C:\SyringePumpData\ProcessedData” on the Picarro CPVU use Windows Explorer.
16. Insert supplied USB stick into CPVU and copy file to “C:\SyringePump”. Double click on the file to unzip it.
17. Move the file  
“CoordinatorSyringePumpRS485PumpSpeed\_2Ambient\_v11.ini” to  
“C:\Picarro\Host”.
18. Move the shortcut files “SyringePump Priming”, “SyringePump Sequencer”, “SyringePump Coordinator”, and “SyringePump Data Processor” to the desktop.

## Theory of Operation

The SDM delivers a liquid water standard at an extremely slow flow rate (0 to 4.8 microliters per minute) through a needle. For reference, a standard drop of water is about 50 microliters.

The needle tip is inside the vaporizer and 2mm away from a tube carrying dry air. The flow rate of air is approximately 300 sccm. The combination of high temperature, fast dry gas flow, and slow liquid flow allow the delivered standard to evaporate fully as it exits the needle.

Because of the low flow rates all the fluidics connections used are very fine (diameters of approximately 100 micrometers). These can clog easily if exposed to dust or mineral rich water.

## Operation

1. Point the beige connector ends of the liquid tubing (from right port of pump) into a cup—they will push out a stream of water during the pump priming operation. Do not connect to injector—a large amount of water will go into the vaporizer and saturate it!!!
2. On the computer desktop click on the icon labeled “syringe pump priming”. Verify both syringe pump 1 and 2 are selected. Click ok.
3. Each syringe pump will fill and dispense three times. The first time this operation is performed with dry tubing only one to two dispenses may be visible. The dispensed water will be visible as a narrow stream lasting a few seconds. The software window will automatically close within a minute of the last dispense. When tubing lines are used for the first time or have been dried out then priming should be repeated until each dispense is strong clean jet of water free of bubbles.
4. If no water or only a weak trickle is observed by the third the lines are probably clogged or leaking severely. See troubleshooting for recommendations.
5. Inspect all three connections of each pump for any leaks. Using a small light check both the outside of the connectors as well as the inside for signs of water.
6. Now connect the tubing from each pump to the hex nut on the upper portion of the SDM injector assembly (the two connection points are symmetrically identical). Hold the hex portion of the assembly while tightening in the tubing. It is recommended to mark each hex portion with the number of the connected pump to simplify maintenance and troubleshooting.

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7. Click on the “SyringePump Sequencer” icon on the desktop to create or load the desired sequence of operation. Recommended parameters and details of operation are described in the section “Sequencer Software Operation and Recommendations” of this manual. Click “Apply”.
8. On the computer desktop click on the icon labeled “SyringePump Coordinator”.
9. After 1-2 hours of operation inspect all four connections of each pump for any leaks. Using a small light check both the outside of the connectors as well as the inside for signs of water. Very small leaks take a long time to release enough water to be visible.
10. The cover of the SDM should be put back on. Be sure the front and sides of the cover is inside of the tray edge. Be sure the back of the cover is outside of the tray back wall.
11. The sequence applied in the sequencer software will start and loop indefinitely until the coordinator window is closed. To change the sequence first close the coordinator, open “SyringePump Sequencer”, apply the desired sequence, and finally restart the coordinator.
12. By default the data output from the coordinator will be saved in “C:\SyringePumpData”. The default is to create a single file which can become extremely long for multiple day experiments. How to change defaults and further information are described in the section “Coordinator Software Operation” of this manual.

## Troubleshooting

Symptom: No communication between software and SDM

Recommendations: Verify power is on (LED in front). Verify COM 1 of CPVU is connected to SDM. Remove cover and check internal connections.

Symptom: Water vapor concentration is unexpectedly very low (100-300ppmv) during standards measurement

Recommendations: Check water delivery pathway. Remove cover and disconnect tubing from problematic standard. Run priming software for that pump only (the movement of the syringe pump will be visible by looking from the side of the SDM, there will be 3 cycles). Assuming the pump is working and there is no water then one of the lines is clogged. If there is water then the

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problem is with the needle. Remove the needle from the injector assembly, connect to tubing and check again.

Symptom: Water vapor concentration is unexpectedly high (20000+ ppmv) during standards measurement

Recommendations: Verify the air pump is running. Verify there is a flow of about 250 sccm coming out of the vacuum port of the vaporizer. If both are operating correctly then excess water previously entered the vaporizer. Please contact Picarro for instructions on how to dry out the vaporizer. If the pump is running and air flow is <250 sccm then the line is leaking before the vaporizer or is blocked. Blockage most likely will occur at the orifice at the pump outlet. The pump inlet is equipped with a filter inlet to prevent particles from entering.

Symptom: standard deviation of standard isotope ratio exceeds typical values/water concentration during standards delivery shows large oscillations (250+ppmv) or erratic behavior

Recommendations: there is a partial obstruction or leak in the water pathway. Check all connections for leaks. Carefully look at tubing for bubbles. Disconnect tubing from needle and run priming—stream should be a strong narrow jet of water free of bubbles. Remove needle from injector assembly and check with microscope/10x magnifying glass for encrustations/blockage if priming jet is strong. If priming jet is weak disconnect and replace tubing. If priming jet contains bubbles and tubing connections are good then “palpate the bag”- i.e. pull at the edges, tap, etc to detach air bubbles from outlet area so they can rise to the top. Good bag filling and line connection procedure minimizes this problem.

Symptom: Data displayed in coordinator or processed data files does not match column heading.

Recommendations: The analyzer has an earlier configuration file with a different order of columns. Please contact Picarro for assistance.

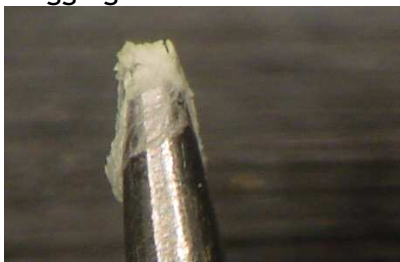
## ***General Note***

Ensuring a consistent vapor concentration of the standard requires a consistent liquid flow. The presence of air bubbles will not only cause sudden drops in the vapor concentration when an air bubble reaches the needle tip but it will also cause oscillations in liquid flow. This is because the air bubble is

compressible and the liquid is pumped by mechanical displacement (syringe) and instead of water flowing the air bubble will undergo compression. This is manifested in strong oscillations in measured vapor concentration followed by a sudden drop when the air bubble finally exits the needle.

## Standards Choice and Bag Filling

The two water standards chosen should be at the upper and lower value of the expected isotopic ratio range of the ambient vapor to be measured. The water standards must be deionized and free of other impurities. The water should be passed through a particulate filter (2 micron size) to remove any insoluble material. The water standard will completely evaporate at the needle tip and any soluble material will be left behind—this will first create a sponge-like reservoir that results in unstable evaporation rates followed by clogging the needle as shown here:



The needles should be inspected periodically under a microscope or magnifying glass to verify no build up of material has occurred.

### ***Bag Filling***

First seal the bottom connector on the bag with the supplied black plugs. Unscrew the metal cap on top and remove the blue septa. Fill the bag until nearly full. Pull at the bag edges to release trapped air. Fill until the water reaches the top of the metal fitting. Press in the septa (some water should leak out). Screw on metal cap. Shake bag—if there is a significant noise then there is a lot of entrapped air—pull bag edges and fill again to remove as much air as possible.

Air can become trapped in the lower portion of the bag and be pulled into the pump lines—this will affect standards delivery. Always fill the bag as much as possible initially and pull on the bag edges before running the SDM.

The bag has a Luer-lock connector with a 10-32 female thread adapter attached to it. The bag holds approximately 45mL of water. Restek Ice Blue silicone septa, 9mm in diameter, are provided with the bags.

## Sequencer Software Operation and Recommendations

The sequencer software is used to frequency, duration and concentration of standards delivery as well as ambient vapor measurement. The software supports two concentrations per standard as well as solenoid vapor valve switching (with appropriate valves) and rotary valve switching (for analyzers equipped with two serial ports and appropriate valves).

Pump 1 and Pump 2 are on the left and right side of the SDM when looking from the front.

The concentration of vapor will be determined by the user programmed liquid flow rate. A rate of 0.02 microliters/second corresponds to approximately 6000ppmv. The vapor concentration is a linear function of the liquid flow rate. Rates higher than 0.08 microliters/second (24000ppmv) are prevented by the software in order to prevent accidental saturation of the analyzer.

The precision of the isotopic ratio measurement is specified for a vapor concentration of 6000 to 20000ppmv. The precision will suffer significantly below 6000ppmv, increasing the measurement duration will compensate to some degree. The dry air source, such as Drierite® condition air with a 200-300ppmv water concentration, can contribute significantly to the measured isotope rate when operating at standard vapor concentrations below 6000ppmv.

For each step in the sequence only one pump or vapor state is allowed. If enabled the rotary valve position is always active.

Vapor 1 state allows the analyzer to pull through the connector labeled “Sample 2” on the back of the vaporizer. No power is supplied to any solenoid valve in this state. For analyzers with an additional valve connected to the “Sample 2” gas will be drawn thru the NO port of the valve.

Vapor 2 state applies power to the solenoid valve connected to the wire pair labeled V4. For analyzers with an additional valve connected to the “Sample 2” and powered by V4 gas will be drawn thru the NC port of that valve.

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The rotary valve position is controlled independently of the liquid pumps and solenoid valves. The default value is “0” which is generally not a valid position. This prevents the rotary valve from inadvertently switching to a new position.

The common line of the rotary valve can be connected to “Sample 2” on the back of the vaporizer. Whenever “Vapor 1” is selected in the software sample will be drawn thru the common line and whichever rotary valve position is selected.

## Recommended Operational Sequence

In order minimize isotopic memory effects it is recommended to first run at the higher concentration of a particular water standard, then the lower. The Picarro analyzer is temperature stabilized and has extremely low drift so calibrations should be run typically every 6-12 hours.

## Coordinator Software Operation

Double click on the icon “SyringePump Coordinator” to start the SDM module. The software prompt will ask for the names of the two liquid standards. After the names are entered or default is accepted the coordinator software will start to run the active sequence (previously applied using the syringe pump sequencer software). The sequence will run in a loop until cancelled by the user.

The upper table in the coordinator window will display measured values from the analyzer.

The coordinator will automatically create a \*.csv file containing the measured values, the file will be saved in the directory C:\SyringePumpData\ under the date and time when the coordinator was started.

To change the file name or create a new file select the “new output file” button and enter a new name. By default the data will be stored in a single \*.csv file- see the Appendix for information on how to change this or the default directory for file saving.

The \*.csv file can be read using Notepad++ which is provided on the Picarro CPVU.

To stop the coordinator click on the “x” in the upper right of the coordinator window.

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In the lower portion of the window the coordinator software will display a log of operational steps and commands. The current step will be at the very bottom of the log. To view the overall sequence of operation open the syringe pump sequencer software. If the sequence has been modified the currently used sequence can be reloaded from the file “active.seq”.

## Automatic Vaporizer Preconditioning by SDM

The SDM has certain preprogrammed operational steps which bring the vaporizer into isotopic equilibrium in a quick and consistent fashion to minimize time required for standards measurement and to simplify data analysis. These steps are in addition to the user entered sequence.

There are different situations when this occurs:

### Case A: Vapor to Standard

Step 1: vapor measurement

Step 2: water standard from pump 1 or 2

3.5 minutes before the end of step 1 the SDM delivers 6 microliters of water over 2.5 minutes followed by 1 minute at the user specified rate. The exit valve of the vaporizer vents this standard vapor out to the atmosphere. Once Step 2 starts the valve connecting vaporizer to analyzer is opened allowing a nearly seamless transition between measuring the ambient vapor and standard vapor.

In the event the vapor measurement period is <3.5 minutes the vapor measurement period is automatically extended, i.e. if the sequence call for a 1 minute vapor measurement then measurement of a standard the actual vapor measurement period will be 4.5 minutes.

### Case B: Between Two Standards

Step 1: water standard from pump 1 or 2

Step 2: water standard from pump 2 or 1

After step 1 is complete the SDM delivers 6 microliters from the following standard over 2.5 minutes followed by 1 minute at the user specified rate. The standard vapor from this automatic step is sent into the analyzer for measurement. When using the analysis software the 3.5 minute automatic step is automatically skipped over by the analysis software.



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## Case C: Between Two Concentrations

Step 1: pump 1 conc. 1

Step 2: pump 1 conc. 2

Syringe pump is refilled at the start of step 2 and then starts to dispense at the user specified rate.

## Data Processing

The SDM includes data processing software for user convenience. To start the software double click on the icon “SyringePump Data Processor” on the desktop. Under the “File” menu select “Load File” and choose the file to be processed. Files are stored under C:\SyringePumpData by default.

Under the Liquid Data Analysis section select the start time for each pump and concentration value. The start time is a delay relative to the actual start of standard delivery; it prevents non-equilibrium values from being evaluated.

Typically a 5 minute delay is sufficient to allow the isotope ratio to reach full equilibrium. For higher concentrations a shorter time will be sufficient, for very low concentrations a longer time will be required. Examine the  $\delta D$  value vs. time for each pump and concentration to optimize this parameter further. The  $\delta D$  value takes longer to equilibrate than the  $\delta^{18}O$  value.

Select the end time of the evaluation period, it is relative to the end of actual liquid delivery. The default is 0.5 minutes which works well generally.

The maximum number of plots is limited to 4. These are useful for quick visual assessment of the data.

The process button in the upper right will process the data for liquid data analysis only. The data will automatically have a 30 second moving average applied to it.

Under the Vapor Data Analysis section select the start and end times in the same fashion as for the liquid data analysis. The start time delay required for vapor may be significantly larger than for liquid data. This is due to concentration effects as well due to the absence of preconditioning steps for vapor analysis. The averaging window size applies a moving average to the vapor data.

The process button in the middle right will process the data for vapor data analysis only.

To process the data for both liquid and vapor press the process button at the very bottom of the window.

Once data is processed one or more files are generated. They are all stored in C:\SyringePumpData\ProcessedData by default. The processed data is saved in

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\*.csv format and can be viewed by double clicking on the blue hyperlinks shown in the Data Processor window. Each hyperlink has text above it describing the contents of the processed file.

If the reported standard deviations (SD) appear high for any particular standard delivery note the time and view the H<sub>2</sub>O concentration for that period. There may be a specific upset such as an air bubble that can be excluded from the data processing by changes in the start/end time. If there is significant oscillation in the water concentration (amplitude >250ppmv) that is an indication of uneven water delivery which is generally caused by loose connections, partially clogged lines/needles, or an air bubble undergoing compression/relaxation as it is pumped through the line.

## Field Deployment Tips

1. Run the SDM in the lab to become fully familiar with its setup and operation before undertaking a field deployment.
2. Plan enough time for setup. Although the SDM sets up quickly it requires 2-4 hours of run time to fully verify performance so it can be left to run unattended. Plan on at least a half day, overnight is ideal. Ideally the SDM can be set up first and allowed to run while other equipment is being set up.
3. Be prepared. The bags should be filled in the laboratory. Extra lines and needles should be packed as well.
4. Don't take shortcuts. The procedures in the manual are based on personal, sometimes painful, experiences gained by engineers and scientists during the development of the SDM. The effort of a proper setup will be rewarded by weeks of smooth unattended operation and good data.

## Appendix

### *Configuring coordinator column output*

Open picarrocrds.ini file  
C:\picarro\host\gui  
Use Notepad++ text editor (right click on file)

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Under section marked [GUI] at approximately line 67 for most systems the default will be:

```
RS232_COLUMNS=1,4,13,15,18, 7
```

The first five values after the = sign must be “1,4,13,15,18” so that the coordinator displays the correct values/columns. If you require additional values to be sent out by RS232 add them after the required values using comments to separate them.

## ***Setting Coordinator File Output Size***

By default there is no upper limit on the size of the coordinator file output. When the SDM is run for prolonged periods the size of the generated \*.csv file will become very large and easily exceed 65536 records which is the upper limit of MS Excel. A new record is generated with each scan reported by the analyzer, the scan time ranges from 5-10 seconds depend upon analyzer configuration.

To limit the size of the output file

Open PicarroSyringePumpCoordinatorV11.ini file

C:\picarro\host\gui

Use Notepad++ text editor (right click on file)

Under section marked [FILES] at line 8 the default will be:

```
max_num_lines=
```

If it is left blank after the equals sign then there is no size limit

To specify a limit on the number of records enter an integer value after the equals sign. For example

```
max_num_lines=2000
```

will limit the file to 2000 records, the coordinator software will automatically create a new file when the 2000 record limit is reached.

## ***Changing the location of the output file***

Open PicarroSyringePumpCoordinatorV11.ini file

C:\picarro\host\gui

Use Notepad++ text editor (right click on file)

Under section marked [FILES] at line 7 the default will be:

```
output="C:\SyringePumpData\"
```

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create a new location by changing the value in “ ”. for example  
output=”C:\UserData\SyringePumpData\”  
would save the outputs in a separate directory within the main user data files  
directory.

## ***Rotary Valve Use***

The COM port to control the rotary valve can be used by only one software application at a time. Depending upon the configuration of your analyzer additional steps may be required.

The default configuration of the L1115-i disables the rotary valve and valve sequencer. In this case the rotary valve control should be connected to COM 2 (the female DB-9 RS232 connector above COM 1) and the “Enable Rotary Valve Control” button selected in the syringe pump sequencer software.

If the rotary valve has been enabled (uncommon) with the valve sequencer software then it must be disabled. To do this Start by going to

C:\Picarro\XXDSYY\GUI

(where XXDSYY is the specific model and serial number of the analyzer)

Open the file “Valve\_Sequencer\_Control.ini” using Notepad. The contents of the file will look similar to following:

```
[VALVES]
VALVE_SEQUENCE_ENABLE=1
VALVE_SEQUENCE_NUMBER=3
MPV_ENABLE=1
MPV_PORT=2
MPV_LOW=-2
MPV_HIGH=8
```

Set MPV\_ENABLE=0. You will need to restart the analyzer software for the change to take effect.