STANDARD DELIVERY MODULE – CRDS SETUP A0101

User's Manual

* This manual incudes the complete information on how to set up and operate the entire SDM-CRDS Setup

PICARRO

Thank you for purchasing a Picarro product. Your Picarro Standard Delivery Module is a quality product that has been designed and manufactured to provide reliable performance.

This manual is an important part of your purchase as it will help familiarize you with the module and explain its features. Please read this manual thoroughly before using your Picarro Standard Delivery Module.

Please contact Picarro or your authorized Picarro distributor should you have questions regarding specific applications or if you require additional information.

Contact Technical Support:

Email: support@picarro.com
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(See “Technical Support” chapter for more information.)

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Picarro, Inc. has prepared this manual for use by its customers as a guide for the proper installation, operation and/or maintenance of the Picarro Standard Delivery Module.

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MUST READ BEFORE USING THE MANUAL

The Picarro SDM (Standard Delivery Module) automatically supplies two calibration standard solution samples into the vaporizer and analyzer system. This manual contains information needed to safely install, operate, maintain, and troubleshoot your SDM.

This manual assumes Basic Water Analyzer Setup is complete. Please refer to the separate User's Manual "**INSTALLATION L2140-, L2130-I or L2120-I Analyzer and Peripherals**".

This manual includes various graphic icons to represent important information in the text. See **Conventions** for definitions of graphic icons. For explanations of acronyms, see **Acronyms**.

CONVENTIONS

Throughout the manual you will see graphic icons representing important information in the text. The purpose of these icons is to provide a visual convention to alert you of a stop in the flow of the manual, where an important note or safety hazard alert is posted.

NOTE	CAUTION	WARNING	REMINDER
			

NOTE is an important procedure of which you should be aware before proceeding.

CAUTION alerts you of a potential danger to equipment or to the user.

WARNING indicates an imminent danger to the user.

REMINDER is a helpful hint to procedures listed in the text.

ACRONYMS

This manual includes various acronyms. For definitions, see below.

Acronym	Definition
AS	Autosampler
DIO	Digital Input and Output Between the Analyzer and the Autosampler. DIO Tells the Autosampler to prepare for an injection, and also to do an injection. Additionally, DIO is the place where the autosampler notifies that an injection has been made.
GUI	Graphical User Interface
ppm	parts per million
sccm	Standard Cubic Centimeters per Minute
SDM	Standards Delivery Module
WLM Purge Port	Wavelength Monitor Purge Port. The Port on the Analyzer the dry gas connects to. This helps improve the performance in humid environments.

SAFETY

General Safey

The Picarro analyzer complies with the following safety standards:

CE	**IEC EN61010-1:2001 (Safety) and EN61326-1:2006 (EMC)** requirements for electrical equipment for measurement, control and laboratory use.
FDA/CDRH	**21 CFR Parts 1040.10-11**

	WARNING: DO NOT OPERATE IN AN EXPLOSIVE ATMOSPHERE! DO NOT OPERATE IN THE PRESENCE OF FLAMMABLE GASSES OR FUMES.
	WARNING: THE INSTRUMENT IS NOT WATER PROOF, AND IT SHOULD BE KEPT PROTECTED FROM EXPOSURE TO ALL LIQUID WATER.
	CAUTION: The Picarro analyzer contains no user serviceable components except the particulate filter and the vacuum pump. Do not attempt repairs; instead, report all problems to Picarro Customer Service or your local distributor. Please contact Picarro if you have any questions regarding the safe operation of this equipment.
	CAUTION: The inlet gas connector on the back panel of the analyzer, and its immediate vicinity, runs hot during operation of the analyzer. Take care when connecting gas lines or working at the rear of the instrument to wear protective gloves or avoid contact with these surfaces.
	CAUTION: The analyzer contains HOT SURFACES and utilizes HIGH VOLTAGES inside the instrument. There are no user serviceable components except the filter within the analyzer and you should not open the analyzer except to replace the filter. Do not open any enclosures within the analyzer.
	CAUTION: The analyzer is heavy. To avoid injury, please use proper lifting procedures when moving or installing the equipment.

Laser Safety

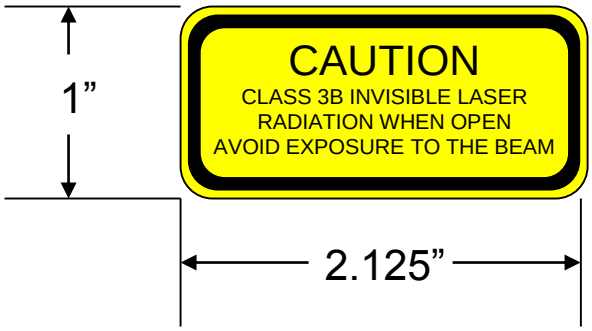
The outside of the Picarro analyzer is classified as a Class 1 Embedded Laser Product, while the inside of the Picarro analyzer is classified as a Class 3B Embedded Laser Product.

	CAUTION: CLASS 3B INVISIBLE LASER RADIATION WHEN OPEN. AVOID EXPOSURE TO THE BEAM.

The lasers inside of the analyzer emit a maximum of 50 mW of CW light in the near-infrared. There are no user serviceable components within the analyzer enclosures and so you should not open any of these enclosures within the analyzer. FAILURE TO FOLLOW THIS INSTRUCTION COULD RESULT IN EXPOSURE TO CLASS IIIB LASER RADIATION, which can permanently damage eyes and skin.

Safety Label

The following label is affixed to the inside of the analyzer.



INSTALLATION | STANDARD DELIVERY MODULE SETUP

USER SUPPLIED COMPONENTS:

Dry air supplies with a moisture level of <200ppmv, 350sccm, and 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.

INSTALLATION STEPS:

1 Gather the following parts before continuing on with the installation.

- SS-400-6 (1/4" Swagelok Union)
- SS-400-6-2 (1/4" to 1/8" Swagelok Reducing Union)
- SS-201-PC (1/8" Swagelok port connector)
- ¼" OD Bev-A-Line IV tubing.
-

2 Set up the analyzer with its external vacuum pump by referring to **Analyzer's Installation User's Manual.**

3 Place the vaporizer on top of the analyzer using 4 spacers of 0.5" (13 mm) thickness to set it to the appropriate height. The right edge of the vaporizer should be flushed with the right edge of the analyzer (when looking from front).

4 Leave the WLM purge port on the Analyzer open as it does not require dry gas. If dry gas is uses, a supply pressure of 2.5 psi +/- 0.5 psi is recommended.

5 DO NOT connect anything to the port labelled Purge on the back of the vaporizer.

6 The port labelled Sample 2 on the back of the vaporizer is where the ambient vapor is sampled from. Attach tubing with a Swagelok 1/8" connector to this port and put the open end at the desired sampling location.

7 If two position sampling is required then a 3 way solenoid valve should be attached to Sample 2. It is controlled in the same manner as the dual liquid/vapour mode described earlier in this manual. If multi position sampling is required then Sample 2 should be connected to a multi position rotary valve.

8 The port labelled Vacuum serves as an exhaust port for excess water vapor generated during SDM calibration runs. Attach tubing with a Swagelok 3/8" connector to this port and put the open end away from and downwind of the sampling location. The image below doesn't have a tube attached to the Vacuum port (see **Figure 1**).

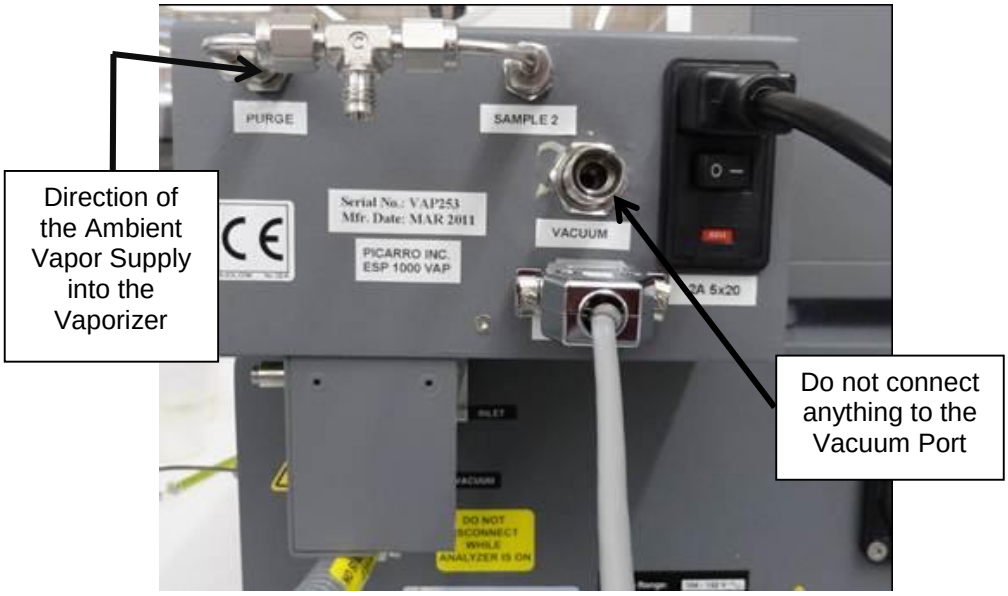


Figure 1 - Back of the Vaporizer.

9 Remove SDM contents from the shipping box.

10 Place the main SDM unit on the top of Picarro analyzer to the left of the vaporizer, front edge even with the front of the vaporizer.

11 Plug in the Power Cord to the back of the SDM: However, keep the power switch off.

12 Connect the serial cable to the SDM and the Picarro analyzer CPVU COM 1 Port (see **Figure 2**).

Figure 2 - Back of the SDM. Black Cord connects to Power. The Grey serial cable connects to the COM 1 Port of the Analyzer. The Semi transparent gas tube connects to the Drierite.

13 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.

14 Tighten up syringe barrels by removing support brace: This can be done by removing the 5 screws with a PH1 Screw driver in the positions shown below (**Figures 3 and 4**).

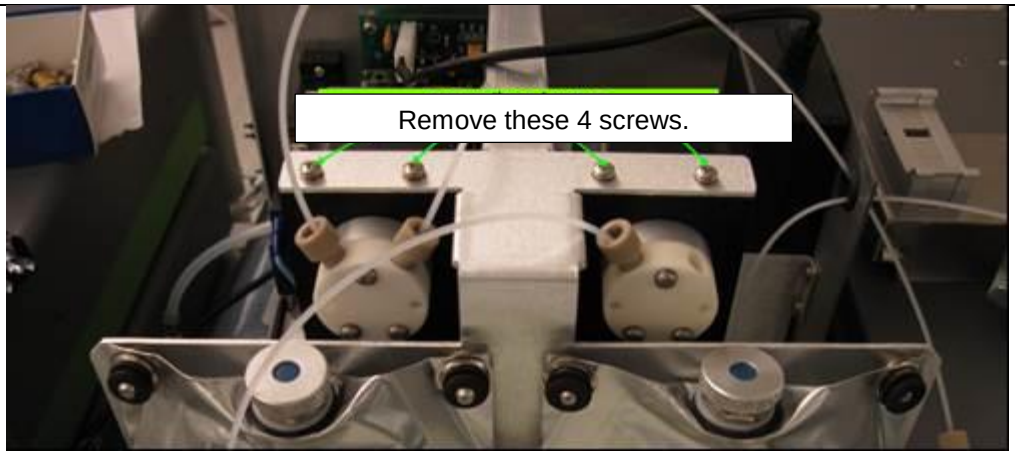


Figure 3 – SDM after the removal of its cover.

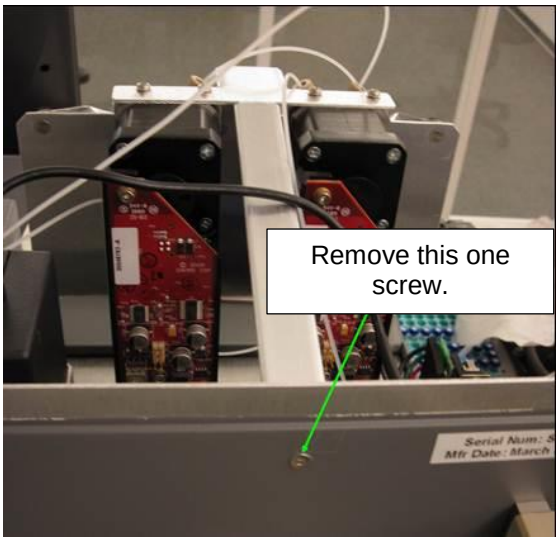


Figure 4 – SDM after the removal of its cover.

15 **Turn the knurled knobs on the ceramic pumps to right and hand tighten (Figure 5):** After tightening, make sure that the white syringe barrels are vertical when looking from front and side

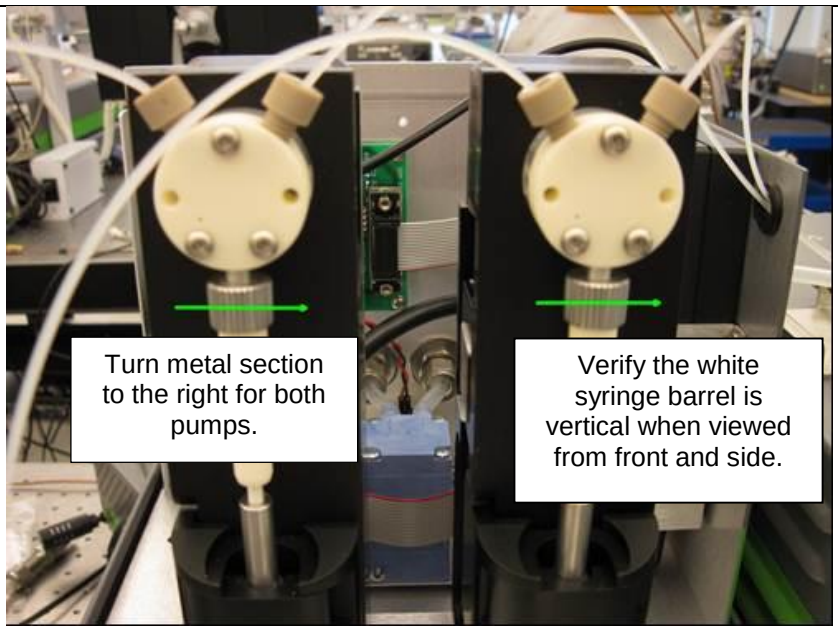


Figure 5 - Turn the knurled knobs on the ceramic pumps to right and hand tighten. Notice how the tubing from the pump going to the Vaporizer leave the SDM through a small round opening on the right wall of the SDM.

16 Connecting the tubing to the ceramic pumps (Figures 5 and 6): Although the two sets of liquid tubing appear identical they have <u>different inner diameters</u> and should be connected as labelled. The Bag to Pump line has a larger inner diameter.

- Connect liquid tubing labelled Bag to Pump to the **left** port on both pumps (seen from the front). The ¼-28 nut with yellow ferrule attaches to the left port. Tighten snugly using your fingers. Tools are not required.
- Connect liquid tubing labelled Pump to Needle to the **right** port on both pumps. Use the tubing segment with yellow and beige connectors. The ¼-28 nut with yellow ferrule attaches to the right port. Tighten snugly using your fingers. Tools are not required.

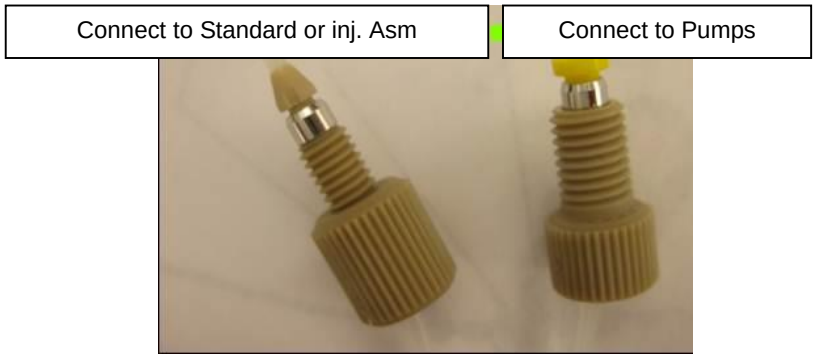


Figure 6 - Two different ends of a liquid tubing. The one on the left attaches to the liquid bag or the Injector Head Assembly. The one on right attaches to the ceramic pumps.

17 Fill the liquid bags with standard water. Label each bag appropriately. Each bag has a Luer-lock connector with a 10-32 female thread adapter attached to it, and it holds approximately 45mL of water. Restek Ice Blue silicone septa, 9mm in diameter, are provided with the bags. To fill the bag, see the images below.

Step 1: First seal the bottom connector on the bag with the supplied black plugs.

Step 2: Unscrew the metal cap on top and remove the blue septum.

Step 3: Fill the bag until nearly full. Fill until the water reaches the top of the metal fitting.

Step 4: Pull at the bag edges and/or tap the bag on the side to release trapped air.

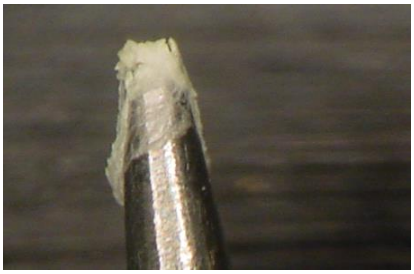
Step 5: Press the bag gently upward to form a water layer on the top. Afterwards, press in the blue septum (some water should leak out), and screw on metal cap.

Step 6 - 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.

Standards: 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:



Clogged Needle of the SDM

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

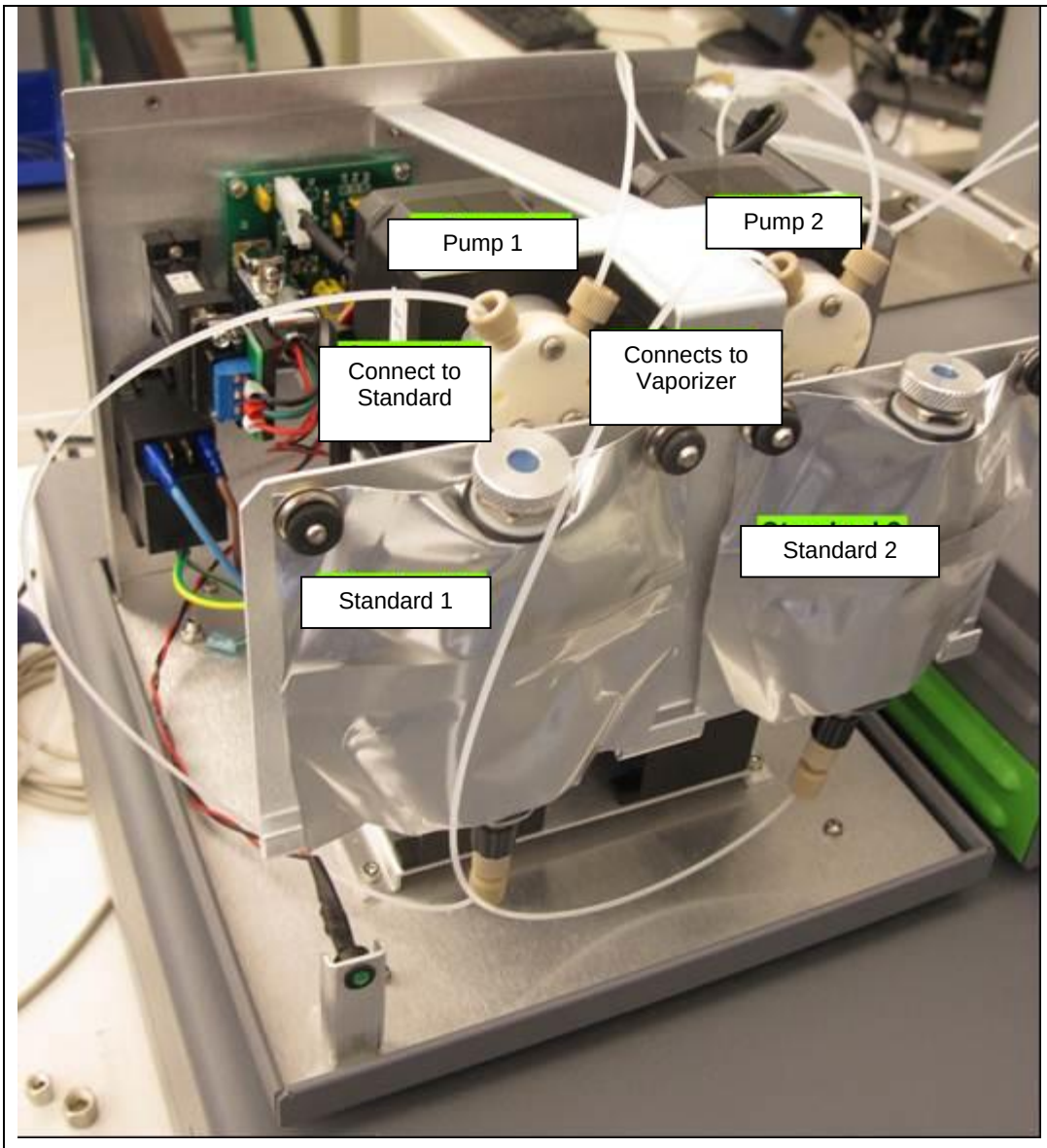


Figure 7 - Overall internal connections for the SDM.

18 Connect the remaining end of the tubing to the Standard Liquid Bags (**Figure 7**)**:** 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.

19 Secure the Standard Liquid Bags (**Figure 7**)**:** Hang the bags by sliding the corner holes on each 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.

20 Remove the Injector Port Cap from the Vaporizer: Remove the original Injector Port Cap (top side of the Vaporizer) and Septum from the vaporizer (Caution HOT). Screw on the lower portion of SDM injector port assembly.

21 Press and twist in upper portion of SDM injector port assembly into the lower portion: (See below for directions on how to make an Injector Port Assembly). 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.

22 Connect air output of SDM to supplied ¼" tubing segment (**Figure 8**) with flow restrictor orifice (orifice side closest to SDM) then to desiccant canister using ¼" tubing and Swagelok compression style fittings.

23 Connect output of desiccant canister to segment of ¼" tubing (**Figure 8**) long enough to reach injector port of vaporizer. The end of the segment should be reduced to 1/8" male Swagelok compression style fitting.

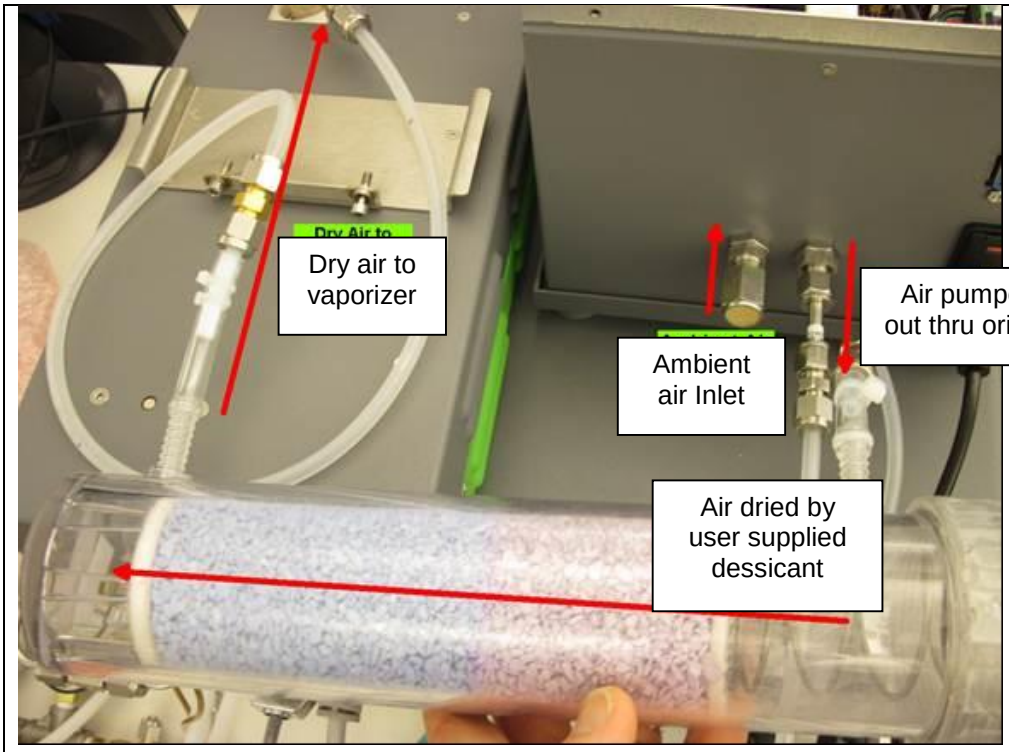


Figure 8 - SDM, User Supplied Desiccant, and the Vaporizer Setup.

24 Connect the Vaporizer to the Analyzer with a grey cable and 1/16" tube (see **Vaporizer to the Water Analyzer**)

25 Connect the black power cable to the vaporizer: Keep the power switch off.

26 Carefully slide the complete system into position (**Figure 9**): Small movements of the components relative to one another is OK, as the units are well locked. However, do not overly force the system: check for obstacles if the unit does not slide easily.

Figure 9 – After the Injector Head Assembly has been inserted into the Vaporizer. To the left is the SDM. To the right is the Vaporizer. At the bottom of the Vaporizer and the SDM is the Analyzer.

27 Power up the system: plug all the power cables (including the one for the monitor) into an appropriate power supply. Switch ON the components in the following order:

1. The external vacuum pump
2. Everything else.

ASSEMBLING THE INJECTOR HEAD FOR THE SDM PORT

1. Make a needle assembly by first hand-tighten a union on to a Needle, and then sliding a spacer and several O-rings onto the needle. Repeat the process to make two needle assemblies.

Figure 10: From left to right: 3 Orings, 1 spacer, 1 needle, and 1 needle union. Screw on the union on the right side of the needle as shown above. Slide in the spacer and three O-rings on the left side of the needle as shown above. Make two needle assemblies.

2. Thread the Needles through the holes in the body of the Injector Port Assembly. Wiggle a little to get the needle in the right position.

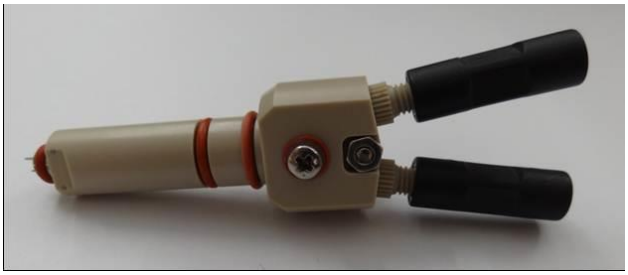


Figure 11: Slide two needles into each holes of the body of the Injector Head Assembly.

3. Use a Spacer Tool to make the depth of the needle coming out of the body of the Injector Port Assembly the same. Push the needle area into the cut out of the spacer tool. Put the entire system on the ground, spacer tool at the bottom, and push down to make sure that the needles are of the same depth. Tighten the screw on the injector head assembly on top of the gas inlet. If the distance is far, adjust the spacing with additional O-rings.

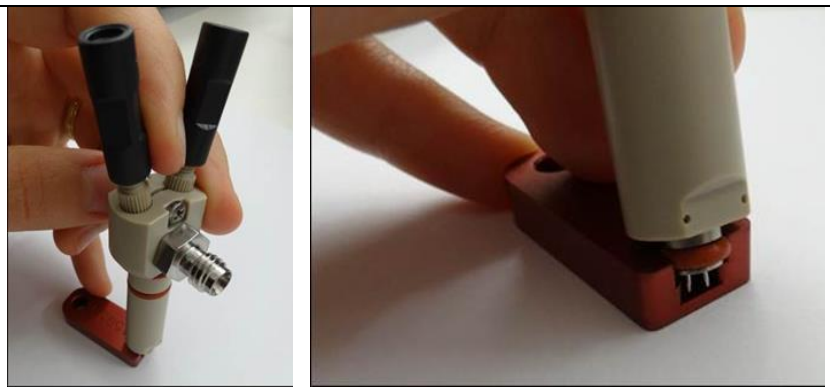
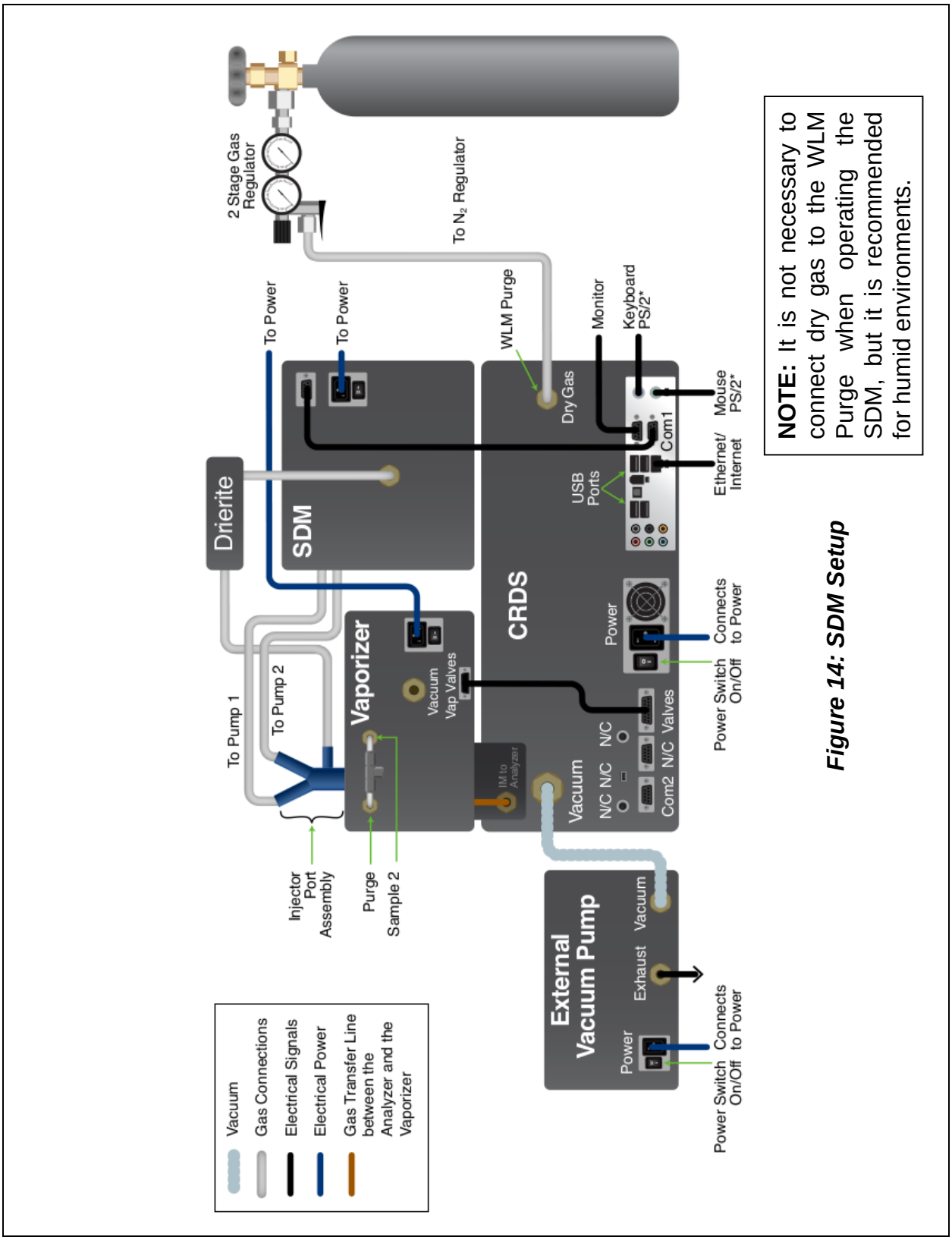


Figure 12: Use the red spacer tool to adjust the depth of the needle coming out of the injector head.

4. Attach the adaptor and the elbow tube to the gas inlet of the Injector Port Assembly. First use hand to tighten the screw. Finalize with a wrench.
5. Place the finalized injector head assembly into the opening on top of the vaporizer.

Figure 13: SDM Injector Head Assembly after being connected to the Vaporizer. Notice how the elbow tube and the adaptor is attached to the gas inlet of the injector port assembly.



NOTE: It is not necessary to connect dry gas to the WLM Purge when operating the SDM, but it is recommended for humid environments.

Figure 14: SDM Setup

BASIC OPERATION OF THE SDM

The Standard Delivery Module (SDM) delivers 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 held inside the vaporizer and 2 mm away from a tube carrying dry air. The flow rate of air is approximately 300 sccm (standard cubic centimeter per minute). 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 of the fluid connections are very fine (diameters of approximately 100 micrometers). These can clog easily if exposed to dust or mineral-rich water.

OPERATION

1) Before continuing, review the important safety notes in **Safety.**
2) Make sure the hardware setup is complete and the system turned on in the correct sequence.
3) Once turned on, the main CRDS Data Viewer of the analyzer will open up automatically on the desktop screen. To understand all the functions of the main GUI, see **CRDS Data Viewer in the OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual**. A sequence of start-up messages will also appear in the Status Log Message window of the main GUI. For definition, see **Common Status Log Messages in the OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual**.
4) **Make sure the temperature of the High Precision Vaporizer has stabilized to 140°C.** Use the up/down buttons on the vaporizer to adjust the set point and allow it to stabilize before proceeding. High temperature is required for proper vaporization since the SDM is not operating in vacuum.
5) Prime the SDM System (see **steps a – e** below):
    a. 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.

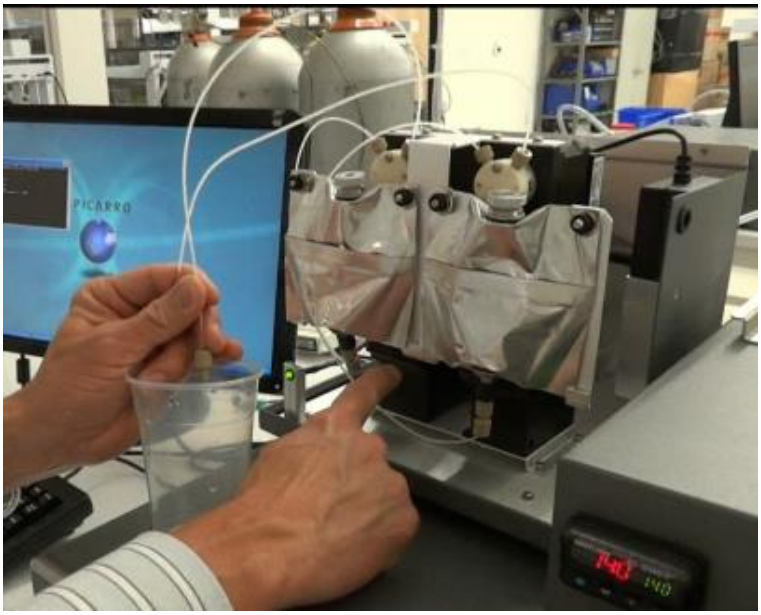


Figure 15: Point the beige connector ends of the liquid tubing into a cup.

	Do not connect the beige connector to the vaporizer—a large amount of water will go into the vaporizer and saturate it!!!

b. On the computer desktop, click on the icon labeled SDM Priming. Verify that both syringe pump 1 and 2 are selected in the Syringe Pump Priming window. Click OK.

c. Each syringe pump will fill and dispense multiple 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. If tubing lines are used for the first time or have been dried out, the priming should be repeated until each dispense is a strong clean jet of water that is free of bubbles.

	If no water or only a weak trickle is observed by the third dispense, the lines are probably clogged or leaking severely. See troubleshooting in this manual for recommendations.

d. Inspect all three connections of each pump for any leaks. Using a small light source, check both the outside of the connectors as well as the inside for signs of water.

e. 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.

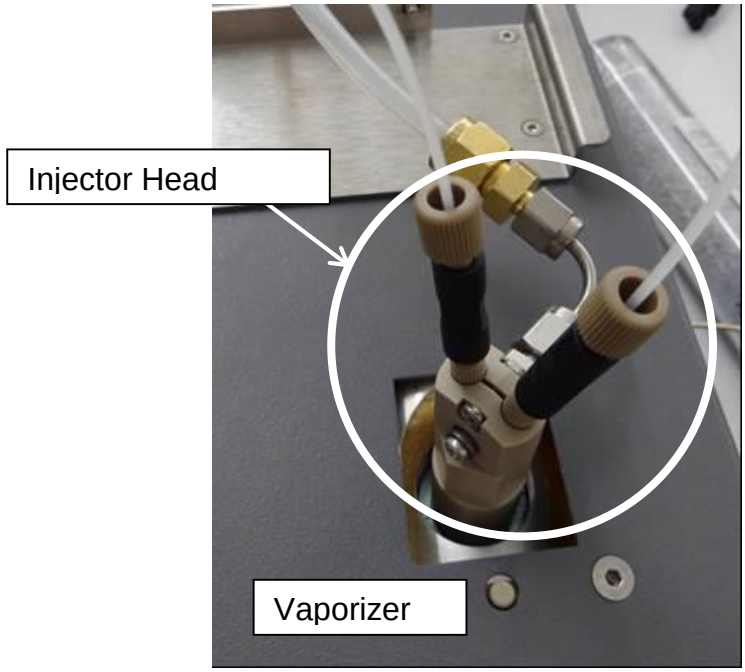


Figure 16: SDM Injector Head Assembly connected to the Vaporizer.

6) Use the Syringe Pumps Sequencer Software to set the frequency, duration, and concentration of standard delivery as well as ambient vapor measurement. Click on the SDM Pump Sequencer icon on the desktop to create or load the desired sequence of operation (see Figure 17). The software supports three concentrations (or flowrates) per standard as well as solenoid vapor switching valve (with appropriate valves) and rotary valve switching (for analyzers equipped with two serial ports and appropriate valves.). By specifying Vapor 1 State, Vapor 2 State, or the Rotary Valve Position on the Sequencer Software window, it is possible to sample air from multiple locations for one experiment sequence. Recommended parameters and details of operation are described below. Pump 1 and Pump 2 are on the left and right side of the SDM when looking from the front. Click Apply after entering the parameters.

	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.

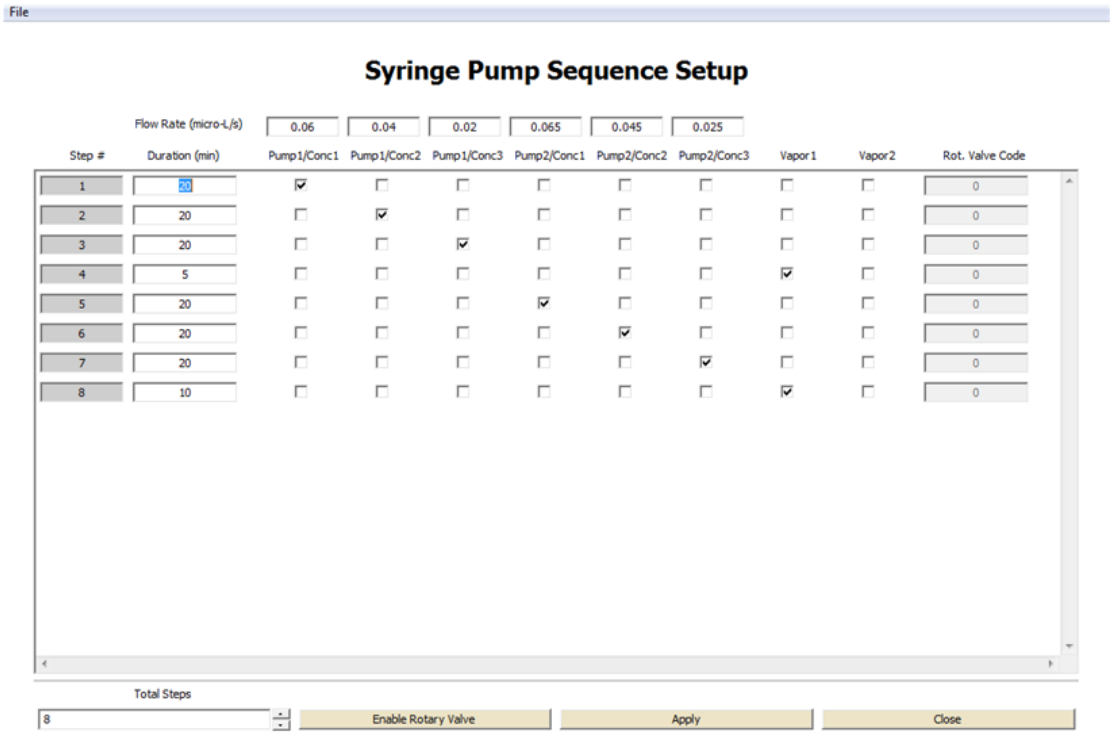


Figure 17: 'Syringe Pump Sequence Setup' window

The Concentration of Vapor:	This 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 6000 ppmv. 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 air through the connector labelled 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 (carrying V4 gas) also connected to the Sample 2, air will be drawn through the NO port of the valve.
Vapor 2 State:	In this state, power is applied to the solenoid valve connected to the wire pair labelled V4. For analyzers with an additional valve (carrying V4 gas) also connected to the Sample 2, V4 gas will be drawn through the NC port of that valve.
Rotary Valve Position:	This 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 through the common line and whichever rotary valve position is selected.

8) Double click on the Coordinator Launcher icon in the analyzer's desktop. The coordinator software allows the analyzer to take measurements from multiple samples,

and is used to control the sample source and match the corresponding real time read out with the sample source. To learn more about the coordinator software (running the software, loading sample description, functions of the coordinator window), see **Coordinator Software in the OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual.** Choose and launch an appropriate coordinator mode from the choices in the drop down menu of the coordinator launcher window. The User Editable Parameters window will pop up. Enter the names of the liquid standards on the window, and the click ok to continue. The SDM Setup can operate in one coordinator mode (see Figure 18).

SDM: Used for measurement of ambient vapor coupled with automated injection of liquid calibration standards. Requires A0211 high precision vaporizer and A0101 Standards Delivery Module. Alternates between analyzing ambient vapor from multiple points and a continuous stream of vaporized standard. The alternation is based on user defined sequence. The calibration measurement takes approximately 20 minutes per concentration/standard. Before operating in SDM mode, set the vaporizer temperature to 140 C°.

9) To learn about the other coordinator modes supported by the Picarro water analyzer (but in different setups), see **Coordinator Software in the OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual**.

10) Once launched, the coordinator will automatically start collecting data.

Figure 18: Coordinator Launcher Window

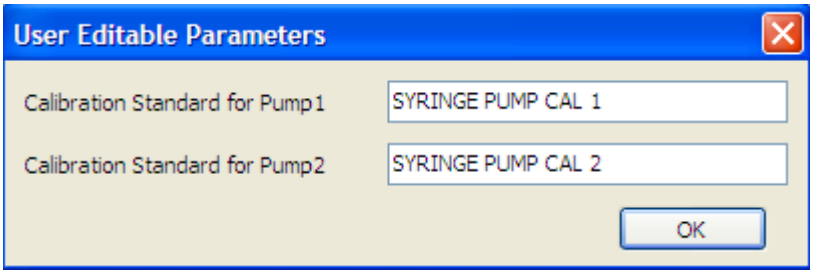


Figure 19: User Editable Parameters

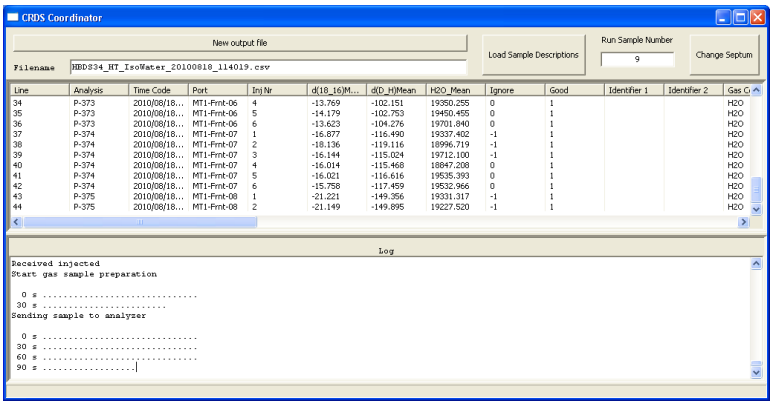


Figure 20: Coordinator Window

11) After 1-2 hours of operation, inspect all the four connections of each pump for any leaks. Using a small light source, check both the outside of the connectors as well as the inside for signs of water. Very small leaks take a very long time to release enough water to be visible.

12) Put the cover of the SDM back on. Be sure the front and sides of the cover are inside of the tray edge. Be sure the back of the cover is outside of the tray's back wall.

13) The coordinator software will start to run the active sequence that was previously applied using the Syringe pump Sequence Software. 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 SDM Sequencer, apply the desired sequence, and finally restart the coordinator.

14) 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. The coordinator will automatically create a *.csv file containing the measured values. The .csv file can be read using Notepad++, which is provided on the Picarro CPVU (Computer Power Vacuum Unit).

15) For information on how to post process SDM Data and how to set the output size and location of SDM Data files, see the **Data** section of this chapter.

16) To calibrate one's system, see **Calibration**.

17) When in need of shutting down the system, refer to **Shut Down in OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual** for directions. To transport and store the system optimally, see **Transportation & Storage.** Also see tips below.

18) **Optional:** When in need of installing a rotary valve control, it should be connected to the COM 2 port back of the analyzer, and the Enable Rotary Valve Control button should be selected in the syringe pump sequencer software in order to make use of this functionality. See **External Valve Sequencer in OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer and Peripherals User's Manual** for more detail.

19) While operating the SDM, please keep in mind the SDM has been pre-programmed with certain operation 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 skipped over by the analysis software.
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

To post process SDM Data by using the Syringe Pump Data Processor window. The SDM purchase includes data processing software for user's convenience.

To start the software, double click on the icon SDM Data Processor in the desktop. The Syringe Pump Data Processor window will pop up. See descriptions of the software below. Under the File menu, select Load File and choose the file to be processed. Raw files are stored under C:\SyringePumpData by default.

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 *.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.

High Standard Deviation?

If the reported standard deviations (SD) appear high for any particular standard delivery note the time and view the H_2O 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.

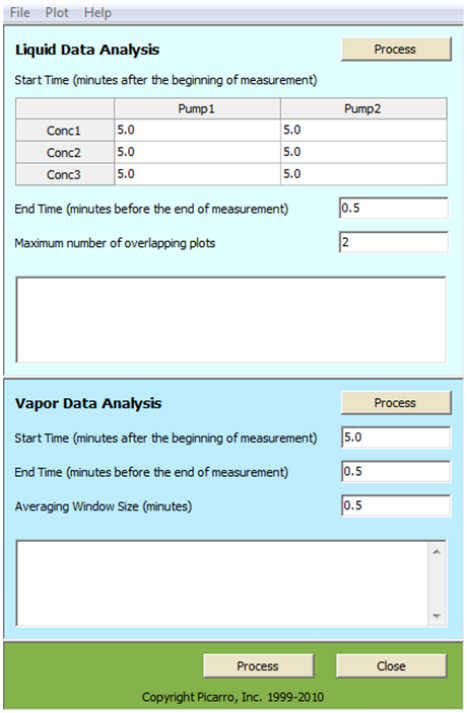


Figure 21: The Syringe Pump Data Processor Window

Under the Liquid Data Analysis section, select the start time for each pump and the concentration value. • **START TIME:** The start time has 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 ∂D value vs. time for each pump and concentration to optimize this parameter further. The ∂D value takes longer to equilibrate than the $\partial^{18}O$ value. • **END TIME:** 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. • **MAXIMUM NUMBER OF OVERLAPPING PLOTS:** The maximum number of plots is limited to 4. These are useful for quick visual assessment of the data. **THE PROCESS BUTTON:** 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. Below are explanations for each sub-section. • **START TIME:** 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. • **PROCESS BUTTON:** The button will process the data for vapor data analysis only. • **PROCESS BUTTON:** To process the data for both liquid and vapor, press the process button at the very bottom of the window.

To limit the size of the output file and to change the location of the output file, see directions in the **Best Practices and Operational Tips and Tricks section**.

CALIBRATION

Picarro recommends calibrating your Picarro L21x0-i analyzer using liquid water samples (see **Calibration in the OPERATION | L2140-*i*, L2130-*i* or L2120-*i* Analyzer**) with water isotope standards that should have accepted values for $\delta^{18}O$ and $\delta^{2}H$. If applicable, the standards should also be calibrated for ^{17}O-excess. Picarro recommends tying all isotope data back to the internationally-recognized VSMOW-SLAP scale.

For vapor phase measurements, an additional calibration procedure is recommended to correct for the water concentration dependence.

Calibrating Vapor Phase Measurements

Unlike water liquid measurements where liquid injection volume and air flowrate are set, the water concentration in vapor phase measurements can vary from 1000 to 50,000 ppm. Picarro water isotope analyzers are factory-tested and corrected for the water concentration dependence of the isotope measurement in vapor phase (see Figure 22).

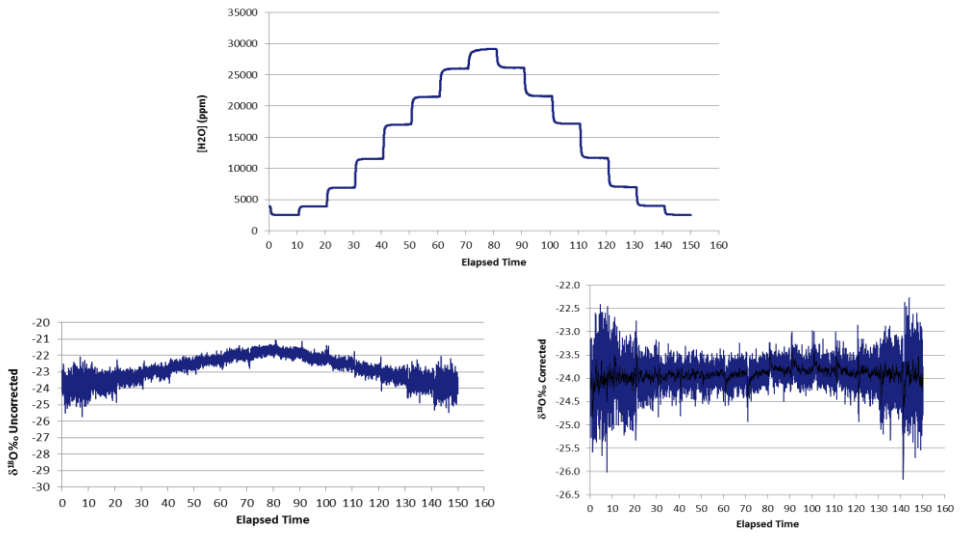


Figure 22: Isobox Testing, (Top) Water Concentration vs Time, Uncorrected (right) and Correct (left) $\delta^{18}O$

In addition to the built-in correction model, we recommend normalizing the analyzer vapor measurements against the results of liquid water test runs using the SDM.

The SDM enables you to automate the calibration test by continuously feeding a liquid water standard with known isotopic composition at various flowrate to a bubbler apparatus. As the liquid water mixes with the constant air flow before entering the vaporizer; the water concentration is controlled by adjusting the SDM pump flowrate.

Using the SDM test results, plot the water concentration on the horizontal (x) axis and the difference between standards' known isotopic composition and the analyzer's recorded isotope value on the vertical (y) axis. This graph will help you to determine the linear relationship between the deviation/correction values and the water concentration.

Determine a linear best-fit equation from the data. The slope and intercept of the best-fit line through these points are the two values that are used to post-process ambient vapor delta values with variable water concentration.

One can then correct the δ measurement at a given water concentration as follow:

$\delta_{corrected} = \delta_{measured} + f(c)$

f(c) is the deviation/correction as a function of the water concentration. f(c) is a linear regression function of the difference between the actual δ and the measured δ at various water concentration.

Figure 23 shows an example on the plots the SDM test results where the standard concentrations varied from 8,000 to 25,000 ppm.

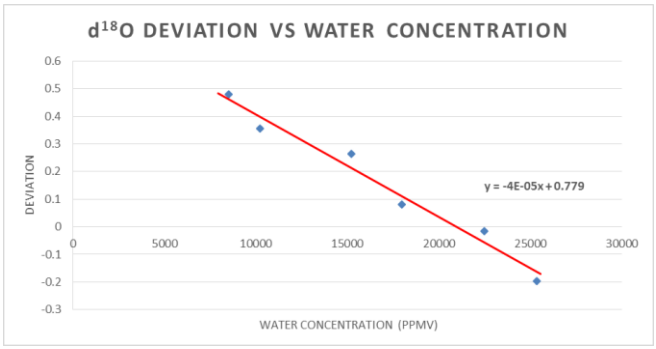


Figure 23: Correction Models for $\delta^{18}O$ and $\delta^{2}H$

BEST PRACTICES AND OPERATIONAL TIPS AND TRICKS

SDM Field Deployment Tips

- Run the SDM in the lab to become fully familiar with its setup and operation before undertaking a field deployment.
- 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.
- Be prepared. The bags should be filled in the laboratory. Extra lines and needles should be packed as well.
- 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.

SDM Output File

To limit the size of the output file and to change the location of the output file, see directions below. 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 it will easily exceed the upper limit of records in MS Excel (65536). 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:

Step 1: Open CoordinatorSDM_G2000.ini file

Step 2: Use Notepad++ text editor (right click on file)

Step 3: Under section marked `[FILES]` at line 8 the default will be: `max_num_lines=`

Step 4: If it is left blank after the equals sign then there is no size limit

Step 5: To specify a limit on the number of records enter an integer value after the equal sign. For example: `max_num_lines=2000`

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

To change the location of the output file:

Step 1: Open CoordinatorSDM_G2000.ini file

Step 2: Use Notepad++ text editor (right click on file)

Step 3: Under section marked `[FILES]` at line 7 the default will be: `output='C:\SyringePumpData\'`

Step 4: create a new location by changing the value in `' '`.

for example `output='C:\MyDocuments\SyringePumpData\'`

This would save the outputs in a separate directory.

TROUBLESHOOTING

The following section lists common problems that may be encountered while operating the SDM (Standard Delivery Module). If, after attempting these procedures, the problem remains unresolved, please see **Technical Support**, or refer to the community site www.picarro.com/community.

Symptom: No communication between software and SDM.

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

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

Recommendation: 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 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.

Recommendation: 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, this means excess water entered the vaporizer previously. 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 behaviour. Amplitude may be worse for lower concentrations.

Recommendation: 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. The pump can generate enough pressured to overcome a partial blockage at higher flow rates but not at lower flow rates. So if the same pump shows oscillation only at low flow rates it is very likely the problem is a partial blockage in the fluid pathway.

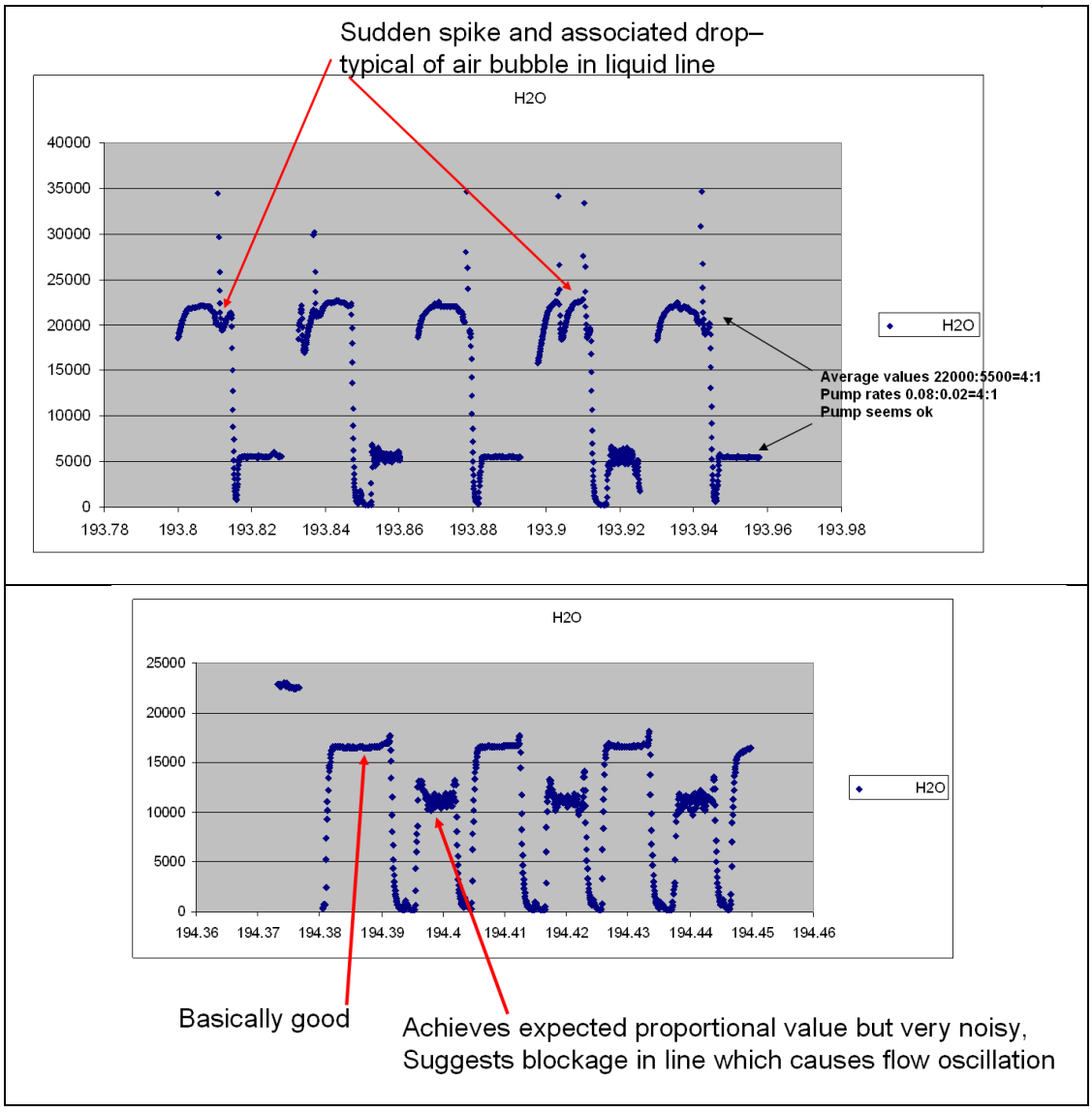
If priming jet contains bubbles and tubing connections are good then "palpate the bag"- i.e. pull at the edges, tap, etc …to detach air bubbles from outlet area so they can rise to the top. Good bag filling and line connection procedure minimizes this problem.

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

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

Symptom: Sudden drop in the vapor concentration?

Recommendation: Ensuring a consistent vapor concentration of the standard requires a consistent liquid flow. The presences 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. This is illustrated in the examples below:


Sudden spike and associated drop–
typical of air bubble in liquid line
H2O
40000
35000
30000
25000
20000
15000
10000
5000
0
193.78
193.8
193.82
193.84
193.86
193.88
193.9
193.92
193.94
193.96
193.98
H2O
Average values 22000:5500=4:1
Pump rates 0.08:0.02=4:1
Pump seems ok
H2O
25000
20000
15000
10000
5000
0
194.36
194.37
194.38
194.39
194.4
194.41
194.42
194.43
194.44
194.45
194.46
H2O
Basically good
Achieves expected proportional value but very noisy,
Suggests blockage in line which causes flow oscillation

WARRANTY CLAIMS

In order to track incidents, and enable our customers to follow progress using the online Picarro Support Community, Picarro has adopted a case number structure for service requests. If you need help from Picarro, please contact us in accordance with these instructions.

1. Contact Technical Support to be assigned a case number.

   Please call: +1 408 962 3991

   Or email: support@picarro.com

   To help us assist you, please provide the following information:

   - Analyzer Serial Number
   - Your Institution
   - A description of the symptom, including error codes when relevant. This will, for example, help us understand whether the problem is related to hardware, software or sample handling.
   - Screen captures, data and photos can also help us
   - We have a number of tools to help customer online and an internet connection will be extremely useful.

2. In some cases, Picarro is unable to resolve the situation remotely and a return is necessary. We will do our best to make this as painless as possible. The first step in the process is to secure a Return Material Authorization number. Your Technical Support representative will email a link to complete and submit our RMA form online. Upon completion of the form, the RMA number will be sent, automatically, as well as additional information regarding the return process, such as appropriate packing, insurance. Units returned without a valid RMA number will not be worked on until the RMA process is complete.

PICARRO

TECHNICAL SUPPORT

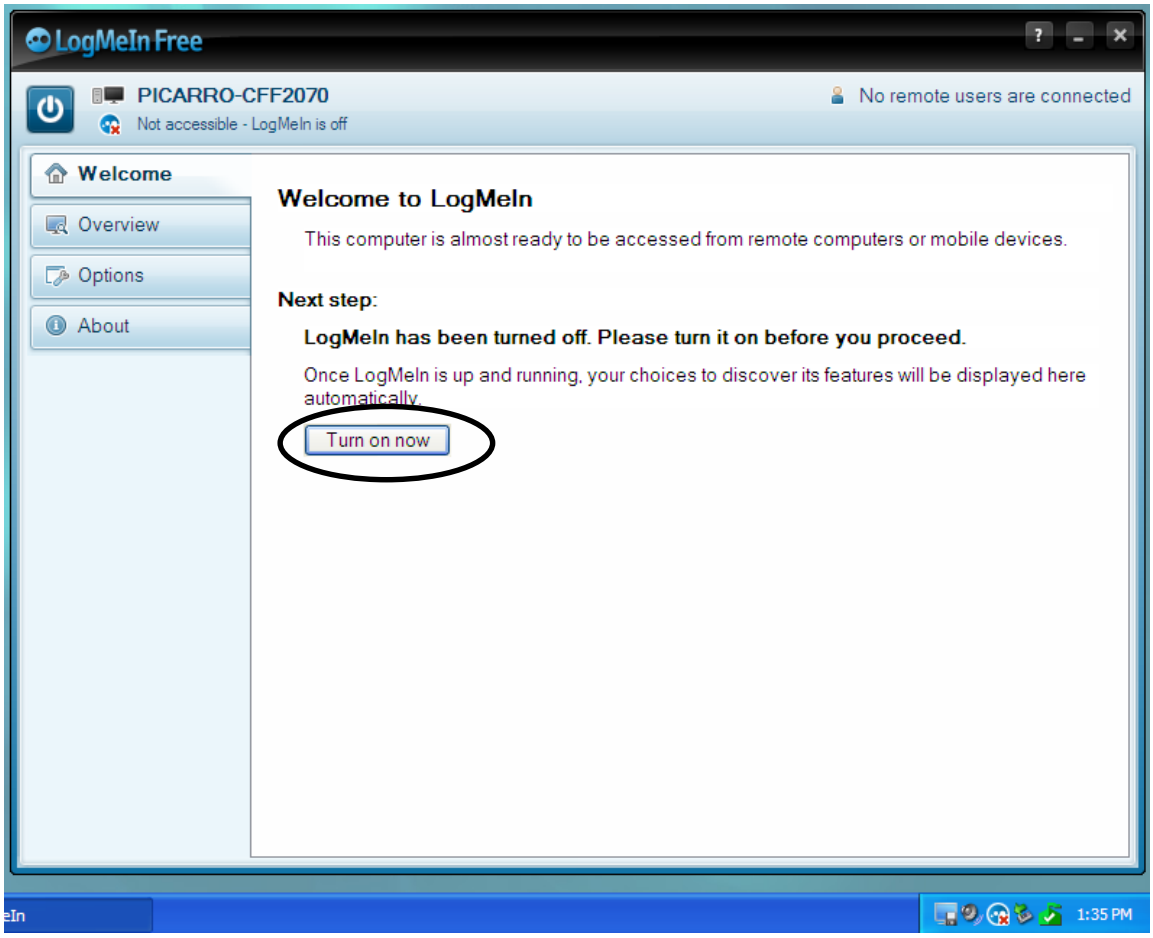
We are committed to helping our customers! Following the steps below will help us get to your problem faster.

- **Visit our popular Community forum at www.picarro.com/community.** It offers a wealth of information with answers to thousands of questions from our customers as well as useful links and updates to operate your analyzer optimally. If this is your first time visiting this forum, you will be asked to login using your username and password, which can be created easily with a special email invitation from Picarro. These invitations are automatically emailed to current customers upon purchase as well as to interested individuals; otherwise, please email us to request an invitation to community.
- **Email us at support@picarro.com.** We will get back to you right away. We highly recommend that you attach data and/or screen shots that you feel might help us diagnose your problem to your email.
    - **Please activate the LogMeIn software before emailing us (see tutorial below).** This activation allows our technical engineers to get access to your analyzer's desktop remotely, allowing us to find and solve your problem quickly. This access can easily be turned off by the user.
- **Call us at 408.962.3991.**

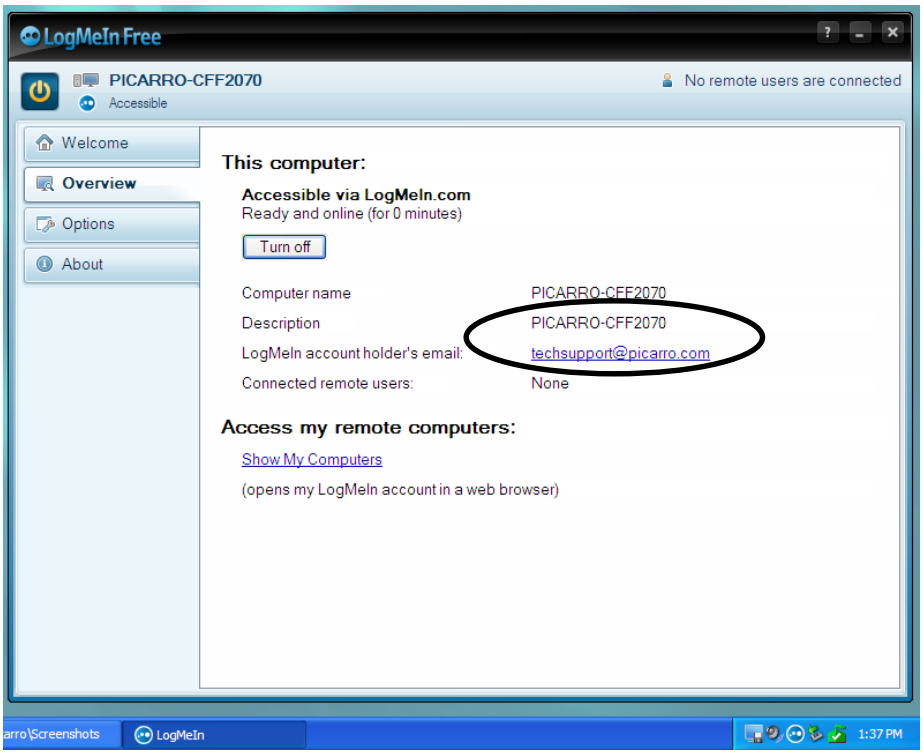
TO ACTIVATE THE LOGMEIN SOFTWARE

1. Click on the “LogMeIn” icon in the Windows task bar at the lower right hand corner. The “LogMeIn Free” window will pop up.

2. Click the “Turn on now” button.



3. Send both the “Description” and “LogMeIn account holder’s email” entries to Picarro, including a description of your problem. The “LogMeIn account holder’s email” shows the account that the instrument is currently on (default is an @picarro.com email).



4. After your problem has been solved, you can turn off Picarro’s access to your desktop by clicking on the “Turn off” button (see screenshot above).