$\begin{array}{c} PIC \Delta RRO \\ \mbox{INDUCTION MODULE} - CRDS SETUP \end{array}$

User's Manual



The

manual includes the complete information on how to set up and operate the entire IM-CRDS Setup

Picarro Induction Module – CRDS SETUP and User's Manual 40039 Rev. B Revised April 2015

User's Manual

Thank you for purchasing a Picarro product. Your Picarro Induction 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 Induction 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.comPhone:408.962.3991(See "Need help from Picarro?" chapter for more information.)

Contact Customer Service:

Email:	orders@picarro.com
Phone:	408.962.3992

Picarro, Inc. reserves the right to change or update the contents of this manual and to change the specifications of its products at any time without prior notification. Every effort has been made to keep the information in this document current and accurate as of the date of publication or revision. However, no guarantee is given or implied that this document is error free or that it is accurate with regard to any specification.

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 Analyzer.

Picarro and the Picarro Logo are trademarks of Picarro, Inc.

© 2015 Picarro, Inc. All rights reserved. 3105 Patrick Henry Dr. Santa Clara, California CA 95054 USA.

Phone 408.962.3900 • Fax 408.962.3200

TABLE OF CONTENTS

INDUCTION MODULE – CRDS SETUP1
INTRODUCTION: Installation, Operation, Data Analysis, Maintenance, & Troubleshooting IM SETUP
SAFETY
GETTING STARTED
INSTALLATION / INDUCTION MODULE MODE SETUP
IM SAMPLE DESCRIPTION
IM METHODS AND TIPS FOR RUNNING THE IM 19
INTRODUCTION:
FLUSHING SAMPLE VIALS AND RUNNING BLANKS:21
RECIPES:
MODIFYING AND CREATING RECIPES:27
CALIBRATING THE IM:
INDUCTION MODULE OPERATION
IM COORDINATOR DATA COLUMNS
TUNING IM DATA PULSE SHAPE
SERVICE & MAINTENANCE INDUCTION MODULE
1. NEEDLE REPLACEMENT INSTRUCTIONS49
2. SCRUBBER CARTRIDGE REPLACEMENT INSTRUCTIONS
3. MICRO COMBUSTION CARTRIDGE REPLACEMENT INSTRUCTIONS52
TROUBLESHOOTING / INDUCTION MODULE
WARRANTY CLAIMS
NEED HELP FROM PICARRO?

INTRODUCTION: Installation, Operation, Data Analysis, Maintenance, & Troubleshooting | IM SETUP

The Picarro IM (Induction Module) extracts water from samples such as soil, plants, tissues or juices and enables isotopic analysis of the extracted water. The IM includes Picarro's Micro-Combustion Cartridge that destroys organic compounds via oxidation, eliminating spectral interferences and dramatically enhancing analyzer performance. This guide contains information needed to safely install, operate, and maintain your IM. It should be used in conjunction with the Operation, Data Analysis, Maintenance and Troubleshooting User's Manual which accompanies your isotopic water analyzer (L2120-*i* or L2130-*i*).

Due to the variety of sample types that can be analyzed by the IM, experimentation and methods development specific to your sample type may be required to optimize the IM's performance.

OPERATION:

- 1) Before continuing, review the important safety notes in the section entitled Safety.
- 2) Make sure the hardware setup is complete and the system turned on in the correct sequence (section Installation).
- 3) Once turned on, the main GUI (Graphical User Interface) of the analyzer will open up automatically on the desktop screen. To understand all the functions of the main GUI, see Appendix J of the analyzer 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 Appendix K of the analyzer User's Manual.
- 4) Complete directions on how to operate the Induction Module setup can be found in section Induction Module Operation.
- 5) For description of individual data columns while operating in IM Coordinator mode, see section IM Coordinator Data Columns.
- 6) To load sample description to the IM coordinator, see section IM Sample Description.
- 7) For information on how to work with different sample types (e.g. leaf, stem, juices, etc.), see section IM Methods and Tips for Running the IM.

- 8) On the analyzer's desktop, there are various useful icons & folders. To learn more, see Appendix H of the analyzer User's Manual.
- 9) To calibrate the IM, see section IM Methods and Tips for Running the IM. To calibrate the analyzer with other peripheries, see Appendix P of the analyzer User's Manual.
- 10) For directions on shutting down the system, refer to Appendix N of the analyzer User's Manual. To transport and store the system optimally, see Appendix W of the analyzer User's Manual.

DATA:

- 11) To learn how to retrieve the data, and set the frequency of file archival and automatic deletion of old files, see **Appendix L** of the analyzer User's Manual.
- 12) To configure data file saving details, including which data elements are written to data files, digital data output (via serial port or TCP/IP), remote data delivery (via email), and general GUI properties, click on the Setup Tool icon in the Picarro Utilities folder in the desktop. For more information, see Appendix M of the analyzer User's Manual.
- **13)** The Picarro isotopic water analyzers allow users to archive data using a highly-compressed, binary "HDF5" or "h5" format. The Data File Viewer program, which comes installed with the hardware, allows one to open and convert h5 files, as well as view the h5 files as graphs. For more information, see **Appendix L3** of the analyzer User's Manual.
- 14) The Picarro isotopic water analyzers come preinstalled with the ChemCorrect software which allows one to screen and quantify contamination in isotopic water samples. To post process data using ChemCorrect Software, see Appendix L4 of the analyzer User's Manual. At this time, ChemCorrect does not interface with data output from the IM and therefore cannot be used to post-process IM data.
- 15) To tune the pulse shapes of IM data, see section Tuning IM Data Pulse Shapes.

MAINTENANCE:

16) With the exception of the particulate filter, the analyzer is not user serviceable. For information on how to change the particulate filter of the analyzer, see Appendix U of the analyzer User's Manual. For maintenance specific to the IM (e.g., replacing the Micro-Combustion Cartridge), see section Service and Maintenance, Induction Module.

TROUBLESHOOTING:

- 17) For information on troubleshooting the CRDS analyzer, see Appendix V of the analyzer User's Manual. The "Cavity Ring-Down Spectrometer Controller" software can be used as a diagnostic tool in troubleshooting the analyzer (see Appendix V1 of the analyzer User's Manual). To troubleshoot the Induction Module, see section Troubleshooting, Induction Module.
- **18)** When in need of direct help from Picarro, see section **Need Help from Picarro?** For warranty information, see section **Limited Warranty.**

SAFETY

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.
0	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.
\otimes	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.
\otimes	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.
\Diamond	CAUTION: The analyzer is heavy. To avoid injury, please use proper 2-person lifting procedures when moving or installing the equipment.
${igsidents}$	CAUTION: The Induction Module has heated components with HOT SURFACES. To avoid injury, be sure to let the system cool prior to replacing the needle, scrubber or micro combustion cartridge.
\Diamond	CAUTION: The Induction Module sample port contains a needle. To avoid injury, do not insert your finger into the sample port when there is no vial present.

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.

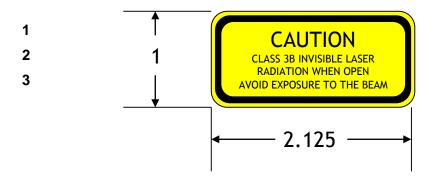


CAUTION: CLASS 3B INVISIBLE LASER RADIATION WHEN OPEN. AVOID EXPOSURE TO THE BEAM.

The lasers inside of the analyzer emit a maximum of 50mW 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.



ΡΙΟΔRΒΟ

GETTING STARTED

INSPECTING THE BOXES

All Picarro products are inspected and tested prior to shipment from the factory. In addition, the instruments are packed in an inner box which is encased in a larger outer box. The shipping container system has been specially tested and proven to be safe for most dropping, crushing or spiking events.

Upon delivery of instrumentation, the package should be checked thoroughly for damage or signs of shock.



In the event of severe damage to the outer box, photograph the damage and contact Picarro (please email picture) for consultation on best course of action.

UNPACKING THE BOXES

CAREFULLY unpack the contents from the boxes. The shipment will come with a packing list. Please consult this list to confirm all contents are present. If any of these items are missing, contact Picarro for a replacement. Inspect each item to assure it is not damaged.

It is recommended that one keep the shipping packages. These shipping packages are also a very good way to ship the system to other labs or field stations, unless they will get wet. Please contact Picarro for options on transporting systems to remote labs.

FACILITY PREPARATION

• Space requirements: roughly 3x3 feet (0.9x0.9m), including the analyzer.



NOTE: Take care to ensure that warm air is exhausted from any enclosure in which the analyzer is mounted.

INSTALLATION | INDUCTION MODULE MODE SETUP

INSTALLATION STEPS:

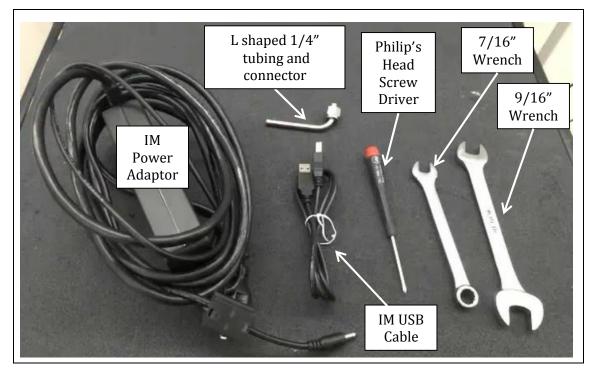
- 1 Inspect the boxes of Picarro products before opening. Carefully unpack the boxes and prepare the facility (see section **Getting Started**).
- 2 Please review the important safety notes before continuing on with the installation. See section **Safety**.
- 3 Please have the following materials ready before getting started.

Not Included:

- Gas regulator supplying 2.5 +/- 0.5psi (172 mbar +/- 35mbar).
- Flow meter (optional, strongly recommended)
- Thermocouple (optional, recommended)

Included:

- USB cable
- Power adapter and cable
- L-shaped metal tubing (1/4" stainless steel with 1/4" Swagelok connection)
- 4 Set up the analyzer and its external vacuum pump by referring to **Appendix F2** of the L2120*i* or L2130-*i* Installation User's Manual.
- 5 At this point you can start installing the IM, however it should not be operated until the analyzer starts making measurements giving water concentration and delta values. If the instrument has been off for an extended period of time, you should let the analyzer sample from ambient air for at least 30 minutes to stabilize.



6 To start installing the IM, gather the following items:

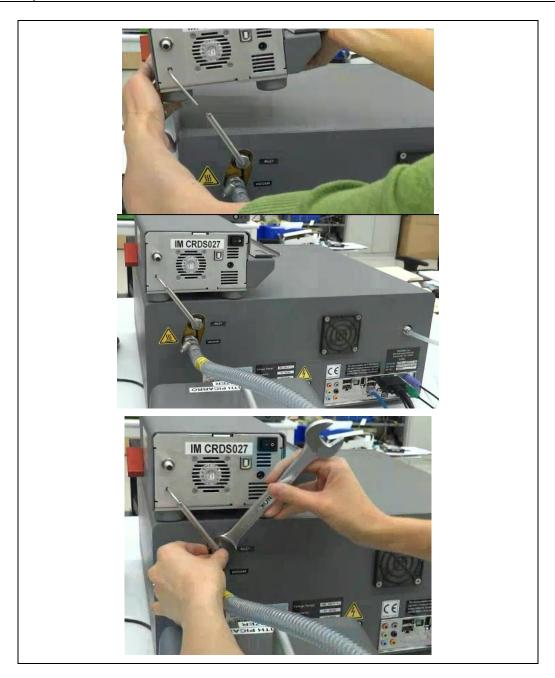
7 Connect and finger-tighten the L Shaped 1/4" tubing to the analyzer.



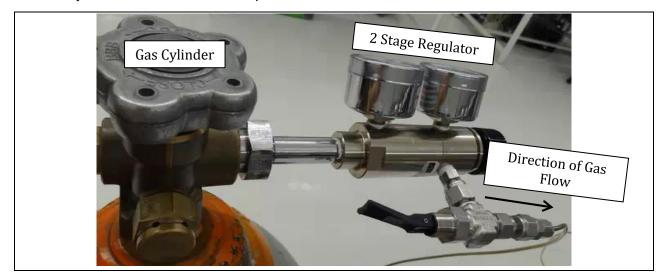
8 Place the IM on top of the analyzer so that the outlet tube extending from the back of the IM is inserted into the inlet adaptor of the analyzer. Make sure the IM is secure on its four feet on the analyzer. If necessary, adjust the angle of the tubing. Use the 9/16" wrench to tighten the connection.

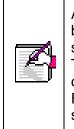


The IM is attached to the analyzer via an open split. An open split is used because the flow rate out of the IM (\sim 150 sccm) is higher than the flow rate of the analyzer (\sim 40 sccm).



9 Connect a source of dry gas to the back of the IM at a stable pressure of 2.5 psi. You can only use zero air as a source of dry gas. There are many ways to plumb dry gas into the IM. For example, you can use two regulators in-line where the first steps down the pressure of the zero air tank, and the second keeps the pressure stable at 2.5psi. It is recommended that the second regulator be connected to a flow meter, prior to connecting to the IM. The flow meter should read about 150 sccm when the IM is attached and on. During sample analysis the IM flow rate will drop to about 140 sccm.





Although a gas flow meter is not required, it acts to verify the IM flow rate, and it can be helpful in troubleshooting and recipe development. For most samples, the flow rate should be about 150 sccm, corresponding to an approximate input pressure of 2.5psi. The acceptable flow rate range is 90 to 180 sccm. Depending upon the water content of samples and the ease of water extraction, the flow rate may need to be adjusted. For consistency, make sure all samples and standards (from the same analytical session) are run at the same flow rate.

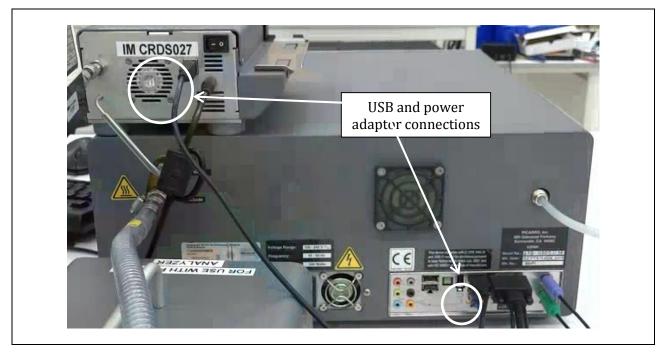


Picarro Induction Module – CRDS SETUP and User's Manual 40039 Rev. B Revised April 2015

10 Once the gas line is ready, connect it to the back of the IM using a 7/15" size Swagelok connector. Finger-tighten the connection and then use a 7/16" wrench to go another quarter turn or so to tighten fully.



11 Connect the cables and wires. *Make sure that the power switch for the IM is off before connecting cables.* Connect the USB to the back of the IM, and the other end to any port on the back of the analyzer. Plug in the power adaptor on the back of the IM.

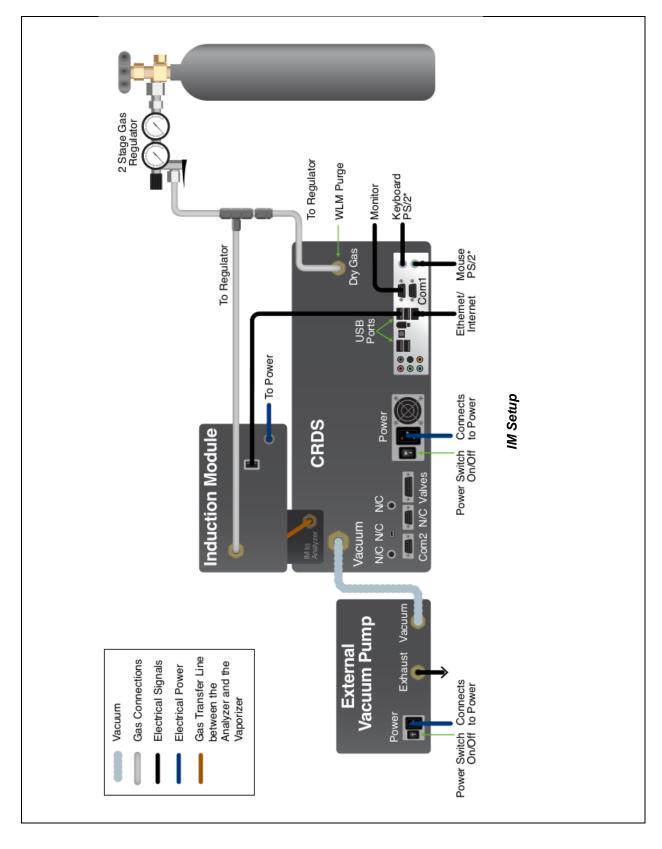


Picarro Induction Module – CRDS SETUP and User's Manual 40039 Rev. B Revised April 2015



IMPORTANT! Make sure that the analyzer and IM are plugged into the same outlet or the same power strip. Failure to do so will likely result in grounding loops that will interfere with the IM or prevent it from functioning.

12 Turn on the IM using the switch on the back of the module.



IM SAMPLE DESCRIPTION

Sample descriptions can be added before, during, or after the data analysis. To add a sample description or name, the sample names must first be entered into the sample description file. An example file can be found here:

C:\Picarro\G2000\AppConfig\Coordinator\example_IM_descrp.csv

The file (.csv) should be opened with Notepad++ software for ease of use. The sample descriptions should be entered in the following format:

Vial, Description 1,"Sample Name 1" 2,"Sample Name 2" 3,"Sample Name 3"

With the descriptions loaded into the file and saved, and with the IM Coordinator running, click on the "Load Sample Descriptions" button on the Coordinator. You will be prompted to select the sample descriptions file from the folder in which you saved it.

By default the order from the sample description file will align with the order in the coordinator output. You can also change the description after they have been loaded by selecting the appropriate description from the dropdown list at the bottom of the Coordinator window. Once selected, click the button to the right labeled "Injected" to add the description to the line of data for that sample. This description will then appear in the resulting data file located in C:\lsotopedata.

The Coordinator will start creating a file at the beginning of a run, but if a new output file is desired, click "New Output File" button at the top of the Coordinator window. This will start a new output file with the date and time in the file name.

IM METHODS AND TIPS FOR RUNNING THE IM

INTRODUCTION:

The Picarro Induction Module is capable of extracting water from many different types of samples, e.g., water on filter paper, matrix bound water in leaves and stems, soil water, and fruit juices. This chapter describes the three basic sample methods, and recommends their use for different types of samples.

- 1. Liquid samples, e.g., calibration water on filter paper. This method can also be used for other liquids, including fruit juices and saline waters.
- 2. Leaves.
- 3. Woody stems.

By no means is this list of methods exhaustive, and the user is encouraged to modify the method and water extraction approach for additional sample types. To modify, or create a new method, see section "Modifying and Creating Methods".

The method files "RecipeMBW.ini" can be found in C:\PicarroG2000\AppConfig\Config\Utilities\. To view the methods, open them using Notepad++ (installed on all Picarro analyzers).

Sample Type	Recipe name
Liquid samples	"Liquids – Higher Temperature" or "Liquids – Lower Temperature"
Leaves	"Leaves" or "Thick Leaves"
Woody stems	"Woody Stems"



Notepad++ is recommended above other text editors as it will preserve the appropriate format of the ASCII text file.

The heating parameters, which are fundamental to each recipe and control how quickly the sample is inductively heated and with what power, are described below. Values of the polyA,B,C correspond to the scalars in the equation $y = Ax^2+Bx+C$ describing the rate of heating (y) in time (x).

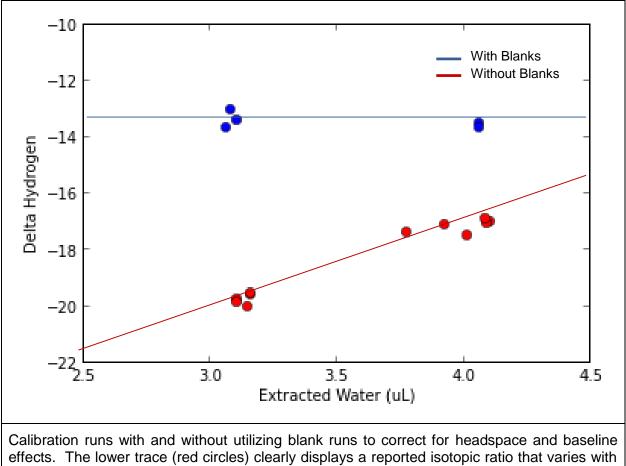
Method Parameter	Description
polyA	2 nd Order (or Quadratic) Polynomial coefficient, A, defining the heating ramp.
polyB	1 st Order (or Linear) Polynomial coefficient, B, defining the heating ramp. If A equals zero, coefficient B results in a linear change in heating with time.
polyC	0 th Order (Constant) Polynomial coefficient, C, defining the heating ramp. C has no dependence in time, and therefore represents constant heating.
H ₂ OLowThreshold	Threshold background H ₂ O concentration (ppm). The method will not start until this background concentration is achieved. We strongly suggest the user does not alter this parameter. Increasing this parameter will increase the probability that the system will experience a concentration-dependent systematic error.
heatTime	Duration of heating time (seconds).
H2OEndHeatThreshold	Threshold H ₂ O concentration for the completion of method. This parameter is no longer used in the updated coordinator (as of August 2013), but remains in the recipe file for backward compatibility.
preheatTime	The time prior to heating. This parameter is no longer used in the updated coordinator (as of August 2013), but remains in the recipe file for backward compatibility.

In each of the methods described below, the parameters and recommended gas flow rate are given. The gas flow rate can be adjusted by slightly adjusting the inlet pressure regulator above or below the nominal 2.5psi. A flow meter must be placed in-line between the regulator and the IM to monitor gas flow. Once a correspondence between gas flow and pressure is determined, the flow meter can be removed if desired.

PICARRO

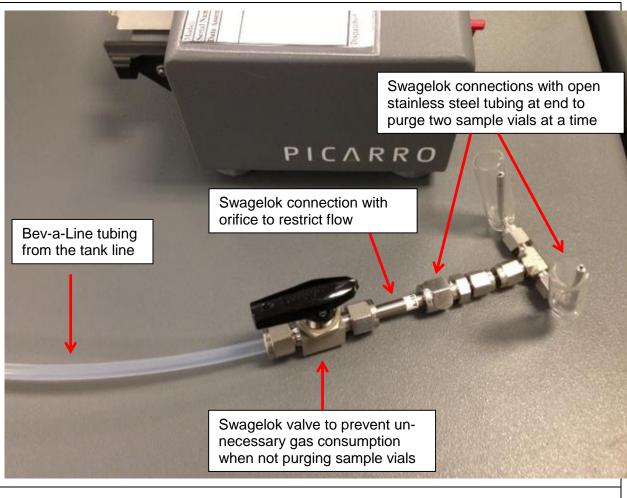
FLUSHING SAMPLE VIALS AND RUNNING BLANKS:

Picarro highly recommends flushing sample vials with zero air and running blanks for both calibration and sample runs. This is necessary to combat headspace error. The magnitude of this error depends on the difference between the isotopic composition of the local atmosphere and the sample, and on the concentration of water in the sample.



effects. The lower trace (red circles) clearly displays a reported isotopic ratio that varies with the total amount of extracted water. Running blanks (blue circles) will correct the possible systematic error.

It is possible to flush sample vials using the same zero air tank as that used for the IM and L21x0*i* analyzer.



An example of the fittings used at Picarro to purge IM sample vials prior to running both samples and blanks. The Bev-a-Line tubing can be teed off the tank line feeding the IM and analyzer. A valve should be installed to stop the flow when not purging vials. We recommend leaving the vial on the purge apparatus while preparing your sample. After the sample is prepared, insert the sample holder into the vial and cap immediately.

The purge apparatus uses an orifice to restrict flow, such that it can be plumbed in parallel with the IM.



Do not turn the Swagelok purge line valve on and off during IM runs. This will result in errors in the reported delta values due to variable flow rates.

RECIPES:

1. LIQUID SAMPLES:

Recipe names: "Liquids – Higher Temperature" and "Liquids – Lower Temperature"

Temperature of recipe – Each Picarro IM is unique and the temperature achieved during induction heating will vary slightly. As a rule of thumb, the "Liquids – Higher Temperature" recipe corresponds to a max temperature seen by the sample of about 180 to 200°C, while the "Liquids – Lower Temperature" recipe corresponds to a max heating of about 100°C.

Sample size: $3 - 4 \ \mu L$ of liquid on a piece of hole-punched filter paper

Sample holder: Tri-fold metal strip

Recommended tools: Pliers, hole punch, syringe

Procedure: The recommended calibration mediums are glass filter paper and Teflon washers. Cellulose filter paper can also be used, but exchangeable hydrogen ions will influence the results. Place the hole-punched sized calibration medium into the tri-folded metal strip. Use pliers to crease the metal and fold down the tab. Inject 3 - 4 mL of liquid water onto the calibration medium and introduce the sample to the IM using the tri-fold metal strip. Insert this into the vial. Details on how to operate the IM are provided in section **Induction Module Operation**.

Ø	Cellulose filter paper – Due to exchangeable hydrogen ions, cellulose filter papers impart memory to the measured isotopic composition of water. Therefore, we recommend the use of glass filter paper over cellulose filter paper. If cellulose filter paper must be used, each sample should be introduced 5 or more times using the same piece of filter paper, while discarding the data until it reaches a plateau. If you are running different samples, use a different piece of filter paper for each sample, and be sure to run multiple replicates of the same water on each piece of filter paper. The calibration will be compromised if you only measure one replicate of each sample.
P	 Picarro recommends the following calibration mediums: Glass filter paper – These filter papers have reduced memory effects (as compared to cellulose filter paper). The same sample should be introduced 3 or more times using the same piece of filter paper, with the first two data points discarded due to memory effects. Picarro has tested Whatman Glass Microfiber Binder Free filter paper, Grade GF/B, 0.86mm. Teflon washers – Teflon washers have limited memory and therefore a sample need only be replicated 3 or 4 times. The disadvantage of utilizing Teflon washers is the difficulty in positioning the washers in the metal sample holder, and the limited absorption capacity.

Recipe parameter values:

Parameter	Liquids – Higher Temperature (heats to approximately 180- 200°C)	Liquids – Lower Temperature (heats to approximately 100°C)
polyA	0.00021	0.00017
polyB	0.00001	0.0000093
polyC	13	10
H ₂ OLowThreshold	250	250
heatTime	180	180
H2OEndHeatThreshold	200	200
preheatTime	0	0

Gas flow rate: ~ 150 sccm (idle); equivalent to ~ 140 sccm during analysis

After data collection: Each liquid sample should be introduced onto the same filter paper for replication. After each sample has been analyzed, the filter paper should be removed from the metal sample holder and replaced with a new piece of filter paper. If using Teflon washers they can be re-used for different samples.

2. LEAVES:

Recipe names: "Leaves" or "Thick Leaves"

Sample size: Dependent on the water content of the leaf. Start with one hole-punch of leaf, and adjust as necessary. The target extracted water is 3 to 4 μ L.

E	Estimated water content – The coordinator output includes an estimate of the total volume of water extracted from each sample under the heading "Est. H2O Volume (microliters)". It is an estimate because the calculation is dependent on flow rate, and assumes a baseline flow rate of 140 sccm during analysis. If the flow rate is higher (lower) than this, the coordinator output will underestimate (overestimate) the H ₂ O volume.
B	Sample size – To minimize the magnitude of measurement error, we suggest a minimum extracted water sample volume of approximately 2 μ L. Sample volumes should be approximately the same size as the volume of water used to calibrate the system, i.e., if you introduce 3 μ L of water of a known isotopic composition onto glass filter paper, target 3 μ L of extracted water for your samples.

Sample holder: Tri-fold metal strip

Recommended tools: Pliers, hole punch

Procedure: Using a hole-puncher, punch a hole in a leaf and collect the sample. Place the leaf sample on the tri-folded metal strip. Use the pliers to crease the metal and fold down the tab. Insert this into the vial. Details on how to operate the IM are provided in section **Induction Module Operation**.

Parameter	Leaves	Thick Leaves
polyA	0.00017	0
polyB	0.00001	0.015
polyC	13	18
H ₂ OLowThreshold	250	250
heatTime	300	600
H2OEndHeatThreshold	200	200
preheatTime	5	5

Recipe parameter values:

Gas flow rate: ~ 150 sccm (idle); equivalent to ~ 140 sccm during analysis

After data collection: Dispose of the sample. The metal sample holder can be re-used for different samples, but should be replaced once the contact between the metal strips degrades.

3. WOODY STEMS:

Recipe name: "Woody Stems"

Sample size: Dependent on the water content of the woody stem. Start with a thin (~ 0.05 mm) cross-sectional slice of woody stem that is about 1 cm in diameter. Adjust the thickness as necessary to target an extracted water content of 3 to 4 μ L.

Sample holder: Tri-fold metal strip

Recommended tools: Razor blade or box cutter (and a cutting surface)

Procedure: Make a fresh cut in the stem, so as to ensure the surface is representative of the plant and has not dried out following field sampling. Cut thin cross-sectional slices along the stem at the appropriate sampling interval (for example, one slice every one cm). Insert each individual cross-sectional slice into the metal strip and fold over the metal tab to ensure contact between the metal and the sample. It is recommended that each sample be prepared just prior to being run (to avoid the sample drying out between being sampled and being analyzed using the IM). Insert the metal strip with sample into the vial. Details on how to operate the IM are provided in section **Induction Module Operation**.

Recipe parameter values:

Parameter	Woody Stems
polyA	0.00003
polyB	0.03
polyC	15
H ₂ OLowThreshold	250
heatTime	240
H2OEndHeatThreshold	200
preheatTime	0

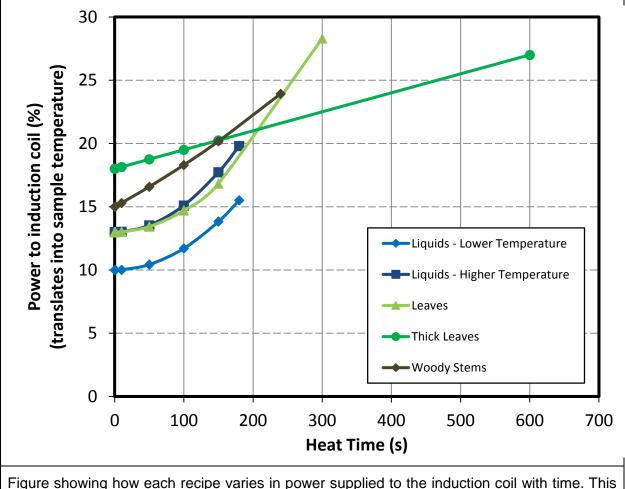
Gas flow rate: ~ 150 sccm (idle); equivalent to ~ 140 sccm during analysis

After data collection: Dispose of the sample. The metal sample holder can be re-used for different samples, but should be replaced once the contact between the metal strips degrades.

MODIFYING AND CREATING RECIPES:

There are infinite different recipes that could be created for running the IM. We have chosen to develop recipes for the most common sample types, but encourage scientists to develop new recipes and methods as necessary.

Each recipe will vary in peak temperature, heating rate and duration of heating time. For example, the recipe "Leaves" heats the sample to a higher temperature, but for a shorter period of time as compared to the "Thick Leaves" recipe.



demonstrates how each recipe will result in a different water extraction curve.

ΡΙΟΔRΟ

The recipes developed at Picarro are optimized for the IM's in-house. The recipes provide excellent starting points for analysis on every individual IM, although it is important to note that each IM is different and therefore small recipe modifications may be necessary. In addition, there are additional recipes available in the recipe file, but they do not automatically appear in the recipe selection window of the IM Coordinator. If you wish to use these, you need to un-comment the recipes in the recipe file. They are not included in the default recipe selection because they have not been rigorously tested at Picarro and may require additional applications development by the user.



It is essential that all matrix-bound water is extracted from each sample because the process includes an inherent fractionation effect. Incomplete extraction will result in inaccurate isotopic compositions. Each of recipe discussed in this manual has been tested at Picarro, and is a recommended starting-point for respective sample type. To determine if all of the water has been extracted from a particular type of sample, see section **Tuning IM Data Pulse Shape**.

To modify or create new recipes, open "RecipesMBW.ini" (stored in C:\PicarroG2000\AppConfig\Config\Utilities\) using Notepad++.

For new recipes, copy a currently existing recipe, e.g., "Calibration – Higher Temperature" and paste it at the bottom. Then adjust the name and recipe parameters as necessary.

For modification of current recipes, copy the recipe in question and paste it directly below the current recipe. Then comment out the current recipe by adding hash marks (#) in front of each line. Change the pasted recipe parameters as necessary.

To access a recipe not shown in the recipe selection window of the IM Coordinator, un-comment the recipe by removing the hash marks (#) in front of each line.

Example of recipe visible in the IM Coordinator recipe selection window	Example of a recipe not visible in the IM Coordinator recipe selection window
[Calibration - Higher Temperature]	#[Whole Leaf]
polyA = 0.00021	#polyA = 0
polyB = 0.00001	#polyB = 0.013
polyC = 13	#polyC = 17
h2oLowThreshold = 250	#h2oLowThreshold = 1000
preheatTime = 0	#preheatTime =5
heatTime = 180	#heatTime = 540
h2oEndHeatThreshold = 200	#h2oEndHeatThreshold = 800

When modifying or creating new recipes, the maximum recommended temperature for induction heating in the IM is 200°C. We recommend testing this threshold by:

- 1. Cut an IM sample vial to remove the bottom.
- 2. Attach a thermocouple to sample holder intended for use in the recipe (e.g., the tri-folded metal strip). Crimp the metal strip tightly to ensure the thermocouple remains inside the sample holder.
- 3. Wrap the sample holder with thermocouple attached with glass wool for insulation, and then insert into the base of the sample vial.
- 4. Run a blank using the open-bottomed sample vial and thermocouple assembly. Close the red door to the IM prior to running the Coordinator sequence.
- 5. Monitor the thermocouple temperature during the blank analysis by using an appropriate data logger, e.g., digital multimeter.



CALIBRATING THE IM:

To report the isotopic composition of samples analyzed by the IM on a recognized international scales (e.g., vs. VSMOW), it is essential to calibrate the system and monitor drift over time. You can do this by running at least two water standards of known isotopic composition on the IM. We recommend using glass filter paper (see section **Induction Module Operation** for details on the operation of the IM and how to run waters on filter paper). Each standard should be run in replicate (using the same piece of filter paper for replicates of the same standard) with the first one or two replicates being discarded. Once you have determined the measured value of each standard, they can be used to construct a regression between the measured and accepted values. This regression can then be used to correct unknown samples to the calibrated scale.



Whenever possible, isotopic standards should be used to calibrate the system using the **same method and recipe** as that used for your samples. For example, if you are running juices using the recipe "Liquids – Higher Temperature", use the same recipe to introduce water of a known isotopic composition, and that has been calibrated against international standards.



Although the IM need not be calibrated each time you use it, you should run at least one working standard during each analytical session to ensure the system has not drifted.

INDUCTION MODULE OPERATION

This section describes how to operate the IM. The specific example given is for running waters using glass filter paper. The operation can be adapted to other sample types (e.g., leaves, juices, etc.)

1. Make sure you have access to the following material.

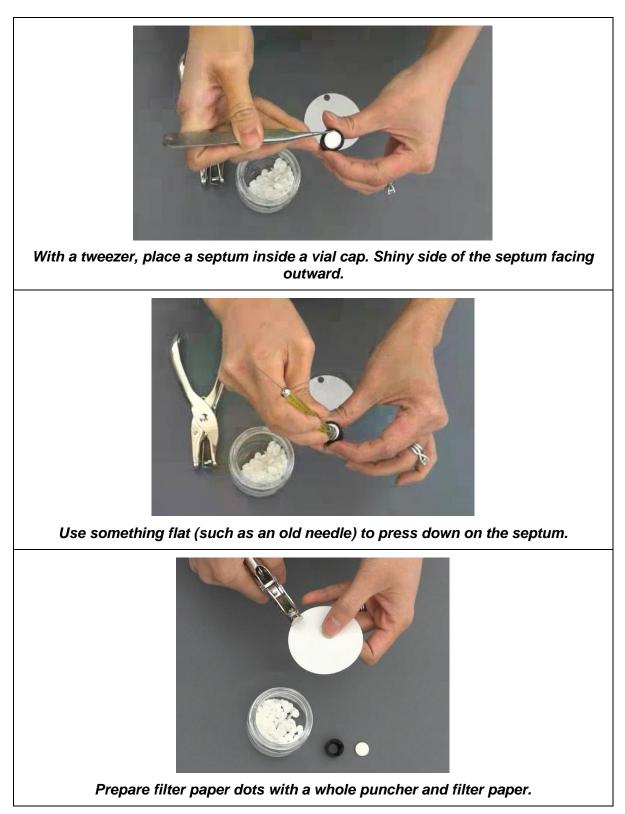
- Tri-fold metal strips (Picarro part # C0333).
- Syringe (10 µL, Picarro part # C0326).
- Filter paper (Picarro has tested and recommends Whatman Glass Microfiber Binder Free filter paper, Grade GF/B, 0.86mm).
- Hole punch
- Pliers
- Tweezers
- 4 mL glass vials, caps and septa (Picarro part #s C0330, C0331, C0332)
- At least two small vials of the water standards intended for calibration use

Þ	Picarro offers IM sample kits of different combinations. The standard kit (Picarro part # C0341) is listed below. Please contact your regional account manager to purchase additional kits or IM consumables.
	C0341 – Picarro IM module kit for 100 flat samples, includes:
	IM tri-fold metal strips x 100
	 IM vials, caps and septa x 100
	IM needle assembly x 1
	IM scrubber cartridge x 1

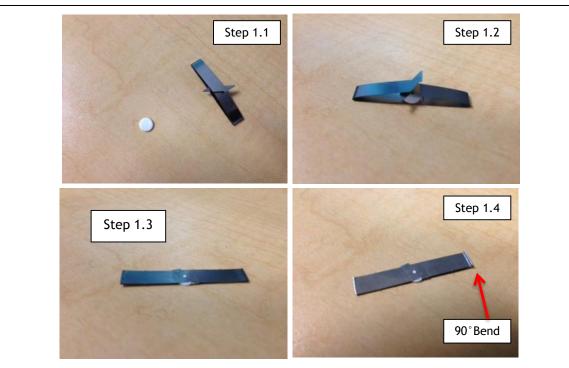
- 2. Make sure the IM Setup is complete and running (see section Installation, Induction Module Mode Setup), with the main Picarro GUI (CRDS Data Viewer) running on the screen. The IM must be hooked up to zero air (or some other completely dry oxygen containing carrier gas) because the Micro Combustion Cartridge requires oxygen to remove spectroscopically interfering organic compounds. Do NOT use nitrogen carrier gas with an IM.
- **3.** Prepare materials necessary to run the IM:
 - a. Vials: The glass vials used to load samples into the IM can be re-used, but we recommended having a handful ready for each analytical session. During the sample run, vials will warm up therefore they should be set aside to cool before being used again.

- b. Vials caps: Place the septum inside the cap with the shiny side facing outwards. If necessary, caps with septum can be re-used (up to 4 times per septum), but we recommended rotating the vial between each sample so that you do not pierce the septum in the same place twice.
- c. Filter paper dots: Filter paper dots can be prepared before or during an IM run. Use a hole punch to cut out circular pieces of filter paper.
- d. Tri-fold metal strip sample holder: When running liquids on filter paper, the sample holders can be prepared during or before an IM run. Separate a tri-fold metal strip from the pack and insert a hole-punched filter paper dot between the flaps of the strip and directly on top of the small hole in the center. Try to line up the paper dot to the round outline on the sample holder. Close the tri-fold metal strip over the paper and fold over the small flaps on either side of the metal strip. Then crimp the middle and ends of the metal strip to ensure the sample holder is flat. We also recommend bending one end of the metal holder to enable the sample to lay flat in the glass vial. If the sample holder does not lay flat within the vial, the heating ramp will be affected, leading to inconsistent peak shapes.

Þ	When running the IM, gloves can be used but are not necessary. The purpose of gloves is to avoid the un-intended introduction of water vapor from the user's hand to the sample.
Þ	The IM is an inductive heating process; therefore the relative location of the filter paper dot within a sample holder, and the relative location of the sample holder within the sample vial are important. Try to be consistent about how you load samples. The more consistent your loading technique, the more consistent your results because each sample will be subjected to the same heating profile.

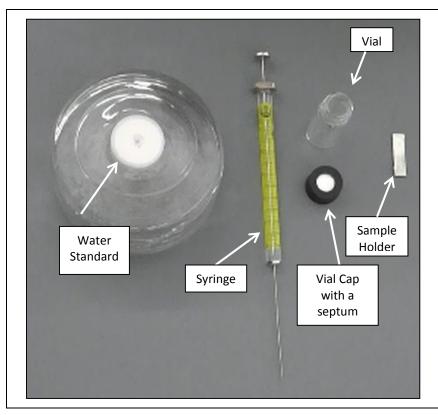


Picarro Induction Module – CRDS SETUP and User's Manual 40039 Rev. B Revised April 2015

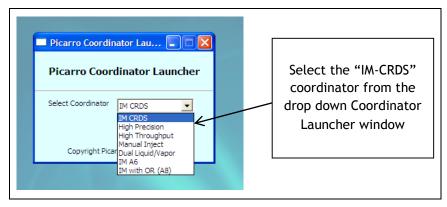


Remove the tri-fold sample holder from the pack (step 1.1). Insert filter paper dots in between the flaps (step 1.2). Line up the paper dots to the round outline on the sample holder. Fold down the two tabs of the sample holder with pliers (step 1.3). Using pliers, make sure the filter paper is in full contact with the sample holder. Bend one end of the metal holder to enable the sample to lay flat in the glass vial (step 1.4).

- 4. Gather the following items:
 - a. Water standard
 - b. Syringe
 - c. Vial with cap and septum
 - d. Sample holder with filter paper



5. Make sure the dry gas (zero air) is flowing and the IM is turned on. Double click the "Coordinator Launcher" icon on the desktop. Choose the "IM-CRDS" coordinator mode. Click "Launch" to continue.



6. The coordinator will now walk you through a number of steps that will allow you to choose between different recipes, and if you are running a blank or sample. The recipes are discussing in section IM Methods and Tips for Running the IM, but as a reminder they are preset heating profiles that can be used for different sample types, such as for leaves, stems, and liquids on filter paper. For filter paper dots, choose either "Calibration – Lower Temperature" or "Calibration – Higher Temperature".

Recipe Selectio	on -	
Current Recipe	Exit	~
Press 'OK' after	Calibration - Higher Temperature Calibration - Lower Temperature Leaves Thick Leaves Woody Stems Exit	

Please note that if you originally received an IM prior to August 2013 you may notice that there is a decrease in the number of recipes available in the standard recipe selector. We continue to test the IM and its recipes, and the list represents those recipes which have been fully tested and can be applied without much additional testing by the user (although we do still encourage you to test the relevant recipe for your sample type). If you wish to access one of the other recipes, please see "Modifying and Creating Recipes" in section **IM Methods and Tips for Running the IM**.

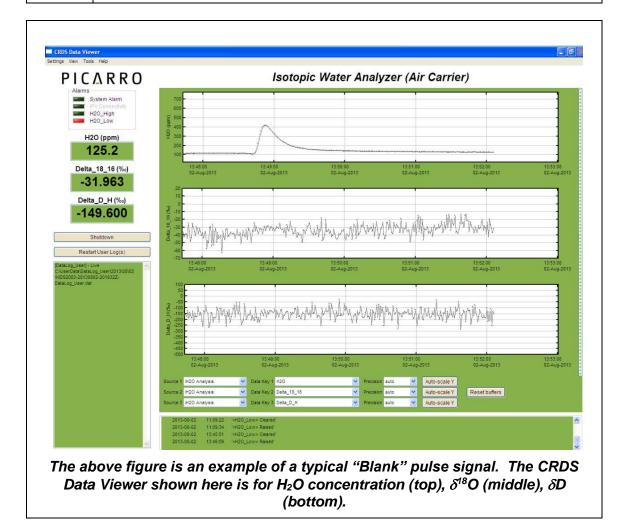
7. The software will then prompt the user to specify whether or not this run is a "Blank" or a "Sample". Blanks are run to correct for errors associated with non-zero baseline along with residual water vapor in the vial headspace. We recommend running at least two blanks before progressing to samples. A "Blank" consists of a capped empty vial. The data from multiple "Blank" runs are averaged and reported in the coordinator output. You do not need to do anything with this blank data, as it is already factored into any samples run in the same coordinator session.

Data Type			×
Current Type	Blank		*
Press 'OK' afte	Blank Sample		
		ОК	

Before running the IM, it is important to check the water concentration baseline. The baseline concentration in ppm can be monitored in the Data Viewer (GUI). When the baseline stabilizes and is no longer decreasing with time, you can start to use the IM. The baseline will vary by instrument and zero air tank gas, but should be below 250 ppm. To speed up the acquisition of this baseline, load an empty vial into the IM. For details on how to load an empty vial, see step 11 of this section. The vial can be loaded as soon as the IM Coordinator has been launched (step 5).



The IM software monitors this baseline, and prevents a sample from being run before this threshold it met, but a time out exists that will allow the software to proceed, regardless of the baseline, after approximately five minutes. This software time out is designed for between sample background monitoring and not the establishment of the overall instrument background. Therefore, for best results, the user should wait until the baseline threshold is obtained when the first setting up the IM, and each time they start running a set of samples.



ΡΙΟΔRΠΟ

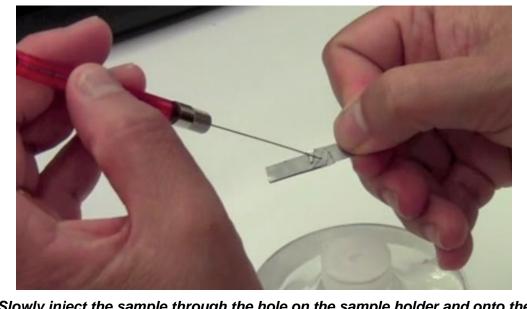
8. After selecting to run a "Blank" or "Sample", you will now be prompted with "Press 'OK' to begin purging...Please prepare the vial while purging." At this point, the IM is being purged with zero air, and there is a 1-minute delay for the user to prepare the sample vial. This controlled delay also helps to minimize timing-dependent errors.



- 9. You can now prepare your sample. Using a filter paper dot and tri-fold metal strip you prepared earlier (step 3), load a water isotope standard. To prevent evaporation, only do this when you have a glass sample vial ready and immediately before you run the sample (preferably the glass vial should be flushed with dry gas before being used, as in "Flushing Sample Vials and Preparing Blanks" in section IM Methods and Tips for Running the IM).
 - a. Measure out 3 μ L of water into the syringe, and then empty the contents of the syringe onto the filter paper through the sample holder hole centered on the filter paper dot. Ensure the water is absorbed by the paper and does not pool on the metal.

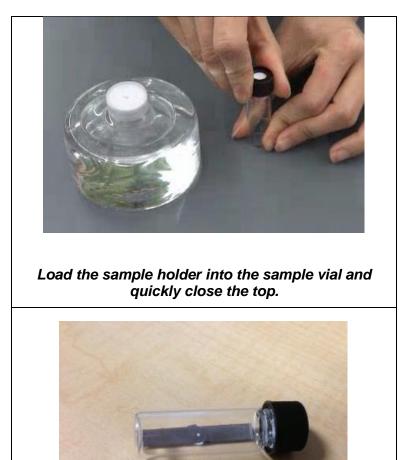


Measure out 3 μ L of water into the syringe.



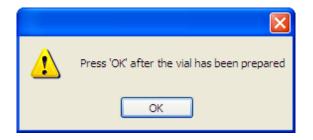
Slowly inject the sample through the hole on the sample holder and onto the filter paper.

b. Insert the prepared sample (tri-folder metal strip, filter paper dot and water) into the sample vial so that sample holder lays flat on the bottom of the vial. Seal the cap (already fitted with a septum) as quickly as possible to prevent loss of water. Each septum is intended for single use, however if one is careful the septa can be reused 3 or 4 times for calibration purposes.



10. After the IM lines have been purged, the software will ask if a sample has been prepared. Assuming you are ready to load a sample, click OK. This will stop air flow to the needle that pierces the septum of the sample vial.

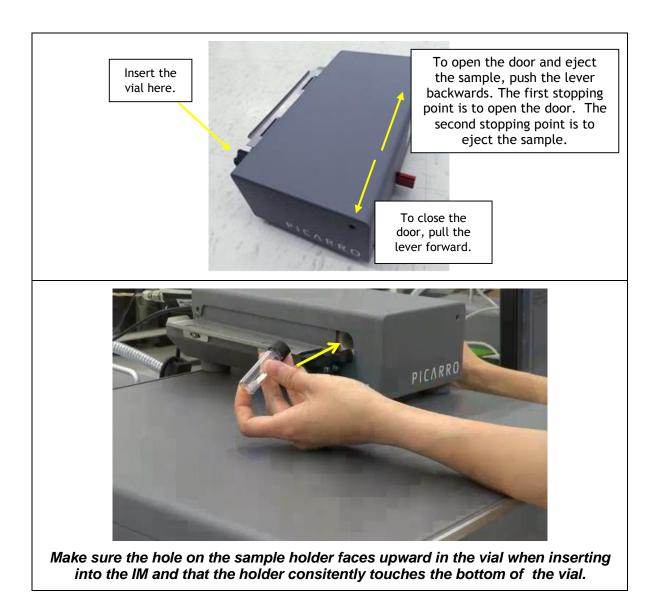
Make sure the sample holder lays flat on the bottom of the vial.



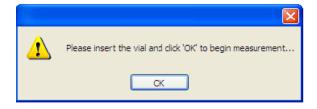
11. The user will now be prompted to insert the sample into the IM and click OK. After which, the heating and measurement process will being. To load the sample into the IM, push the red lever on the right side of the IM backward, and then release it to ensure the door is open. Insert the vial fully into the housing (a slight resistance may be present due to the edge of the cap – lifting the bottom of the vial upwards slightly so that you load the sample at an angle can aid in vial insertion). Push in the vial so that the glass bottom is flush with the edge of the housing. Be sure to hold the unit with the other hand to prevent it from moving while you are inserting the vial. Before closing the door, visually check to see that the sample holder remains flat within the vial and did not move during loading. Close the door by pulling the red lever back to its original position. The red lever should be pulled towards the front to seal the vial inside and to allow the door to close and the sample to heat. There should be an audible click as this happens.



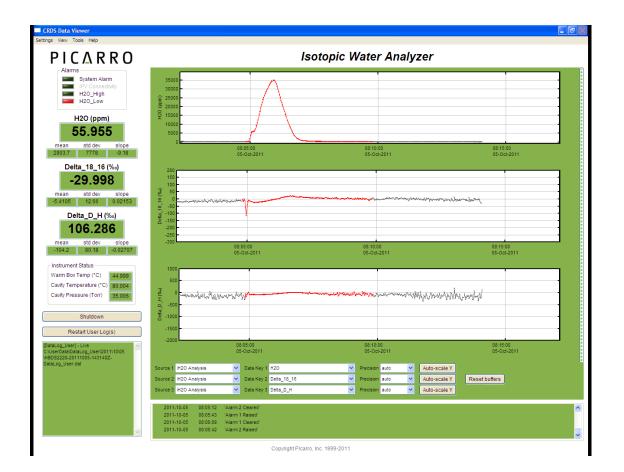
There is a safety interlock on the door of the IM. The induction heater will not be activated unless the red lever is in the operation position (lever is at its forward-most position). If the door does not close, air will still flow through the vial and the analyzer may detect some residual water vapor from the sample even if it is not heating.



12. Once the sample vial has been inserted and the door of the IM is closed, click OK.



13. Heating will now begin. The full cycle of one measurement is defined by the recipe that is being used and typically takes a few minutes to complete. As the heating progresses, the measurement of the "pulse" of water from the sample will be shown on the analyzer GUI:





When you first run the IM, observe the flow rate and target 140 sccm during the measurement cycle. Adjust the regulator as necessary to obtain a flow rate of 140 sccm.

14. After the pulse has been analyzed by the software, the section used for the analysis will be indicated in red on the Data Viewer screen (see image above). The resulting values will also then become visible in the Coordinator window:

New output file							Run Sample Number					
Filename	Filename HIDS2083_IsoWater_201306						Load Sample Descriptions		2	Chan	Change Septum	
Sample		Resistance 99999.9	Start Time	End Time 2013-08-20	Recipe	Sample Type	Description	Delta O18	Deita D -127.383	Est. H2O V	and the second second second	
	176	33333.3	2015-00-20	2013-00-20	Calbrador	Didlik 1		-21,300	-127.303	7.553	4720814	15822
					Recipe S	olection		Ĩ	X			
						Recipe Calibrat	ion - Lower Ten	nperature				
<						K' after selecting			2 2			3
								Ок				
	ecting data. ting sample :											1
Valve 2 ON												
	volume in mi	croliters of	Water Extra	acted: 7.55								
Run count= Resistance												
99999.90												
Sample Descrip	tion (select or ent	er new descriptio	n)								ſ	Injected

If you were running a blank, you will also be prompted to choose whether you want to include it or discard it:

ption	Include	~	
	Indude		
lease c	Discard	e e	ewer' window and choose whether to include this run in the blank aver

15. After measurement of the sample is complete, the user will be prompted to remove the sample vial as below. To remove the vial, push the red lever gently backwards – this drops the door. Then push the red lever backwards again to eject the sample vial.





Exercise caution when removing the vial from the IM. It may be hot. Do not put your directly fingers into the IM sample chamber when there is no vial present as the needle can pierce skin.



If the vial is stuck inside the IM with the door still closed, do not push hard on the lever to open the door. This means the vial has been pushed against the door and cannot be ejected. In this case, push against the door so that the vial goes back in. Then lightly push the lever towards the back so the door drops, and eject the vial.

16. You are now ready to run another sample, and the recipe selection box will pop up again (step 6). To re-run the same calibration water, be sure to re-use the same filter paper dot and sample holder.



Due to filter paper memory, it is essential to re-use the same filter paper when running calibration standards (or any other liquid sample) in replicate. The first analysis will always be affected by absorption of atmospheric water vapor to the filter paper and therefore will not reflect the true isotopic composition of the sample. We recommend running at least four replicates of each liquid sample introduce when using glass filter paper, with the first one or two discarded. More are needed if running cellulose filter paper (see section IM Methods and Tips for Running the IM).

IM COORDINATOR DATA COLUMNS

Within the Coordinator, data will be displayed under the following headings:

- 1. Sample: Sample number for the current Coordinator session.
- 2. Lifetime Cycle: Cumulative number of samples the IM has run.
- 3. **Resistance (Ohms):** Resistance of the Micro Combustion Module.
- 4. **Start Time**: Time the sample started heating.
- 5. End Time: Time the sample finished heating.
- 6. Recipe: User-selected recipe used to run the sample.
- 7. **Sample Type**: Lists if the sample was a blank. The blanks are numbered when included in the average, and labeled without a number when they are excluded from the average.
- 8. **Description**: Sample description, as entered by the user.
- 9. Delta 018: Oxygen isotope ratio as defined by standard delta notation (¹⁸O/¹⁶O):

$$\delta^{18}O = \left[\frac{\binom{^{18}O}{^{/16}O}_{sam}}{\binom{^{18}O}{^{/16}O}_{std}} - 1\right] \times 1000$$

- 10. dD: Hydrogen isotope ratio as defined by standard delta notation (D/H)
- 11. Est. H2O Volume (microliters): The estimated μL volume of water in the sample. This value is calculated by assuming the flow rate during an IM run is 140 sccm. If the flow rate is higher (lower) than this, it will be underestimated (overestimated). This coordinator output is useful to ensure your samples yield approximately the same amount of water as your standards.
- 12. Integrated H2O Sig: The integral for the curve measuring total water. Units are in ppm.
- 13. Maximum H2O (ppm): The maximum value of water vapor during the pulse. Units are ppm.
- 14. Spectroscopic
- 15. **Baseline Shift**: The integrated baseline spectroscopic signal, normalized to the H₂O signal. Elevated levels correlate to large alcohols in the sample, and may be representative of contamination.
- 16. **H2O18 Sig**: The integrated area of the H₂¹⁸O spectral peak (this corresponds to the Galpeak_77_INT in the L2120-i coordinator).
- 17. **H2O Sig**: The integrated area of the H₂¹⁶O spectral peak (this corresponds to the Spline_max_INT in the L2120-i coordinator).
- 18. **HOD Sig**: The integrated area of the HD¹⁶O spectral peak (this corresponds to the Galpeak_82_INT in the L2120-i coordinator).

There are additional columns for the blanks run. These values are used to calculate the average blank signature and these values are used to correct the remainder of the data compiled in the same coordinator file.

Data collected by the Coordinator can be found in CSV format within the folder: C:\lsotopeData.

TUNING IM DATA PULSE SHAPE

The IM should perform well when samples give appropriate pulse shapes. A desirable pulse shape is one which has a peak in the 10,000-30,000 range and which is broad enough that most of the pulse duration is occupied by a portion of the pulse above ~2000 ppmv.

There are generally two types of pathological pulses: Fast and Slow pulse.

1) FAST PULSE

Tall, narrow pulses are generally undesirable because most of the spectroscopy is taken during a period of rapid concentration change and because the ringdowns during the tail end of pulse are taken when the concentration is too dry. Pulses with this signature can be called fast pulses. A fast pulse can be slowed down in two ways:

1. Reduce the heating by adjusting the recipe.

2) SLOW PULSE

Wide, shallow pulses are generally undesirable because most of the spectroscopy is taken with low concentrations and in certain circumstances the sample may not be completely dried out before the end of pulse. Pulses with this signature can be called slow pulses. A slow pulse can be sped up in two ways:

1. Increase the heating by adjusting the recipe.

PULSE SHAPES OF DIFFERENT TYPES OF SAMPLES

- **DRY SAMPLES:** In certain circumstances the amount of water in a sample is too low to get a desirable pulse shape. In this case, we recommend increasing the sample size.
- WET SAMPLES: In other circumstances the amount of water in a sample is too large to get a desirable pulse shape. In this case, we recommend decreasing the sample size.

HEATING AND PULSE SHAPES

The IM inherently introduces a fractionation process (the isotopically lighter isotopologues of H_2O , e.g., $H_2^{16}O$, are converted to water more easily than the heavier isotopologues of H_2O , e.g., $H_2^{18}O$) therefore it is essential to ensure all matrix-bound water is extracted for the accurate determination of the isotopic composition of the sample. To ensure this is occurring, monitor induction coil heating during sample introduction. The peak water concentration should be achieved prior to the heating coil power turning off. You can monitor this in the Coordinator. The heating coil is now longer being supplied with power when you see the following output in the Coordinator log:

"Induction Coil Off

Still collecting data ... "

When you see this output, be sure that the H_2O concentration is already decreasing in the Data Viewer window. If it is not, consider adjusting your recipe heating ramp or time, or altering your sample size (only alter the sample size if the extracted water is below $2\mu L$).

PICARRO SERVICE & MAINTENANCE | INDUCTION MODULE

This chapter contains the following information:

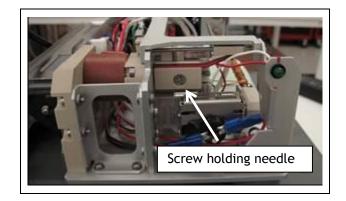
- 1. Needle Replacement Instructions
- 2. Scrubber Cartridge Replacement Instructions
- 3. Micro Combustion Cartridge Replacement Instructions

1. NEEDLE REPLACEMENT INSTRUCTIONS

When maintenance is required for the IM, the Coordinator will prompt the user to either: **change the needle**, or **change the scrubber cartridge** with a message such as that below:

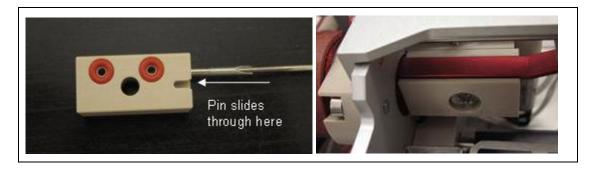
⚠	Estimated lifetime of needle reached. Inspect or replace needle before continue.
	ОК

First ensure that there is no sample vial in the IM. Using a screwdriver, open the lid of the IM box. Be aware that if the machine has been running, the inside of the IM will be hot, especially the Micro Combustion Cartridge and the scrubber cartridge, both of which run at high temperature.



To remove the old needle, first remove the screw holding the needle and then remove the needle by sliding it to the right. The new needle can now be inserted. To avoid leaks, check that the orings have remained in place on the needle block. The back edge of the needle base should be

flush against the valve block (located further towards the back of the machine than the needle). The pin on the valve block should line up with the appropriate place on the needle, and the red extraction arm should be in the groove on the top of the needle base. Note that the right edge of the needle base and the valve block may not line up with each other. Replace the screw that holds the needle in place and close the lid of the IM.

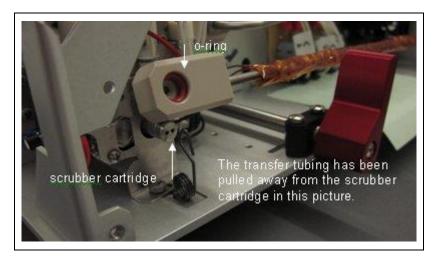


ΡΙΟΔRΟ

2. SCRUBBER CARTRIDGE REPLACEMENT INSTRUCTIONS

When maintenance is required for the IM, the Coordinator will prompt the user to either **change the needle** or **change the scrubber cartridge** with a pop-up message.

Open the lid of the IM. The scrubber cartridge is located as shown in the photo below. Pull the spring back and detach the transfer tubing. If the IM has been running, the cartridge will be extremely hot. Pliers can be used to pull the cartridge out; it should be put aside to cool down before it is handled.



Take the new cartridge and push it into the opening until it is sealed by the o-ring in the back. Be sure to push the new cartridge all the way in when inserting it, otherwise the IM will not be able to transfer the gas to the analyzer. (When placing a new cartridge, the orientation of the two holes in it is not important.)

Before replacing the transfer tubing back over the scrubber cartridge, change the o-ring in the transfer tubing. This can be accomplished by piercing the o-ring with a small screwdriver and pulling it out. Be careful not to damage the other parts of the transfer tubing in the process. Close up the IM when finished.

3. MICRO COMBUSTION CARTRIDGE REPLACEMENT INSTRUCTIONS

Please visit Picarro's Community website to watch the IM cartridge replacement video, or follow the instructions below. A username and password are required to access Community. If you do not have access to the Community, please register on our website or contact support@picarro.com.

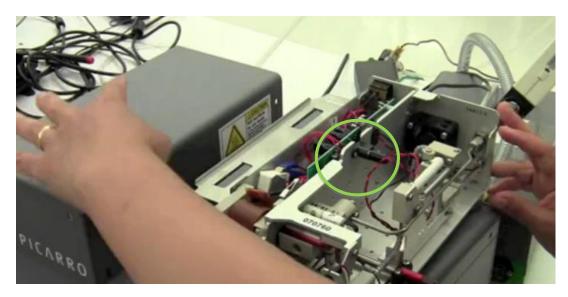
http://www.picarro.com/community/picarro_community/changing_the_im_cartridge

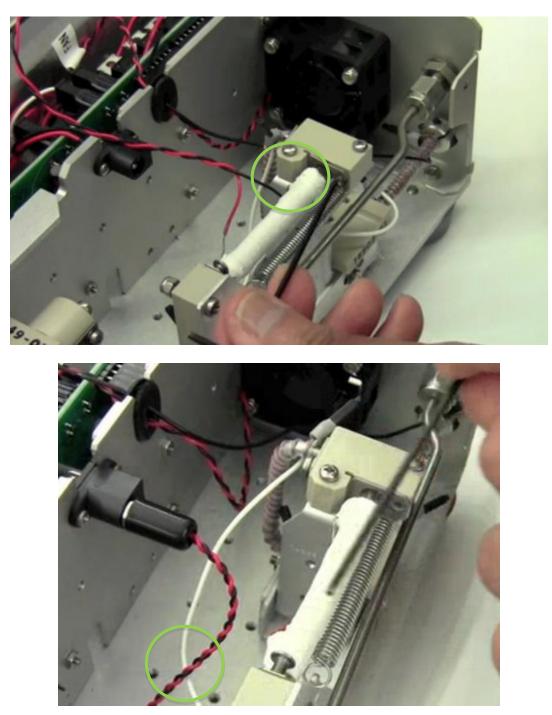
Step 1: Turn off the IM.

Step 2: Remove the cover of the Induction Module.



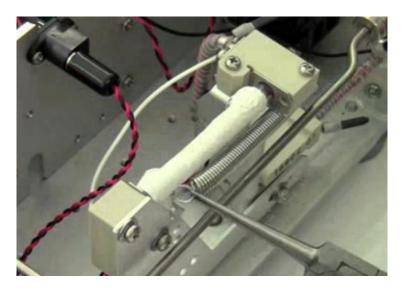
Step 3: Unplug the red-black wire (circled in green).



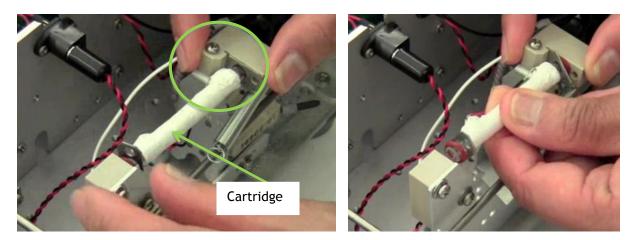


Step 4: Remove the two screws (circled in green) holding up the cartridge.

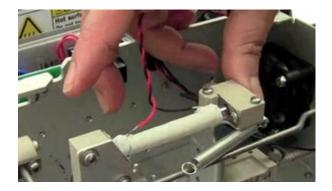
Step 5: Unhook the spring.



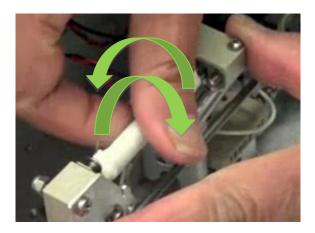
Step 6: Gently push the cartridge holder (circled in green) out and remove the cartridge. Also remove the two o-rings (red) and the red-black wire that are attached to the cartridge.



Step 7: Install the new cartridge with metal plates and two new o-rings. Gently push the cartridge holders inward to secure the cartridge.



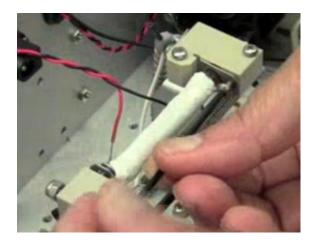
Step 8: Turn the cartridge back and forth to make sure it fits well.



Step 9: Re-hook the spring.

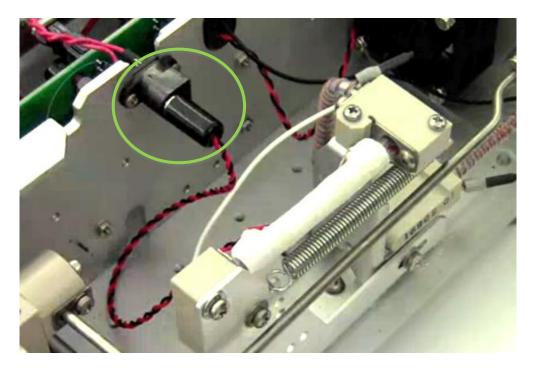


Step 10: Tighten the screws back on to secure the metal holder to the cartridge holder.



ΡΙΟΔRΟ

Step 11: Plug the red-black wire back in (circled in green), which should not push against the cartridge (the cartridge will get very hot during IM operation). Afterwards, replace the cover.



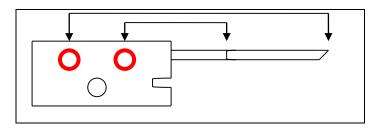
TROUBLESHOOTING | INDUCTION MODULE

The following section lists solutions to a common problem that may be encountered while using the Induction Module. If, after attempting these procedures, the problem remains unresolved, please contact Picarro Customer Service at support@picarro.com. More troubleshooting information is also available at the www.picarro.com. More troubleshooting access to the Community, please register on our website or contact support@picarro.com.

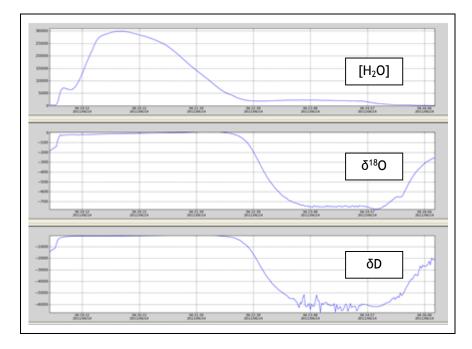
1. Symptom: No communication between IM and software. (Coordinator says, "MBW not found.")

Recommendations: Verify that the USB is connected to the IM and the analyzer. The following steps can be performed to rectify a USB problem:

- Unplug the USB cable from the analyzer and plug it into a different USB port (try one of the ports on the back of the analyzer). This may force a re-recognition of the IM USB device.
- Go to Control Panel → System → Hardware → Device Manager. Right Click on "Ports". Select "Scan for Hardware Changes". This may force a re-recognition of the IM USB device.
- Go to Control Panel → System → Hardware → Device Manager. Click on "Ports" to expand the list of attached Ports. Find the one marked "Arduino UNO" and uninstall. Right Click on "Ports". Select "Scan for Hardware Changes". This will force a reinstallation of the IM USB device driver.
- Reboot the Windows PC. This may force a re-recognition of the IM USB device.
- 2. Symptom: During "sample" stage of Coordinator, gas flow rate is low (approx. 40-50sccm). Recommendations: Verify that the flow rate cannot easily be increased and that the gas pressure is not low. Check the needle for clogs. If the needle is clean, contact Picarro support for further assistance.
- **3. Symptom:** In "ready" or "purge" state, the water concentration does not decrease. **Recommendation**: Make sure the scrubber cartridge is completely inserted in the IM and that the micro combustion cartridge is completely inserted in the IM with appropriate o-rings.
- 4. Symptom: After the sample is inserted and starts heating, the water concentration stays constant at about 14,000 ppm (and the sample is not expected to have this concentration). Recommendation: Verify that the septum did not get dislodged when inserted into the IM, and that the micro combustion cartridge is completely inserted in the IM with appropriate orings.
 - If pieces of septa are lodged in the needle, the pulses will not be delivered to the analyzer. Therefore you need to identify the location of the clog, and try to clean it out. For example, use an air duster or something similar and apply the nozzle to the appropriate hole to blow the piece of septum out. The opening on the far left of the needle (as shown below) corresponds to the opening at the tip of the needle. Similarly, the opening on the right (as shown) corresponds to the opening in the middle of the needle.



- 5. Symptom: Green light of the Induction Module is blinking. Recommendation: This either means that the micro combustion cartridge is making poor contact or is burned out. Replace the cartridge by following the Micro Combustion Cartridge replacement instructions in section Service & Maintenance, Induction Module.
- 6. Symptom: Does your data have an upward curve at the end of the pulse?



Recommendation: When analyzing plant matter in the IM, overheating can occur (this is uncommon if you use the recommended recipes, but given that each plant is different, the user should be aware of this possibility). Overheating the sample can result in the sample turning black or brown (as if it were burned), and this is typically associated with the H₂O concentration pulse shape having an upward curvature towards the end of the pulse (see Data Viewer figure above). Overheating a sample may also affect the hydrogen isotopic composition (δ D), and drive it to decreasing isotopic values. If this happens, monitor the isotopic composition and H₂O concentration peak shape for the next few samples, as it may adversely impact these samples. Also decrease the temperature ramp of the recipe or use a different recipe. For example, if you are using the "Calibration – Higher Temperature" recipe, consider switching to the "Calibration – Lower Temperature" recipe.

7. Symptom: Do you think your system might be leaking?

Recommendation: There are several ways to verify if the system is leaking, the most rigorous of which is to use the analyzer itself. Before proceeding, make sure the IM has a needle installed, an empty vial with cap and septum in the sample chamber, and that the IM is hooked up to a zero air or N_2 tank at 3 psi (samples should only be run on the IM using zero air, but you can use N2 to leak check the system).

Now use Windows Hyperterminal to execute commands to actuate the valves in the IM. In Hyperterminal, use the commands s1, s3, and s4 (one command per line) to activate valves 1, 3, and 4. Over a period of 10 minutes or so the system should dry out to well below 1000ppmv. Over a longer period (e.g., 1 hour or more) the system should continue to dry out and decrease to below 250ppmv. This is the typical background for a leak-free system. If a flow meter is available the system should be flowing ~150 +/-50sccm with the micro combustion cartridge off.

If the system does not achieve low backgrounds (less than 250 ppm), check the following:

- 1. Leaky septum (has the septum collapsed into the vial or is it punctured from overuse?)
- 2. Leaks around micro combustion cartridge o-rings.
- 3. Leaks around the scrubber cartridge tube o-rings.
- 4. Leaks around a mis-seated needle or missing needle o-rings.

If none of these checks resolve the problem, it may be symptomatic of a larger problem. Please contact support for assistance with checking the following:

- 1. Malfunctioning or blocked valves.
- 2. Valves missing o-rings, seated improperly, or not adequately tightened.
- 3. Blocked manifold.
- 4. Miscellaneous blocked tubing.
- 5. Miscellaneous tubing leaking around manifold or other face mount o-ring seat.

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:

- o 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 NEED HELP FROM PICARRO?

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

STEP 1: Visit our popular Community forum! 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. <u>www.picarro.com/community</u>

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 and to interested individuals. Please contact us to request an invitation to community (<u>support@picarro.com</u>).

STEP 2: If you can't find the answer your question in the Community, **please activate the Logmein software before emailing us (see directions 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 be turned off easily by the user.

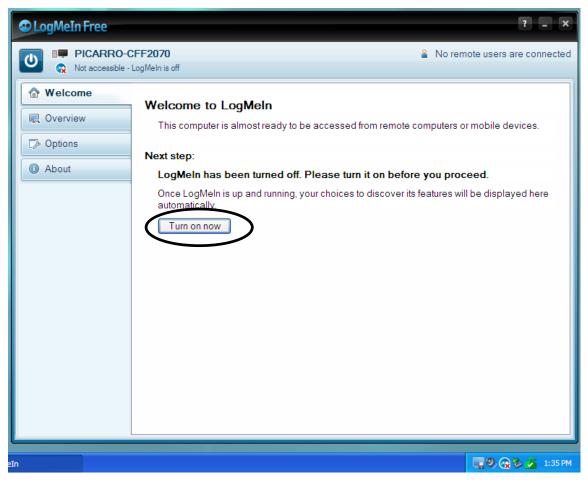
STEP 3: Email us! (<u>support@picarro.com</u>) Please feel free to attach data and/or screen shots to your email that you feel might help us diagnose your problem. They always do! We will get back to you right away! You can also call us at 408.962.3991.

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).

👁 LogMeIn Free			? _ ×
PICARRO-C	FF2070	🔒 No re	emote users are connected
Welcome Overview About	This computer: Accessible via LogMeIn.com Ready and online (for 0 minutes) Turn off Computer name Description LogMeIn account holder's email. Connected remote users: Access my remote computer Show My Computers (opens my LogMeIn account in a we		>
rro\Screenshots 📀 LogMeIn			🛄 🧐 💿 🗞 🏂 1:37 PM

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).