Liaison Interface
For the CM-CRDS

User's Manual

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1. INTRODUCTION

Q: What is a Liaison?

LIAISON is meant as a universal link between the Picarro isotopic CO_2 analyzer, G1101-*I,* and the various commercially available sample preparation systems for Bulk Stable Isotope Analysis (BSIA) of solid and liquid samples through combustion using CM-CRDS or for Total Inorganic/Dissolved Inorganic Carbon (TIC/DIC) isotope analysis in solid or liquid samples through acidification using Automate-CRDS. The basic operation of LIAISON is focused on collecting the CO_2 from combustion or acidification process into a gas sampling bag for isotopic homogeneity purposes. The sample collection duration is 4 minutes at a flow rate of 70-80ml/min. The measurement time from the bag by the CRDS is 8 minutes at a flow rate of ~25ml/min followed by 2 minutes of carrier gas baseline, a total of 10 minutes. The main component of the Liaison interface is a 6-port 6-position rotary valve with a customized rotor disc to allow three parallel operations:

1. Collection of sample from front-end in one bag.

2. Measurement of previously collected sample by CRDS from a second bag.

3. Purging and evacuation of a 3rd bag in preparation for the next sample collection from the front-end.

The three above operations take place in parallel within duration of 10 min. The bag size is 0.5 L.

LIAISON is designed for everyday use and maintenance by laboratory technicians with limited training. The Liaison Coordinator, a python platform-based software module, serves as an interface for the end-user to input the number of samples intended to analyze and the sample description and for the analysis data to be reported in real time.

DIAGRAM OF A LIASION

2. PICARRO LIASION INTERFACE QUICK START GUIDE

2.1. Facility Preparation and Supplies Not Included with CM-CRDS Purchase

2.1.1. FACILITY PREPARATION

A. **Space requirements**: 3 × 3 × 3 feet (0.9 × 0.9 × 0.9 m, L × W × H)

B. **Power Requirements**: 110V/220V for the Liaison. 220V power supply for the CM Unit. If the rating of the site is 100V, a step-up transformer (110V to 220V, 1000 Watts) is required.

C. **Gas cylinder security**. Chains or cage to securely hold gas cylinders (nitrogen and oxygen) in place. Be sure the set-up meets safety requirements as outlined by the gas supplier as well as the health and safety codes of the install site.

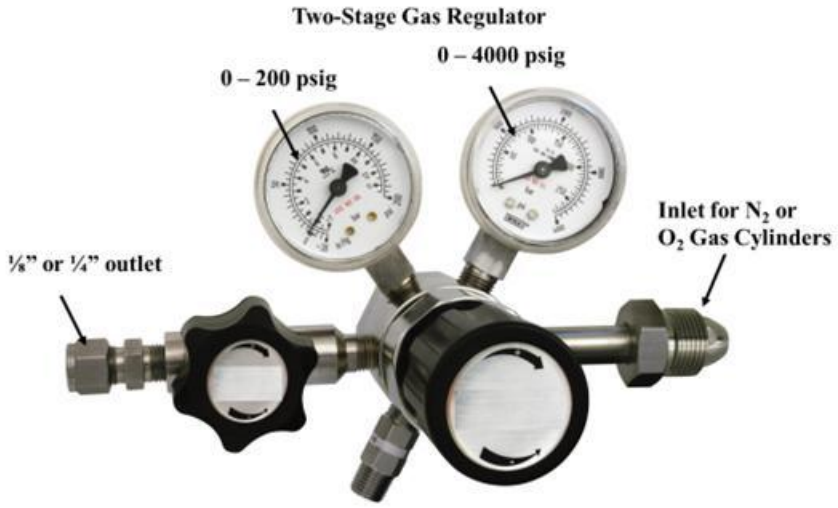
2.1.2. GAS

A. **Ultra-High Purity (UHP) Nitrogen supply**. If the Nitrogen dry gas is supplied from a high-pressure cylinder, a dual stage regulator is recommended to bring the pressure down to the required 35 psi. If an internal Nitrogen gas supply system is to be used, be sure facilities managers supply the appropriate pressure regulator.

B. **High Purity Oxygen tank with regulator**. If the oxygen gas is supplied from a high-pressure cylinder, a dual stage regulator is recommended to bring the pressure down to the required 35 psi.

   a. **Cylinders of Ultra High Purity (UHP) grade oxygen (O_2) and nitrogen (N_2) gases.** Cylinders should be of a pressure of ~3000 psi to ensure long-term supply. Determine a secure location to place the cylinders (check with your Health and Safety department when doing this). It is most convenient if they are placed in close proximity (< 10 feet, 3 meters) of the analyzer. **Order info:** Praxair (a1), Air Liquid (b2) OTHER SUPPLIERS.

   b. **Two-stage pressure regulators for the gases**. These should be rated for 0 – 4000 psig (inlet) and 0 – 200 psig (outlet). Verify with your supplier that the threading of the inlet fitting is the appropriate type as some gas cylinders differ. The outlet tube fitting should be ⅛", although a ¼" fitting can also be appropriate. Order one two-stage regulator for each gas. **Order info** (example models): Praxair (aa11), Air Liquide (bb22), Fisher Scientific (cc33), VWR (dd44).



2.1.3. OTHER PARTS

A. **1/8” diameter metal tubing** (stainless steel) for gas delivery.

   a. The length will depend on the distance between the analyzer and gas supply. Measure the distance between the location of the gas cylinders and where the analyzer will be placed, and add to this distance least 5 additional feet (you can’t have enough tubing!). **Order info:** Swagelok SS-T2-S-028-20

B. **Swagelok connections** to supply N_2 dry gas to the Combustion Module and Liaison Interface.

   a. ¼” to ⅛” stainless steel adapter with ¼” connector. For use only if you purchased a two-stage regulator with a ¼” outlet. **Order Info:** Swagelok SS-400-6-2

   b. Stainless steel cross (4-way) connector for 1/8” tubing. The main nitrogen gas line will be split in three ways (to the Liaison, to the CM carrier gas and the CM valve control). This connector splits the flow while minimizing any chance of additional leaks. **Order info:** Swagelok SS-200-4

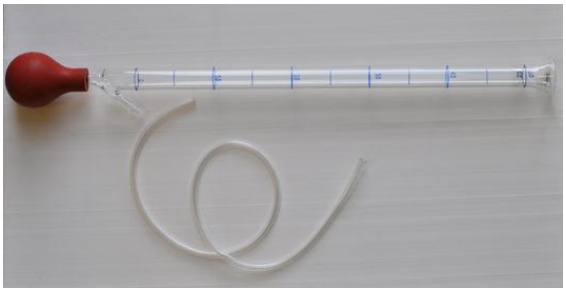
   c. Reducer to go from 1/8” to 3 mm tubing. The supply of N_2 to the CM pneumatic valve control is delivered via a 3 mm outside diameter (o.d.) plastic tube. This adapter allows you to easily supply this flow from the

cross connector. **Order Info:** Swagelok SS-3M0-R-2

d. **Reducer to go from 1/8" to 2mm tubing.** The supply of N_2 carrier gas to the CM unit is through 2 mm o.d. stainless steel tubing. This reducer looks identical to piece **1.1.6**, but with a 2mm inlet. It will allow you easily supply this flow from the cross connector**. Order Info:** Swagelok SS-2M0-R-2

e. **Stainless Steel Nuts and Ferrules for 1/8" tubing**. Spare nuts and ferrules are invaluable, be sure to order an additional set of ten. **Order info:** Swagelok (SS-200-SET and SS-202-1).

C. **Flow meter** to adjust the output N_2 gas flow from the metering valve and Combustion Module to 75-80 ml/min (note: a bubble meter is supplied with the order).



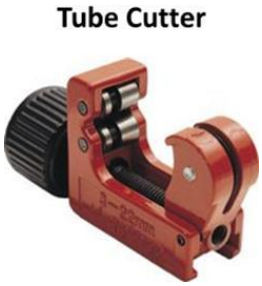
D. **Snoop** for rapid detection of leaks in Swagelok connections (note: a 1:1 solution of Methanol: Water in a squirt bottle can also be used).

   a. Use this to test gas leaks in any connections upstream the Liaison. Leaks will at the least increase your gas consumption, and at the worst pose a health and safety hazard by displacing ambient air and reducing O_2 to toxic levels or even pose a potential explosion hazard.

E. **Optional:** 1/4” Bev-A-Line or Teflon tubing to connect the Liaison exhaust port on the back panel and channel the effluent gas away. It also helps to minimize the pump noise.

2.1.4. TOOLS

A. **Stainless steel tube cutter**. Tubing will need to be cut and this tool is invaluable.



B. **Scissors** or **knife**. Plastic tubing will need to be cut.

C. **Wrenches**. (Get the specific sizes)

D. **Screwdrivers**. (Get the specific size)

2.2. List of Parts Included with CM-CRDS Purchase

2.2.1 CABLES INCLUDED WITH THE ORDER

A. **Power cables** to deliver power to the Combustion Module, Liaison, CRDS analyzer and pump. Ensure that the power rating of the install site was specified at the time of the order so the correct plugs are delivered.

B. **DB-15 Male-to-Female cable** to connect the “Solenoid Valves” connector on the Liaison back panel to the DB-15 female port on the top right hand side of the CRDS DAS. This is labeled with “Liaison Sol. Val.”

C. **DB-9 Male-to-Female cable** to connect the “Rotary Valve” connector on Liaison’s back panel to the “COM2 MPV” on the CRDS back panel.

D. **DB-25 to DB-9 cable** to connect the “Contact Closure” connector on the Liaison back panel to the “CN1” port on the Costech analyzer back panel.

2.2.2. OTHER PARTS INCLUDED WITH THE ORDER

A. **Gas sampling bags** (3) attached to the bag retainers + one extra gas sampling bag.

B. **Transfer tube** (1/8" o.d. × 1 foot long, necessary fittings are included) to connect the Liaison "Output CRDS" port to the CRDS "Inlet" port. Both ports are located on the back panels of the Liaison interface and the CRDS, respectively.

C. **Swagelok metering valve**. This regulates the flow of N_2 into the Liaison. The metering valve has a directional flow. The INLET of the metering valve is marked as *IN*, and should be connected to an N_2 line and adjusted to deliver *75-80 ml/min flow rate*. Then, the OUTLET of the metering valve, marked as *OUT*, will be connected to the "N_2 Inlet" port on the Liaison back panel.

D. **Swagelok 1/8”- 2mm female-female nut convertor**. The nut and ferrule should be used to connect the 2mm SS-tubing from the Costech analyzer to the “*Sample Inlet*” port on the Liaison front panel.

E. **1/16” and 2 mm stainless steel metal tubing.** The smaller diameter tubing is used for the Combustion module gas connections.

F. **Fittings and ferrules for 1/16” and 2 mm tubing**.

G. **3 mm polyethylene tubing.** This tubing is used for the gas delivery to the pneumatic valve control of the CM unit. The tubing is blue to distinguish it from Teflon, which is clear.

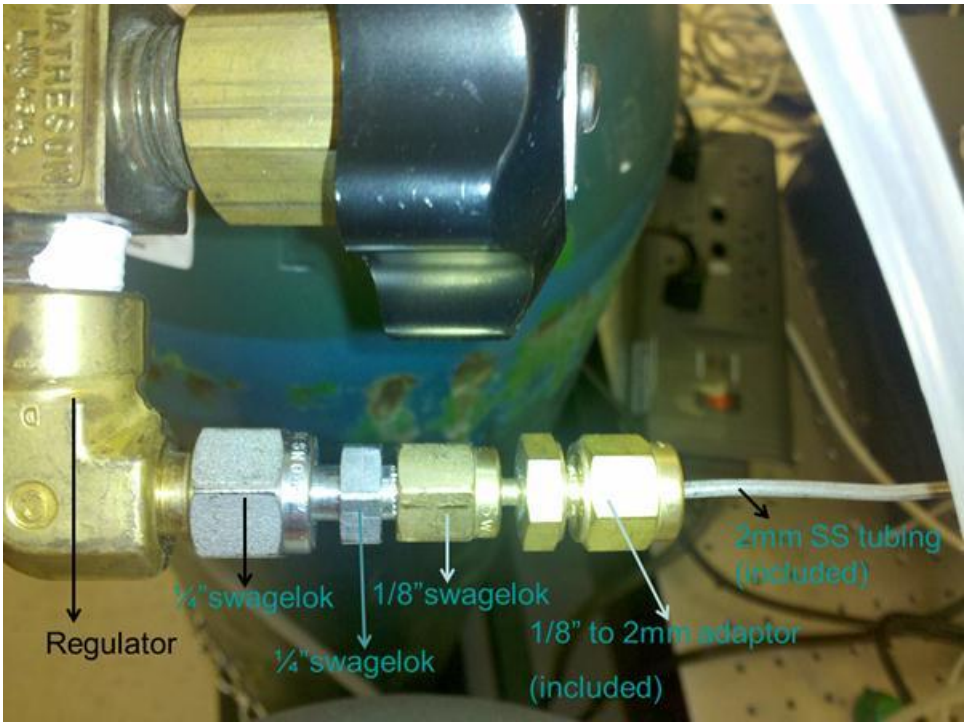
2.3. Set-up Instructions

Be sure to record all necessary set-up steps in **the *CM-CRDS Installation Qualification*** document checklist provided. Also refer back to the ***Combustion Module User's Manual*** and ***Picarro G2121-i manual*** wherever needed. If instructions are unclear, or support needed, contact support@picarro.com

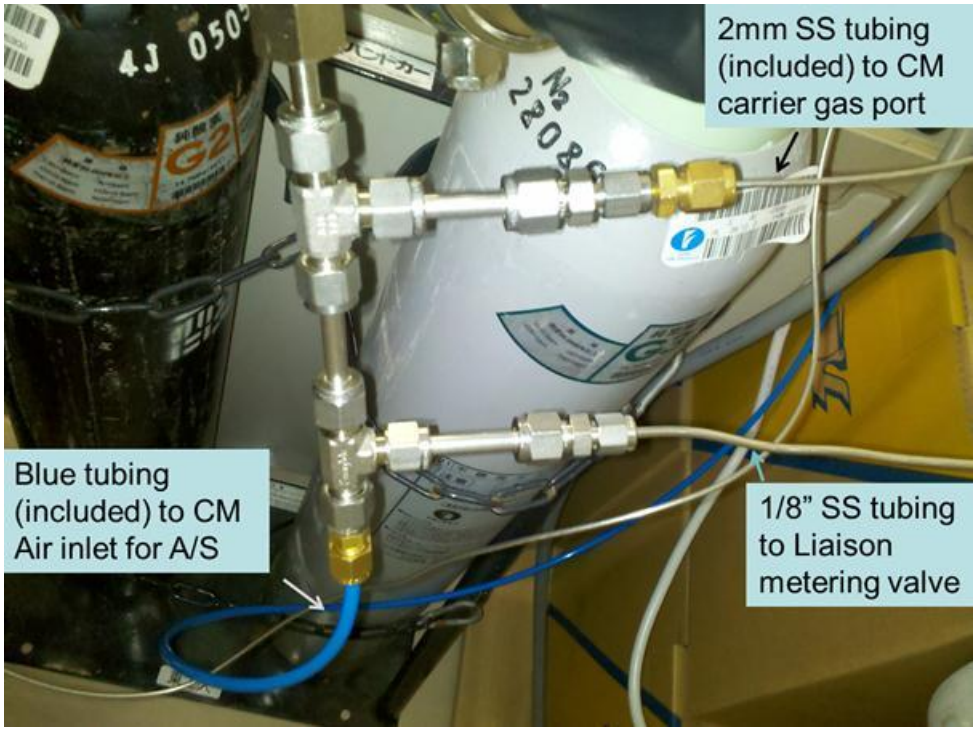
2.3.1. Verify facility preparation is ready, and all parts are included.

2.3.2. Setup gas delivery lines and connections to deliver N_2 to the Liaison unit and N_2 and O_2 to the CM unit. Refer to the ***Combustion Module user's manual*** or the **Automate Instructions Manual** and refer to the Instructions and images below for details.

Image Below: Oxygen Tank after Regulator



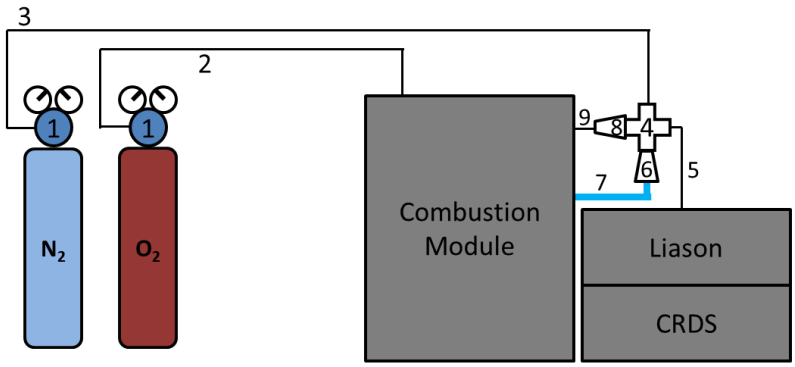
Below: N2 Tank (Configuration after Regulator)



A. Read all instruction manuals, including those of products not purchased from Picarro. The assembly is to ensure the proper delivery of high pressure gases and if care is not used or equipment used in an improper manner injury may result.

B. Note on measuring tubing. Often times, tubing will not be connected via a straight-line from one fitting to another (especially over large distances). In some cases a right angle must be made, or lines run across a ceiling, etc. So whenever a step calls for "measure the distance between", take these winding turns and angles into account. Also, always cut a piece a little bit longer than expected to allow for some degree of slack, and it is always easier to deal with too much tubing than it is to deal with tubing that is too short.

C. Placement. Make sure the gas cylinders and CM-CRDS analyzer are placed in their permanent location. Place the CM-CRDS unit in such a way as to allow at least 4" of space between the back of the system and any obstructions (e.g., wall, other instrumentation). This will be enough space for all connectors, installation and troubleshooting potential problems. At this stage it is OK to proceed with the connections between the CRDS and its pump (see main instruction manual). A rough schematic of how the connections flow is shown:



1 – Two-stage gas pressure regulators
2 – Stainless steel (SST) tubing (1/8" o.d.) line for O_2 gas delivery to Combustion Module.
3 – SST tubing (1/8" o.d.) line for N_2 gas delivery to Combustion Module and Liason.
4 – Four-way SST connector for 1/8" SST tubing.
5 – SST tubing (1/8" o.d.) for delivery of N_2 carrier gas to Liason
6 – Adapter to go from 1/8" o.d. tubing to 3/8" o.d. tubing
7 – Plastic tubing (3/8" o.d.) for Combustion Unit valve control (supplied with unit)
8 – Adapter to go from 1/8" o.d. to 2 mm o.d. tubing
9 – SST tubing (2 mm o.d.) for delivery of N_2 carrier gas to Combustion Unit

D. Installing the regulators. When ready to start the installation, connect the two-stage regulators to the gas cylinders. Make sure the regulators are screwed in tightly. Leak-check the connection between the cylinder and the regulator. This is done by tightly shutting off the outlet valve (the knob that is furthest from the cylinder, ON and OFF are usually indicated by arrows). Carefully turn on the flow of gas by slowly turning the knob on top of the cylinder. If you hear a steady hiss, immediately turn off the main flow. Check to be sure the outlet valve is properly shut and the regular properly tightened into place. If there is no hissing sound, apply small amounts of Snoop to all connections (keeping a paper towel beneath those connections). If no bubbles form then the connections are correct. If bubbles do appear, then continue to tighten the connection in question. Sometimes undoing the connection and applying one turn of Teflon tape to the threads will help stop a leak, but do not do this unless necessary. If the regulator outlet accommodates a ⅛" tube, and

then proceed to **(D)**. If not, follow **(C)**.

a. **Regulator Outlet.** If the regulators do not accommodate ⅛" tubing, then be sure to connect the adapter that will. The most typical size thread is one that fits ¼"tubing. If this is the case, then attach the short ¼" connector to the outlet, followed by the ¼" to ⅛" adapter.

E. Connect the O_2 cylinder. The O_2 flows into the CM unit via 2 mm stainless steel tubing. It is possible to use 2 mm tubing through the entire length, but this tubing is delicate and can be pinched or cracked at its connections. So be sure the tubing will always be "out of the way" and in a secure location where nothing will touch it. Measure the distance from the regulator to the O_2 inlet at the back of the CM unit. Cut a piece of 2 mm o.d. stainless steel tubing that is slightly longer than this. The tube should be somewhat slack to allow for small movements that come about during regular maintenance (e.g. changing of a gas cylinder), otherwise the line may crack and cause leakage. At the outlet of the regulator, connect the reducer that goes from ⅛" to 2mm **(pg. 7 of this document).** Connect the 2 mm o.d. tube to this outlet, tightening the swage so it remains in place. Place the 2 mm fitting and ferrule **(pg.11 of this document)** at the opposite end of the line. Connect the tubing to the O_2 inlet at the back of the CM unit and tighten.

F. Cross (4-way) connector. Decide where you will want to place your cross (4-way) connector. Consider this the "central" connection as it delivers gas to both the CM and Liaison units. The exact position does not matter, so long as it is in close proximity (≤ 30 cm) to both the CM and Liaison units.

G. Setting the Main N_2 Line. Measure the distance between the N_2 regulator and the cross connector. Cut a piece of ⅛" stainless steel tubing slightly longer than this. Position the cross so its outlets are facing up/down and not towards you. Tightly connect the tube to the regulator and then the cross connector. You will want to have the line coming straight into the cross (i.e., no awkward turns or bends to get it to fit). Now number the four connections from 1 to 4 clockwise, with "1" being the Main Line entry.

H. Setting the Liaison N_2 Line. Flow will go from the connector to the metering valve. So attach the metering valve to the Liaison (do not tighten it into position yet). Measure the distance between the cross connector port 2 (numbered above) and the inlet of the metering valve. Cut a piece of stainless

tubing just slightly longer than this. Tightly connect it to both the Cross and the Metering Valve. Disconnect the metering valve from the Liaison.

I. Setting the CM Pneumatic Valve Line. The inlet for the CM unit pneumatic valve gas line is for 3 mm tubing. Attach the ⅛" to 3 mm adapter **(pg. 6 of this document)** to the cross connector port 3. Measure the distance from the CM inlet to the cross connector port 3 and cut a piece of blue plastic tubing of the appropriate length. Attach the blue plastic tubing to the adapter outlet (do not over tighten) and then to the CM unit pneumatic valve inlet with the appropriate nut.

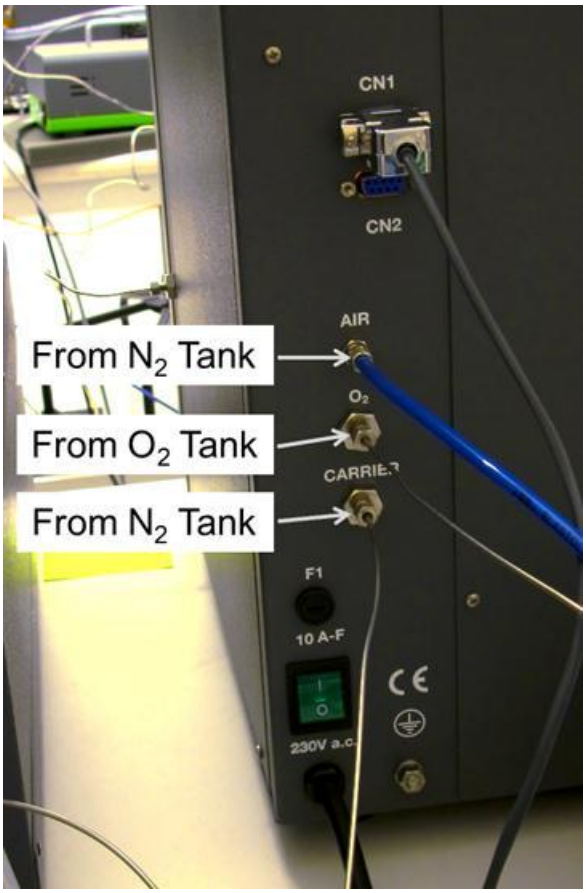
J. Setting the CM Carrier Gas N_2 Line. The inlet to the CM unit carrier gas line is for 2 mm tubing. Attach the ⅛" to 2 mm adapter **(pg. 7 of this document)** to the cross connector port 4. Measure the distance from the cross connector port 4 and the CM unit carrier gas inlet and cut an appropriate length piece of 2 mm stainless steel tubing. Tightly connect the tube to the adapter 2 mm outlet. Place the appropriate fitting and ferrule **(pg. 11 of this document)** over the other end of the tubing and tightly attach it to the CM carrier gas inlet.

K. Checking the O_2 Line. Check the tightness of each of the connections with a wrench. Once this is done, turn on the O_2 main valve and set the pressures to their appropriate setting. Make sure the secondary outlet valve on the regulator is not shut off. Check all O_2 connections with Snoop. If no bubbles form then the connections are OK. If bubbles do form at any connection, then they must be tightened.

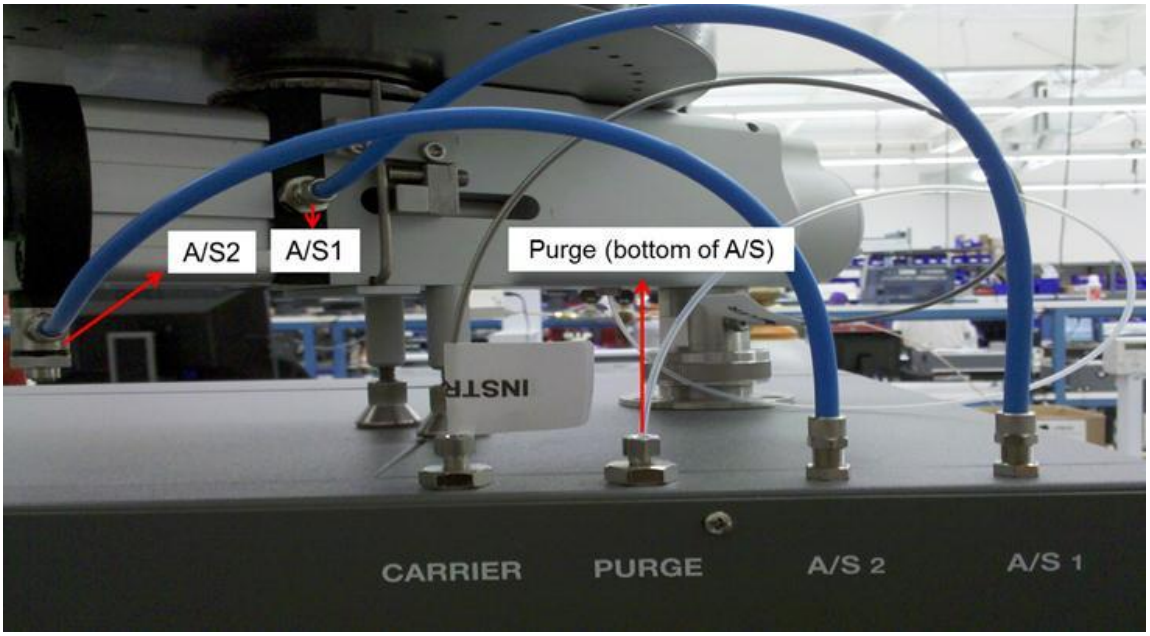
L. Checking the N_2 Line. Repeat **(K)** for all the N_2 connections.

M. When the gas lines are set, and no leaks are evident move on to the full installation of the CM-CRDS unit. Refer to instructions and images below for details. Also refer to the *Combustion Module User's Manual* and the *Picarro G2121-i manual*.

Below: CM Back Panel



Below: CM Back Panel & A/S



2.3.3. Install front-end (Combustion Module) according to the ***Combustion Module users' manual*.**

2.3.4. Install the Picarro isotopic CO_2 analyzer *to the right* of the combustion front-end according to the ***Picarro G2121-i manual*.**

2.3.5. Power up the Picarro CRDS analyzer and pump to allow the CRDS to stabilize while exposed to room air.

2.3.6. While the analyzer is stabilizing, place the Liaison interface on top of the Picarro CRDS analyzer. Make sure the front panel of the Liaison interface is flush with the CRDS analyzer front panel.

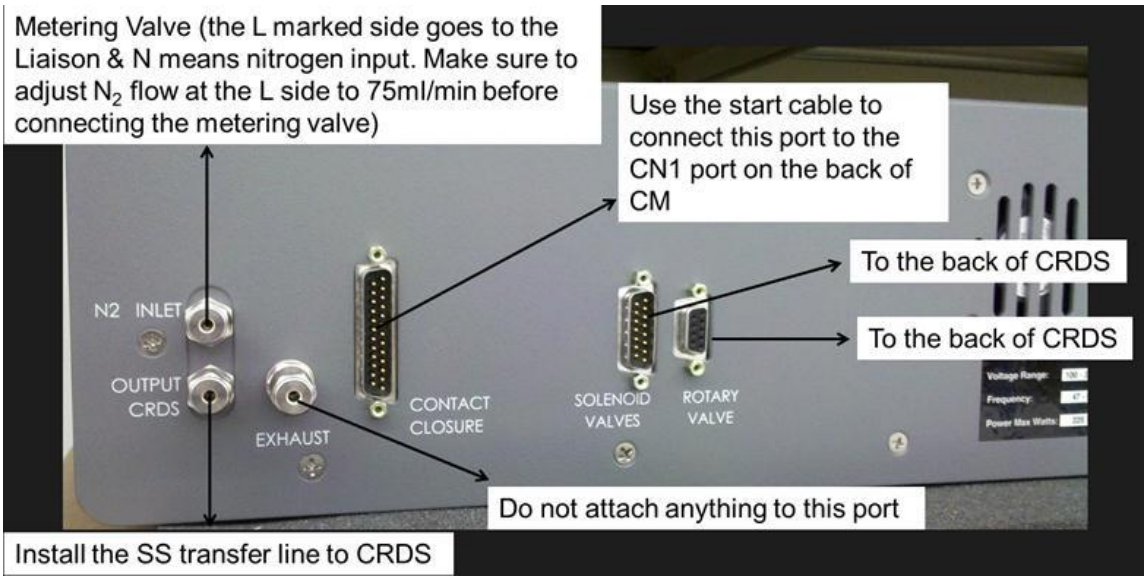
2.3.7. Once the CRDS is stable, connect "Output CRDS" port of the Liaison interface to the "Inlet port" of the CRDS analyzer with the Transfer Tube.

2.3.8. Attach the nitrogen dry gas line to the inlet of the Swagelok metering valve. Please note that the metering valve has a flow direction indicated by the arrow etched on the valve body: IN=> OUT, with N_2 gas flowing IN.

2.3.9. Adjust the metering valve for a flow of 75 – 80 mL/min OUT.

2.3.10. Attach the outlet of the Swagelok metering valve to the "N2 Inlet" port on the Liaison back panel.

Below: Liaison Back Panel



2.3.11. **Optional**: Attach the “Exhaust” port on the Liaison back panel to a hood or outlet.

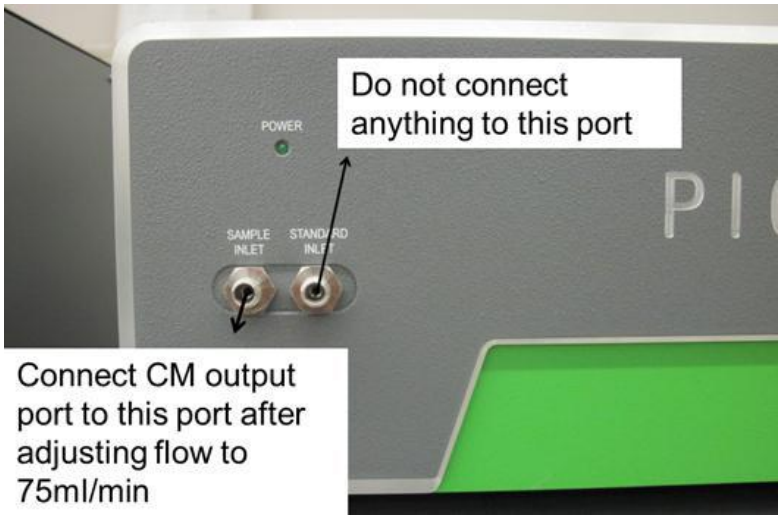
2.3.12. On the back panel of the Liaison Interface, connect the light grey “Liaison Sol. Val.” Cable to the “Solenoid Valves” DB-15 male connector and the other end to the DB-15 female connector located at top right-side of the CRDS DAS back panel.

2.3.13. On the back panel of the Liaison Interface, connect the black “Rotary Valve” cable to the “Rotary Valve” DB-9 Female connector, and the other end to the “COM2 MPV” DB-9 Male connector on the back panel of the CRDS.

2.3.14. On the back panel of the Liaison Interface, connect the dark grey “Contact Closure” cable to the “Contact Closure” Male DB-25 connector, and connect the DB-9 end to the “CN1” DB-9 Male port on the back panel of the Costech Combustion front-end.

2.3.15. Adjust the flow from the CM unit to 75 – 80 mL/min, measured at the 2mm SS-tubing outlet of the unit. Once set, connect the 2mm SS-tubing to the “Sample Inlet” port on the Liaison interface front panel using the 1/8” to 2 mm adapter.

Below: Liasion Front Panel



2.3.16. Keep the “Standard Inlet” port on the Liaison front panel capped as it is not needed for the operation of the interface. **For calibration purposes, it is recommended to use solid standards that will be combusted similar to the samples intended for analysis.**

2.3.17. Connect the power cord to the Power Input Module on the back of the Liaison Panel and switch the Liaison interface on.

2.4. Getting started

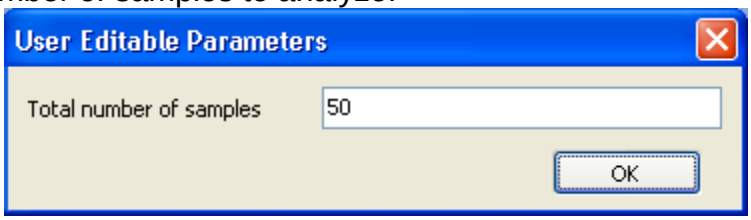
2.4.1. For best results, the sample loaded on the combustion front-end should provide a CO_2 concentration of 2000-4000 ppm, which corresponds roughly to 400 – 1000 µg of carbon. The exact amount of each sample type must be determined independently. Each analyzed sample will be labeled as “good” in the coordinator if it appears as a square pulse with a flat, stable plateau (see Troubleshooting guide below). The isotopic data for samples generating concentration pulses <400ppmv will NOT be analyzed and the data will NOT be reported in the coordinator.

2.4.2. Load the samples you intend to run on the Costech Autosampler and place the CM system in “remote” mode (on the front panel, push “Work” followed by “Enter”, then “Remote” followed by “Enter”).

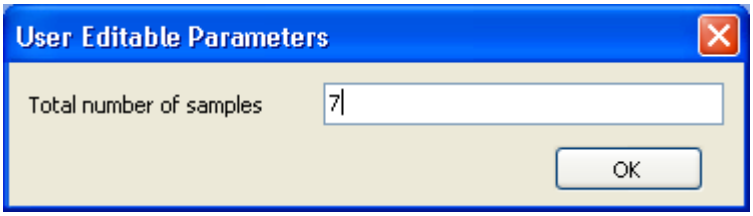
2.4.3. If the CM system is coming out of standby mode, it will take time (up to 45 minutes in some cases) to stabilize. The system is ready to run when the LED lights on the front display are all green.

2.4.4. Double-click on the desktop icon “Liaison Coordinator”.

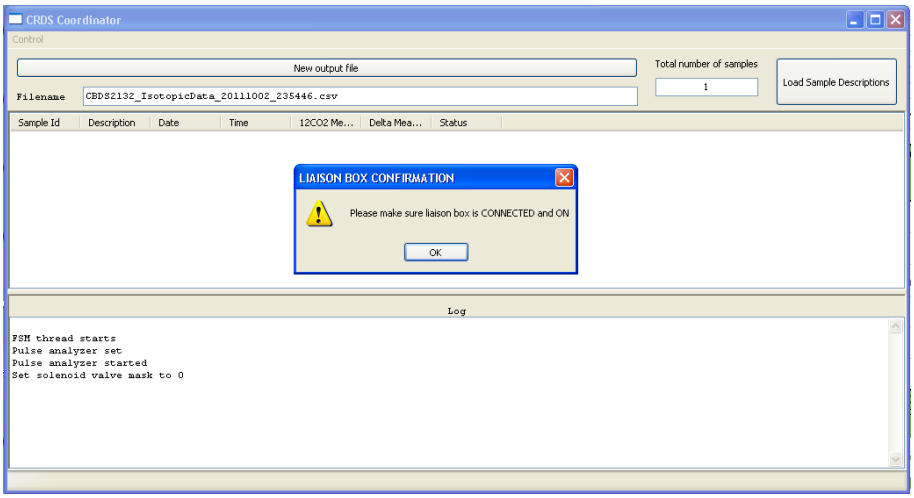
2.4.5. The following pop-up window will show “50” by default as the intended number of samples to analyze:



2.4.6. Input the number of samples you actually intend to run and click ok. An example is provided below:

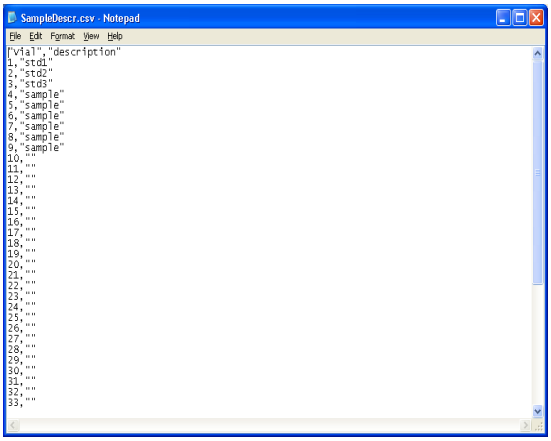


2.4.7. The following window will be displayed. It asks you to make sure that the Liaison interface is connected and turned on:



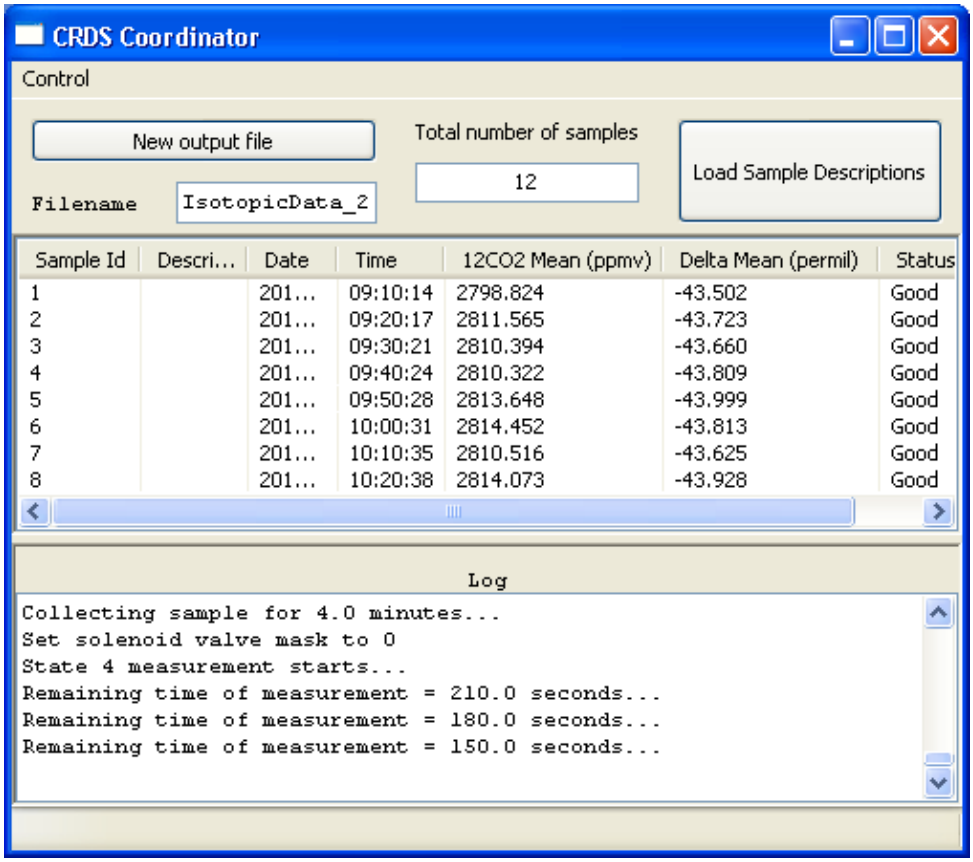
2.4.8. When the three LED lights on the front panel of the CM unit are lit green, click OK. The Liaison interface coordinator will then run a routine to evacuate the three gas sampling bags before starting the sample analysis.

2.4.9. To load the sample description, you need first to enter the sample description in the format provided in the sampledescr.csv file template. The sample description needs to be entered between quotation marks, as shown below:



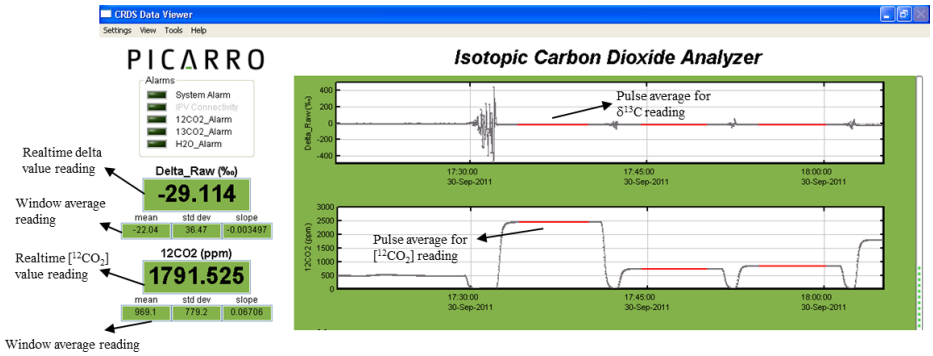
2.4.10. The template file is located in C:\Picarro\Host. You can load the sample description at any point during the analysis by clicking on the “load sample description” button and choosing the relevant sample description file.

2.4.11. As the samples are analyzed, the results will be reported in the upper part of the coordinator as displayed below:

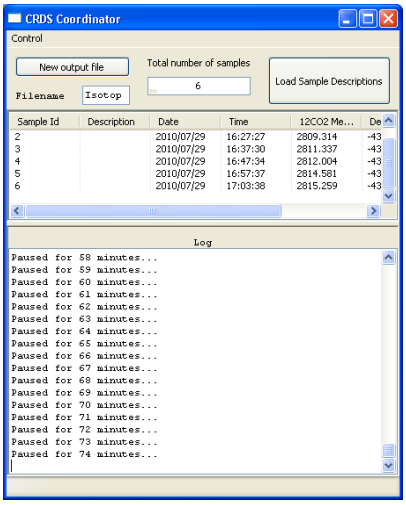


2.4.12. The bottom half of the coordinator window serves as the log/status of all the processes taking place within the Liaison interface during sample analysis. It also displays the error messages when an error occurs.

2.4.13. During sample analysis, it is possible to display two different windows in the GUI ([$^{12}CO_2$] and δ^{13}C_raw) in real-time. The CO_2 generated from each sample analysis is measured from the corresponding gas bag of the Liaison interface where it was collected. The CO_2 signal detected is displayed as a square pulse with the baseline on either end of the CO_2 pulse is generated from the CO_2-free N_2 carrier gas (see the lower display panel, below). The middle section of the plateau of the CO_2 square pulse generated from the sample combustion is averaged and marked with red. The upper panel of the GUI displays the corresponding delta values of the sample square pulse and the middle section is also averaged and marked with red. This red section corresponds to the δ^{13}C value reported in the Liaison Coordinator. The sample-to-sample analysis time is 10 minutes:



2.4.14. When the sample analysis is completed, the coordinator will be paused. The user can then click on the upper right hand side red button of the coordinator window to terminate the coordinator session



2.4.15. The output results file is a *.csv format saved in the C:\Picarro\IsotopicData folder. The file is automatically saved with the naming system: “CxDS2xxx-YYYMMDD.csv” If you wish to analyze the data file before the run is over, do not open a live copy as this can cause some data to be lost. Rather, copy the file and paste it in a new folder, and conduct any data analysis on the copy.

2.4.16. To start a reporting data in a new output file during sample analysis, you can press the “New Output File” button in the Liaison Coordinator at any time during the run.

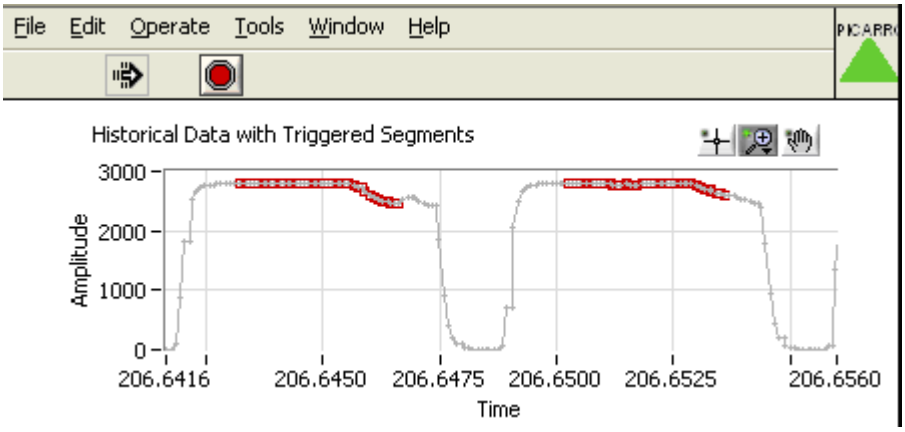
2.4.17. At the end of the run, the CM unit should automatically switch to standby mode after 15 minutes of inactivity.

2.5. Troubleshooting

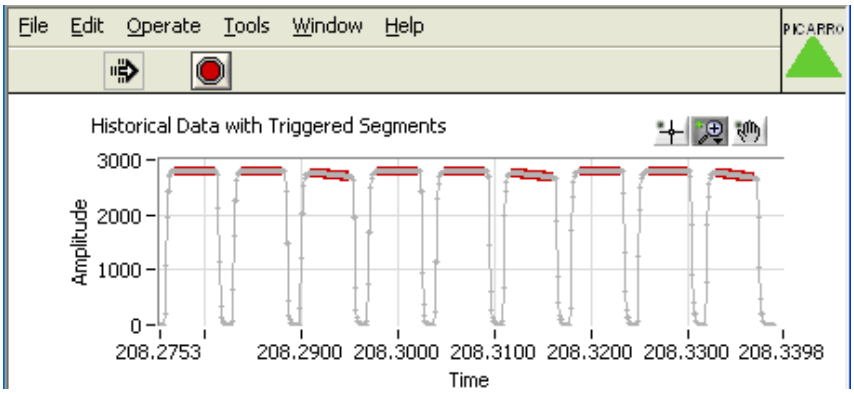
	Please be advised that the window shown below might not be identical to the User Interface version of your analyzer. The purpose, however, is to show how the pulse quality is affected due to leaks in the gas bags.

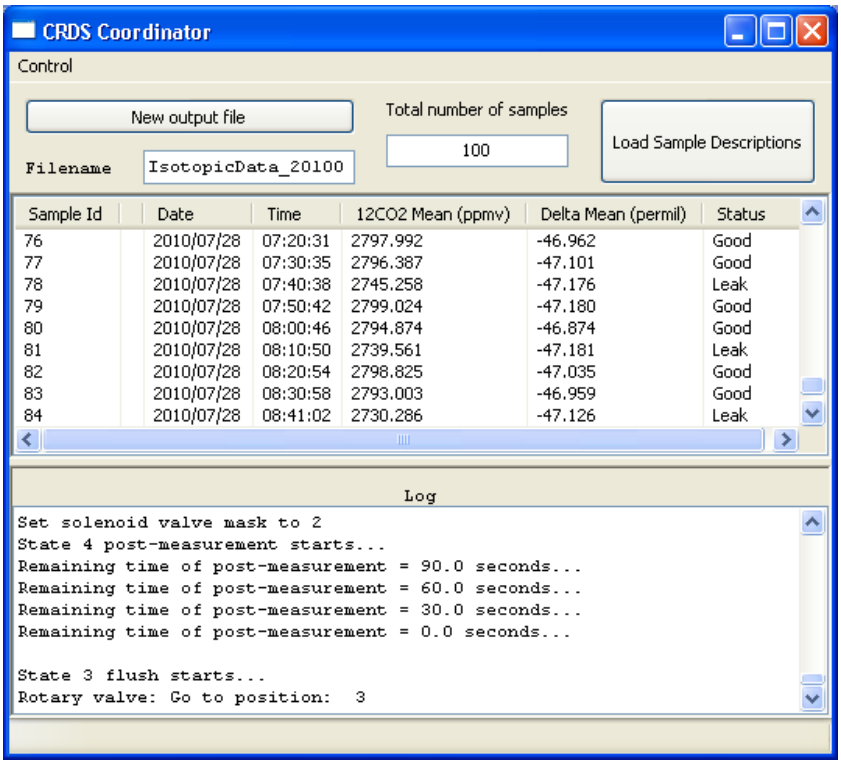
2.5.1. If your case is not shown in the examples below, contact support@picarro.com for assistance in troubleshooting.

2.5.2. The Gas Sampling Bags are consumables that can deteriorate with usage duration. An extra bag has been provided with the Liaison interface for your convenience. (More bags can be ordered directly from Picarro). Any induced wear and tear in one of the gas sampling bags will eventually form a gas leak on the bag surface or seam. The leak will be noticeable from the data quality and the plateau of the CO_2 square pulses as shown below:



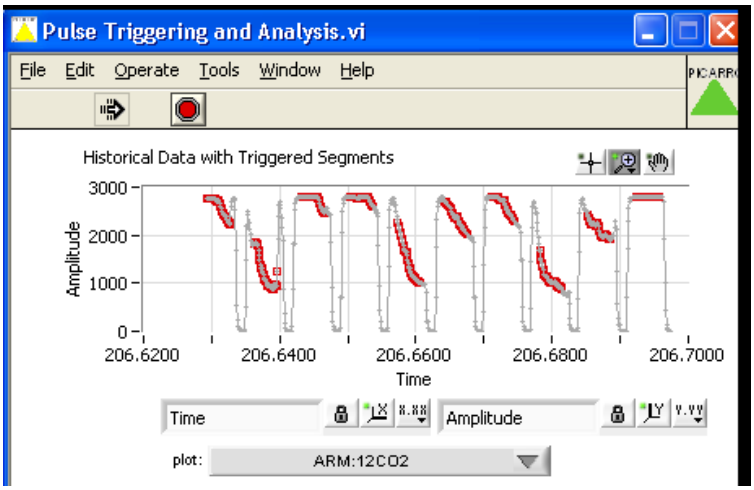
2.5.3. The Liaison coordinator monitors the slope of the plateau of each pulse and reports back in the status column of the coordinator as “good” or “leak” as shown in every third pulse below:





2.5.4. The periodicity in the “leak” pulses, such as the example shown above, indicates that the leak is coming from one specific bag since the three bags sequentially alternate with analyzed samples, as the multi-port valve rotates at the end of each sample analysis. The leaky bag needs to be replaced.

2.5.5. In case where the “plateau” of all the pulses is deteriorating, as shown below, this is indicative of a leak in one of the lines inside the Liaison interface or a line connecting to it.



2.5.6. In this case check the connections between the Liason interface and CRDS analyzer as well as the quick-connect/press-fit connections at the bag valve.

2.6. Shutting down the CM-CRDS System

1. When you're done with your analyses and you want to bring the CM-CRDS system immediately into a standby mode (gas saver mode + standby temperature), you need to press "st-by" then "enter". Another option is to have the CM in "Automatic Standby" mode which puts the CM in standby after 45 minutes of inactivity. Please follow the instructions in the CM manual to enable the "Automatic Standby" mode.

2. Make sure that the Liaison coordinator is shut down by clicking on the red button upper right hand side of the coordinator window.

3. Power down the Liaison interface using the on/off switch on the Liaison back panel.

4. Expose the isoCO_2 CRDS to room air by disconnecting the ¼" female Swagelok fitting at the inlet of the CRDS located on the back panel of the isoCO_2 CRDS analyzer. It is recommended to install a 3-way valve at the CRDS inlet in order to easily switch between room air necessary for Standby mode and to the N_2 flow through the transfer line from the Liaison to the CRDS when in Work mode.

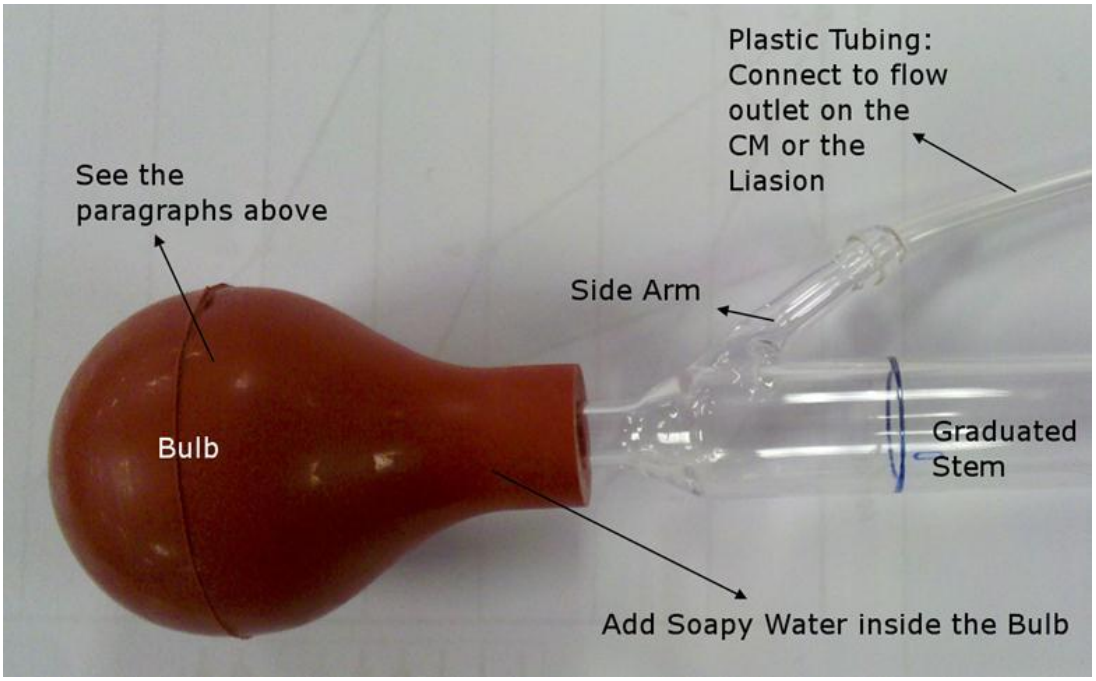
3. PICARRO LIASION INTERFACE ADDITIONAL GUIDE

3.1. HOW TO USE A BUBBLE FLOWMETER TO MEASURE THE GAS FLOW RATE

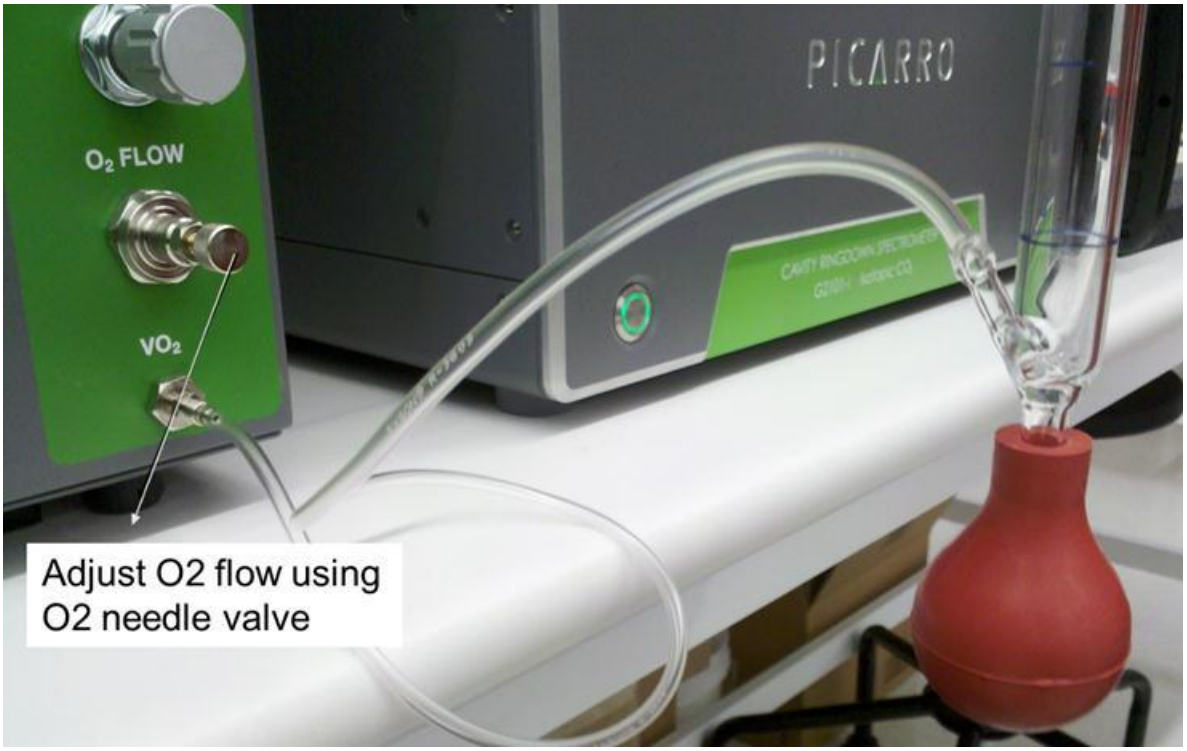
3.1.1. Bubble Flow-meter Connection.

After adding the soapy water inside the bulb, pump the bulb to form bubbles. Then, use a hand watch or a timer to measure the time it takes for the bubble to move up between two circles (10ml) inside the graduated stem.

Divide the traveled volume by the measured time to get the gas flow rate



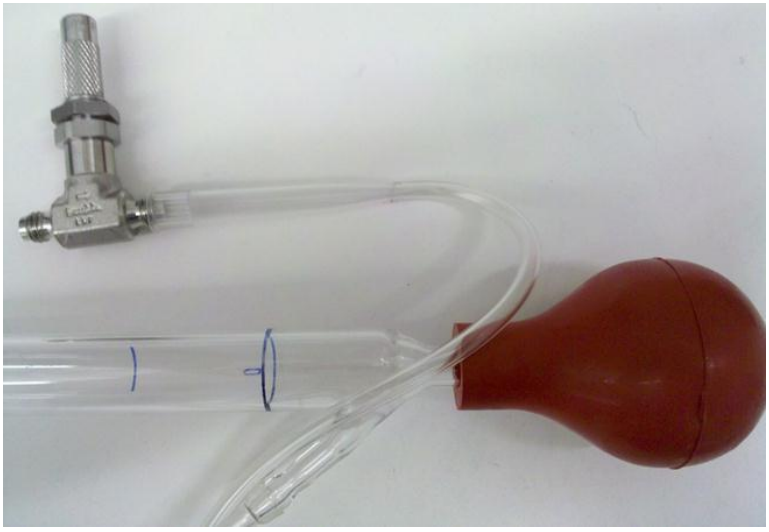
3.1.2. Connect to the VO2 Vent (30ml/min).



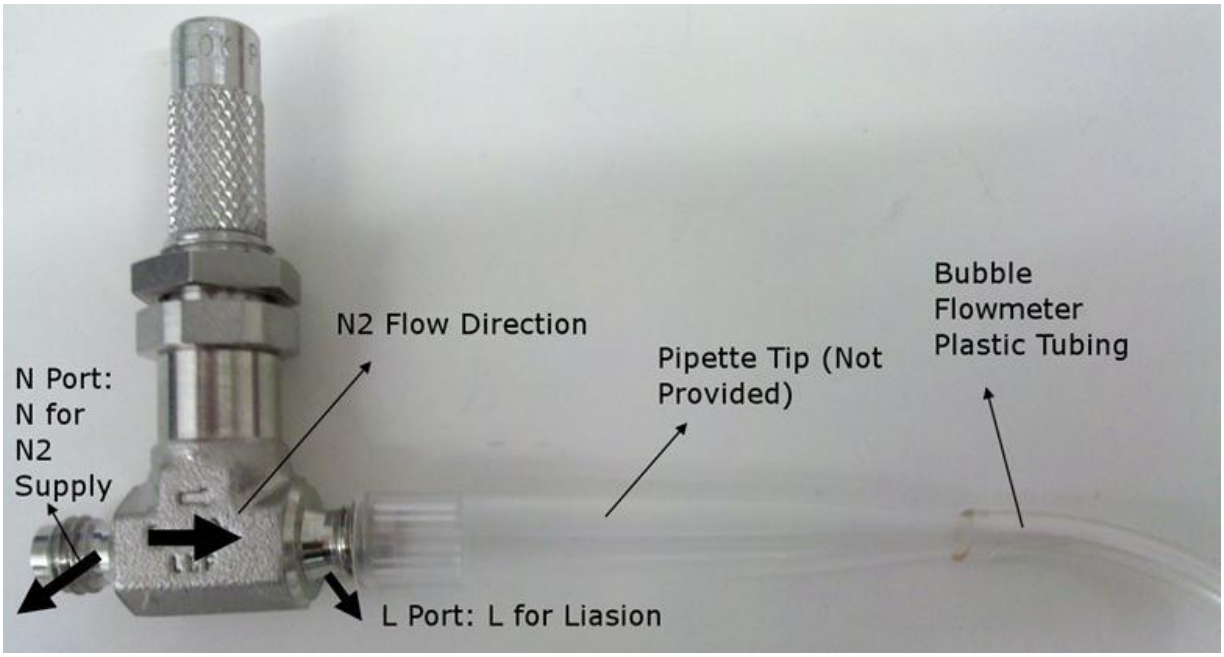
3.1.3. Connect to the CRDS output (70ml/min).

3.1.4. Use the Tube Fitting in CM Kit to make the Connections.

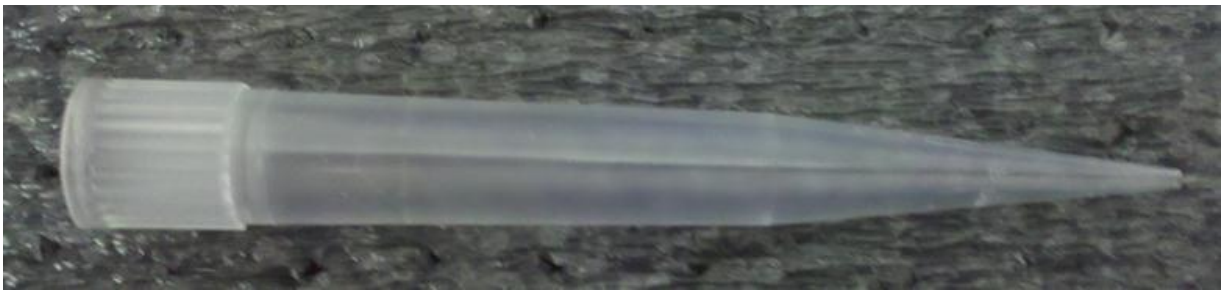
3.1.5. Connect the Flowmeter to the Needle Valve.



3.1.6. Connect the Flowmeter to the Needle Valve L Port 70ml/min (Close-Up).



3.1.7. Use the Pipette Tip to make the connection above (Not Provided)



3.2. INSTRUCTIONAL VIDEOS

3.2.1. Below is an instructional video on how to weigh sample and how to seal sample into capsule.

http://www.photoshop.com/users/costech_analytical/assets/ea3d6efc6a3342368a026fe8e38f173f

3.2.2. Below is an instructional video on how to use the auto sampler. It also shows what a flash combustion looks like.

https://www.photoshop.com/?user=costech_analytical&galleryid=7b5f16a0dc0b448ab664399ebacb2880&wf=share&trackingid=BTAGC

3.3. FAQ

The following are questions and answers we've compiled over the years while working with Picarro customers.

3.3.1. **Regarding** Bag Installation and Flow Check

A. Make sure that the CM is in "work" mode (flows are on by pressing work + enter).

B. Adjust the flow coming out of the CM (CRDS output port) to 70-80 ml/min using the SS fitting provided and left inside the CM cabinet.

C. Adjust the N2 purge flow connected to the back of the Liaison interface to 70-80 ml/min using the connected needle metering valve.

D. You might need to adjust/bend the bag holder bracket legs little outwards to attach the leg hooks to the bag holes. The bag holes position might slightly change from bag to bag. hence the need to adjust.

E. Please remember that you have an extra bag that came with the installation kit.

F. As mentioned in the Liaison manual, Gas Sampling Bags are considered consumables, since they develop fatigue marks with time and need to be replaced as a preventive maintenance procedure or when deemed necessary (obvious fatigue mark, creases, ripped bag seam, etc).

G. When the combustion/reduction reactor is replaced, please make sure that the leak check has been follwed. Briefly, plug the CRDS output port with one of the brass fittings or SS fitting with cap provided and left inside the CM cabinet, make sure the CM is in work mode (flows are on), raise the carrier gas pressure to 1 bar, leave it there for 1min and then back out the gauge knob and watch for any drop in pressure: no drop in pressure = no leaks. Possible leaks points are normally the upper collar connecting the Autosampler to the combustion tube (use provided key to tighten) and the bottom quick connect fitting of the combustion tube.

3.3.2. Leak Troubleshooting?

A. To find out where the 200ppm of CO2 is coming from in Blank samples, remove the purge Teflon tube that goes to the bottom of the Autosampler and check that

it has 100ml/min of N2 coming out of it. If not, then you can adjust it using the purge knob (see the manual attached). This will help push out any atmosphere seeping into the Autosampler. Also, please make sure that you have the plastic lid/cover of the Autosampler on, when running samples. Then make sure to reconnect the Teflon tubing to the bottom of the Autosampler.

B. Check the O2 flow out of the Vo2 vent at the front of the CM. It has to be 30ml/min. If it's not, then please adjust it using the bottom needle valve. The needle valve is locked in one position using the nut, so loosen the nut, adjust it and then tighten the nut again while making sure that the O2 flow is still at 30ml/min, and then program the coordinator to run three samples but load only empty tin capsules (blank) on the A/S.

C. The oxygen tank purity should be at least 99.995%. To test whether the O2 is contributing to the CO2 background, make sure you exit the "remote" mode by pressing "abort" and then "enter", Press the O2 key (the key between “chns” and “comb”), this will put the O2 pulses on hold, then press "remote", then "enter" and tell the coordinator to run 3 samples, but do not load any samples onto the A/S. If the background is low (20ppm or lower), then we will know that the high CO2 background is coming from the oxygen tank. If the background is high, then the problem is coming from the combustion tube or the Autosampler. At the end of this test, please press the CHNS key again to allow the O2 pulses to discharge during combustion.

D. If the high background still exists after steps A & B & C: To test if the high background is coming from the Autosampler or the combustion tube, please run the following test:

E. Make sure you exit the "remote" mode by pressing "abort" and then "enter", Press the F1 key (this will disable the A/S), then press "remote" , then "enter" and tell the coordinator to run 3 samples, but do not load any samples onto the A/S. If the background is low (20ppm or lower), then we will now that the combustion tube is ok and the problem is coming from the A/S. If the background is high, then the problem is coming from the combustion tube. At the end of this test, please press the F1 key again to engage the A/S.

3.3.3. Questions about Memory Effect

A. **Q: Can I increase my oxygen loop to Semi-Micro?**

There is no major issue with increasing the oxygen loop to Semi-micro. Any excess O2 will be trapped by the copper pellets (Cu => CuO) in the bottom half of the reactor, which is the reducing catalyst. The only caveat is that the Cu will get oxidized much faster in case there is lots of excess O2 to be trapped. Normally, even after 1000 analyzed samples with micro O2 loop, only half of the Cu gets oxidized, so my assumption that the reducing copper should hold up to 1000 samples easily. Keep in mind that the O2 micro loop is 2.5ml, the semi-micro loop is 5ml.

B. **Q: How can I avoid incomplete combustion?**

a. Make sure that your sample is crushed into a fine powder to maximize its exposed surface area and hence its contact with O2.

b. Minimize the sample quantity to be combusted for it to barely exceed the minimum sample amount required by the system. I noticed when I checked your sample runs a month ago that you had samples that generated CO2 signals of 10,000ppm. Try to use the smallest sample amount possible that will generate 2000ppm or little more. This will prevent the combusted sample from smoldering in the ash insert and leading to carry over effect.

c. Use blank tin capsules between samples (sample, empty tin capsule, sample, empty tin capsule, etc…). This will reduce your sample throughput by half but will ensure the generation of carry over and will minimize the need to repeat some of the sample analysis. Blanks will help remove the memory between samples.

d. Clean your alumina ash insert very frequently, I would suggest on daily basis. You have 2 inserts have one cleaned up daily and ready to replace the used one when you start your run.

e. Use gloves, wipe the carousel bottom plate to prevent any cross-contamination from sample-to-sample, wipe the sample prep plate all with IPA and let it dry. Also use canned air to flush any remainder sample on the spatula, plate or carousel.

C. **Q: Can we use air for the pneumatic Autosampler?**

Regarding using air for the pneumatic AutoSampler, you will save a little, since the pressurized gas is used to push the piston in one direction to drop the sample and then is blocked again. The major consumption of N2 is by the CM itself at 80ml/min when running continuously. The Liaison uses N2 only to purge the bags and generate a baseline (500ml every 10min). Normally, when scientists use Helium (expensive gas nowadays!) as a carrier gas, they opt to use N2 or Air for pneumatics to save money. However, if you can afford a new tank regulator, go for it.

3.3.4. Lousy Precision?

A. A lousy precision is normally an indication of either incomplete combustion or a leak.

B. The interspersed blank tin capsules will address the incomplete combustion possibility.

C. Also, please check that the O2 flow from the front vent is adjusted to >30ml/m (best if it's 35ml/min) to make sure that we have adequate O2 flow for complete combustion.

D. Regarding the potential leaks possibility, here are few things to check:

E. Check if the CM is leak tight up to the "CRDS" outlet fitting (I believe you did that and your confident of a leak free front-end). This will guarantee that the analytical flow from the gas tank to the CRDS outlet fitting is leak-free.

F. Check that the connection from the CM to the Liaison inlet is tight and leak-free. The Liaison inlet currently uses a Teflon ferrule that might need to replace with a metal reducing fitting (2mm to 1/8") if need be.

G. The transfer line from the back of the Liaison (1/8" nut, CRDS output) to the CRDS inlet (1/4" nut connected to a 1/8" nut). Please make sure that all the nuts are tight and leak-free.

H. The connections to the gas sampling bag valves. Gently check if the connection is tight.

I. Please make sure that the N2 flow to the back of Liaison is held at 60-70 ml/min, for it to thoroughly flush the bags between runs. You might need to install an inline pressure regulator prior to the needle valve.

3.3.5. More Questions

A. **Q: Why does my coordinator show an overly high concentration of CO2 even after the completion of sample analysis?**

The high concentration is coming from the remainder of the gas in the gas bag and the tubing. If you close the coordinator, and then choose to "run the final state" when prompted, the bags will be evacuated. This will cause the background line to drop to the atmospheric level.

Is the sample in a powder form and if so, is it well homogenized? Samples have to be finely crushed and homogenized. You might be having incomplete combustion due either to the large sample you're packing and due to a crystal form instead of powder. The optimal CO2 signal should be between 2000ppm and 5000ppm.

The sample carousel contamination can take place if the tin capsule breaks while on the carousel or it's contaminated from the outside. To clean it, just take the carousel of off the A/S and use isopropyl alcohol to wipe the contamination. You might have to use a wetted cotton swab to get the job done. It is also possible to separate the bottom plate of the tray from the one with holes and wipe it clean. But I would address the sample form and sample size as well.

B. **Q: What do I do with a large sample?**

In case you have a large sample you can always choose the semi-micro oxygen loop. This will deliver 5ml of oxygen instead of the 2.5ml per sample.

C. **Q: Can I use Atropine/Acetanilide as my standard?**

The Atropin or Acetanilide standards are not isotopically calibrated. If you want to use them as standards, back-calibrate them from the USGS 40 (-26.389permil). Run 5 replicates of Atropin or acetanilide with 5 replicates of USGS40.