

AutoMate Prep Device Instructions Systems with contract closure interface for use with Picarro IsoCO₂ and Liaison

Jason Curtis 5-29-13
Jason@automatefx.com
352-275-8642

Initial set up of AutoMate

The AutoMate is composed of 4 parts:

One box with the electronics (called the Controller box)

One box with the pumps, valves, flow controls (called the Wet box)

The carousel

The computer with the AutoMate Prep Device software

Positioning

(See positioning image, page 9)

Place the carousel on a lab counter. Then place the Wet box (the one with the flow meter on the front panel) to the right of the carousel. Then place the Controller box on top of the Wet box. (Don't put the Wet box on top of the Controller box as liquid might get on the electronics).

Tubing

All tubes are labeled – just match them to the label on the part they go to.

Compressed tank gas (HP or UHP N₂) runs through 1/8" nylon tubing from tank regulator to the back of the carousel. Inside the carousel is a tee that splits the tank gas into 2 streams. One runs the pneumatics that move the needle assembly. The other stream goes into a solid CO₂ scrubber (Ascarite, NaOH on a media). Following removal of CO₂, this is now considered the carrier gas. The carrier gas runs to the 1/8" quick fitting labeled "Carrier Gas In" on the Wet box.

The other 2 inputs to the Wet box are "Acid In" and "DI Water In". The fittings are such that you cannot reverse the bottles. Once you get the whole thing set up and acid and water in the bottles you will need to prime the liquid lines (instructions below).

The output from the Wet box is through a 1/8" Teflon tube that runs to the longer needle. The tubing simply slips over the needle. This slip junction is leak tight. If you need to cut off the tubing remove as little as possible. Then use something like a ballpoint pen to slightly flare the tubing. Then slip it onto the needle. (Batches of 1/8" tubing have varying ID and some work to slip over the needle and form a leak tight seal while most batches do not - Contact AutoMate FX if replacement tubing is needed) Do not attach the tube from the Wet box to the shorter needle -- If this is done the liquid in the sample vial will end up in the scrubber and eventually in the Liaison.

The shorter needle is attached to a shorter piece of 1/8" Teflon tubing also by a slip fit. The other end of the tubing goes into the side arm of the post sample scrubber. The Teflon tubing is inserted into a piece of 1/4" OD, 1/8" ID tygon tubing that acts as a spacer in the screw fitting that attaches to the scrubber. The output tubing from the needle attaches to the side arm of the post sample scrubber. Do not attach the output from the shorter needle to the body of the scrubber -- If this is done all the liquid in the scrubber will end up in the Liaison.

The output from the main body of the post sample scrubber travels through a 1/8" Teflon tubing into the top of the Nafion water scrubber and out the bottom of the Nafion water scrubber and then into the Liaison.

Scrubbers

Pre-scrubber is mounted horizontally on the right of the main instrument. This pre-scrubber holds Ascarite (NaOH on a media) or another CO₂ absorbent. Put a wad of glass wool or quartz wool one end of the glass trap and then fill trap with absorbent and then put a wad of glass wool or quartz wool at the other end. The glass wool or quartz wool acts to trap the absorbent so that it does not get blown out of the trap.

Post-sample scrubber is necessary to collect any acid vapors or acid drops and keep them from getting to the Liaison and the Picarro analyzer - this requires simply filling the scrubber about half way with deionized water. Change weekly or if amount of water decreases.

The Nafion trap removes water vapor from the carrier gas / sample gas stream. Nafion is a semi-permeable membrane through which water passes but almost nothing else does - including N₂, CO₂, and Helium. The trap requires a counter flow of dry gas. The counter flow is tank HP or UHP N₂ after it is pressure regulated. This counter flow runs outside the semi-permeable membrane inside the stainless steel tubing and out the top side arm. The short white tube is a constriction that allows only about 250 ml / min of gas flow.

Cables

There are 4 serial cables and one power cord (US style only. Other country types require a standard power cord like used for computers). Each connection is labeled and/or different style so they cannot be incorrectly attached. (1) A 25-pin cable connects the Controller and Carousel. (2) A 15-pin cable connects the Controller and the Wet box. (3) Two 9-pin cables with RS232 to serial adapters connect the controller and the computer. Both must be attached. One 9 pin cable is the communication between the AutoMate (Port "To Computer") and the computer ("Left USB port"). The other 9 pin cable is the contact closure signal from the AutoMate (Port "To Computer 2") to the computer ("Right front USB port"). The 2 pin cable (that arrived with the Liaison) attaches the Picarro Liaison to the AutoMate control box screw terminals.

Compressed Gas

Carrier gas can be a variety of gases. The best is ultra high purity N₂. Industrial N₂ and compressed air are not recommended as the Ascarite scrubber will clog quickly.

Software installation

Software for the Picarro IsoCO₂ - Liaison - AutoMate Prep Device is pre-installed.

Updated versions of AutoMate software will run with no issue. Just place them in the AutoMate folder on the Desktop and double click on them. You can place a shortcut on the desktop.

An installer is included in case a new computer is needed (Win 8, Win7 and WinXP).

AutoMate control software

(See screenshot on page 9)

The graphical interface for the AutoMate is all on one screen for simplicity. The various functions are roughly divided into groupings.

Across the top are some general buttons.

"Close AutoMate Application" = quit the program

"STOP After Current Sample Complete" = Stop Autorun when current sample finished

Just below this across the top are a series of dots with text above. These dots light up to show the sequential happening in an auto run. They are mostly self-evident. "Controller" is the step where the software checks communication with the controller.

On the left side of the screen are a series of buttons with pull down menus. The top one is "System Control" which controls many AutoMate functions. Use "System Control" with caution as you can do bad things like start the acid pump running and then leave it on, consequently pumping acid all over the place. The rest of the buttons (5 in total) control parameters during autoruns. See below for specifics of these autorun parameter buttons.

Most of the System Control selections are self evident. Here are a few that might require explanation:

"Get system status" = check the status of all the functions of the automate and update the current status lights

"Test Controller Communications" is somewhat repetitive as this is also done in "Get System Status" above but it is also useful other times.

Autorun parameter buttons have pre-set value that should work for most situations. They can be modified as needed but once the software is turned off and rebooted they go back to the original values. If you find that you need different values, AutoMate FX can re-compile the software with your specific values so that whenever the software starts your specific values come up.

"Sample Purge Time" = Time to purge atmosphere from vial prior to acid injection.

"Acid Inject Time" = Time to inject acid. The main requirement here is that the level of the acid in the vial is high enough so that the longer needle is in the liquid. If the liquid level is lower than the longer needle run time will be much greater.

"DI Inject Time" = Time to inject water following sample analysis. This is mainly to clean the insides of the tubing and valves. The outside of the needle is squeegeed off when the needles withdraw.

"DI Purge Time" = Time to allow the water in the valves and lines to be blown out by the carrier gas

"Sample Run Time" = Length of time for each sample.

In the middle of the screen are controls for the data table. "Table Functions" is a button with a pull down menu. Here you can create a new table.

Below the Table are a second series of dots that show the current status of the functions of the AutoMate. Most are self-explanatory. Grey is off and green is on.

Needle down = needle assembly is in the full down position

Needle up = needle assembly is in the full up position

Supply pressure = carrier gas pressure is above 6 psi

Vial in position = carousel is lined up directly under the needle assembly

Carousel zero = carousel position zero is lined up directly under the needle assembly

Carrier gas = carrier gas is flowing

Controller = Automate software is communicating with the Controller

At the bottom center of the screen are a series of read outs that show timing during runs and the position the carousel is in

At the bottom right of the screen is a pull down to select the vial position, a button to go to the above select position, and a button to go directly to zero.

During an autorun the software looks at the table to determine which samples to run.

Basically it looks at the "'1" = Run Vial column' from the top and finds the first row with a "1". Then it looks to see if the "Run Status" cell has any data. If there is no data it runs that sample. If there is data in the "Run Status" cell it assumes that the sample has been run and looks for a row with a "Run Vial" cell = 1 but no data in the associated "Run Status" cell. It will then run that cell. Once all the rows with "Run Vial" have associated data in the "Run Status" cell the autorun ends.

Note: Once you enter a "1" for the last sample you must click outside the cell to get the software to recognize that the cell has been populated. Otherwise that last sample will be skipped.

Priming liquid lines

The AutoMate system is shipped with no liquid in the lines for safety. You must remove the air in the lines and replace it with water and acid. Here are the priming instructions:

Fill and lightly cap the water and acid bottles (caps must not be tightened - air needs to be able to get in otherwise a vacuum forms). Put Exetainers without caps in positions 1,2,3, Advance carousel to position 1. Put needle down. Turn on Carrier gas. Click on “acid on for 2 seconds” button, then select ok. Repeatedly select “Acid on for 2 seconds” for about 6-10 times until acid flows to the vial. Make sure that you do not overfill the Exetainer. If needed turn carrier gas off, move needle up and move to next vial, put needle down, carrier gas on. Then repeat for water using “Water on for 2 seconds” button. Repeatedly select “water on for 2 seconds” for about 6-10 times until water flows to the vial. This only has to be done when first filling the bottles, unless you let the bottle go dry.

If you have problems with priming a pump see the troubleshooting section at the end of this document. Also contact jason@automatefx.com

Sample loading

Samples are loaded into Exetainer vials. These are 12 ml screw top vials with septa tops. They are made by Labco in the UK. 12ml Borosilicate Vials - Round Bottomed. 938W or 538W. <http://www.labco.co.uk/usaandcanada/>

Note: About 1 in 100 of the Exetainers are slightly too large in diameter for the AutoMate carousel. AutoMate FX suggests discarding any abnormally large vials. Caps can be used 3-5 times each. Vials can be washed and dried and reused forever. New caps and septa can be purchased from Labco.

DIC - Use macro-pipette to load 5 ml sample, blank, or standard into the Exetainer. Place cap on. Sample is ready. (Sample vials can also be capped in a glove bag filled with N₂ if desired).

Carbonates - Cut strips of weighing paper about 1cm wide by 5 cm long. Fold the strips lengthwise. Place strips on balance and tare. Add material onto weighing paper until correct weight is reached. Record weight. Then pick up strip with sample using a forceps and slide into a vial until the strip is near the bottom. Tap powder off so that it all gets to the bottom of the vial. Place cap on. Sample is ready. (Sample vials can be capped in a glove bag with N₂ if desired).

The septa seal surprisingly well and do not need to be tightened very much. If the cap puckers down then it is tightened too much. Examine a septa. You will notice a slightly raised ring in the rubber at about the same diameter out from the center as the opening in the cap of the vial. Watch closely when you tighten a vial. When this ring start to shrink toward the center slightly the vial is tightened enough.

Samples can be placed in standard test tube racks. Sample vials can be written on with marker. Tape is not recommended unless it is kept right near the top of the vial (this is because the holes in the carousel are just slightly bigger than the vials).

Supplies

The AutoMate requires three main consumables that must be replenished on a regular basis. These are DI water, acid (nitric, hydrochloric, sulfuric acid or phosphoric are all compatible with the materials in the AutoMate - do not use perchloric), and a CO₂ free gas (high purity N₂ or ultrahigh purity N₂).

AutoMate FX highly recommends 10% phosphoric acid. 1N nitric and 1N hydrochloric acid also work but see the needle will not last as long (see consumable section below). Special coated needles help with 2N sulfuric acid – contact AutoMate for more info.

Recipe for 10% Phosphoric acid

Wear gloves and goggles

60 ml 85% ortho-phosphoric acid (reagent grade)

440 ml DI water

Add acid to water, mix, and wait a minute or so. Container will be slightly warm.

Water and Acid are stored in 1-liter bottles with the outflow through a tube at the top. One filling of bottles should last about 6 to 8 sample runs for carbonates and 20 or so runs of DIC. There is no safety to stop the run if the liquid bottles are allowed to go empty so check at the start of each run. Two cautions. First, the acid and water quick connects on the back of the Wet box are not valved. If they are unplugged the contents of the bottles will flow out. Fill the acid and water bottles with a beaker and a funnel. Second, do not tightly cap the water and acid bottles when running the AutoMate. This will cause a vacuum to form and eventually no liquid will flow.

Other more long lasting / durable consumables are Ascarite (or other CO₂ absorbent) for the CO₂ scrubber, needles, and septa top Exetainer vials. The Ascarite in the trap will last a long time if the input gas is low in CO₂. The needles will last for differing times depending on the acid that is used. The needles are made of 316 stainless and are most readily attacked by nitric and sulfuric acids, then less so by hydrochloric acid, and least by phosphoric acid. Septa that are shoved into the vials are a pretty good indication that one of the needles is dull or broken. This will almost always be the longer needle because it actually sits in the acid during sample reaction. A simple tool is included to help install new needles to the correct depth in the needle holder assembly. See instructions later in manual.

Running samples on the AutoMate

The carousel holds 45 vials total.

Place samples in carousel. You will have to use the software to turn carousel to get samples into the position right under the needle guide. Use the vial button and goto vial button.

Enter sample names and a "1" for each sample to run into the table.

Ensure that water and acid bottles have enough liquid.

Check gas supply pressure (basically the amount of gas left in the cylinder) and delivery pressure on the gas tank regulator (set to about 30 psi).

Check that the carrier gas pressure on the front of the wet box is about 8 or 9 psi. If it is not, you must adjust the pressure regulator. This is under the carousel. Lift the front of the carousel until the carousel is at about a 30-degree angle. Then reach under the front right side and pull the knob downward to unlock the regulator adjuster. Then turn knob while watching the gauge on the front of the Wet box. When adjusted to 8-9 psi push knob upward to lock.

Prepare post sample scrubber with deionized water and connect into flow path.

Check system status lights (Use "System Control" and "Get system status"). The following should be lit - needle up, supply pressure, vial in position, controller.

NOTE: After samples are in carousel you MUST click the "Goto Zero" button in the lower right side of the screen. (This fixes any alignment errors in the carousel). The carousel might turn all the way around or not. If it turns around, wait until it goes back to zero and then start the Picarro software.

Once the needle goes into the first vial and flow starts check that the carrier gas flow rate is about 75 ml / min on the front of the wet box. See Liaison manual for exact flow rate.

Times:

DIC water samples:

Acid = 1 second

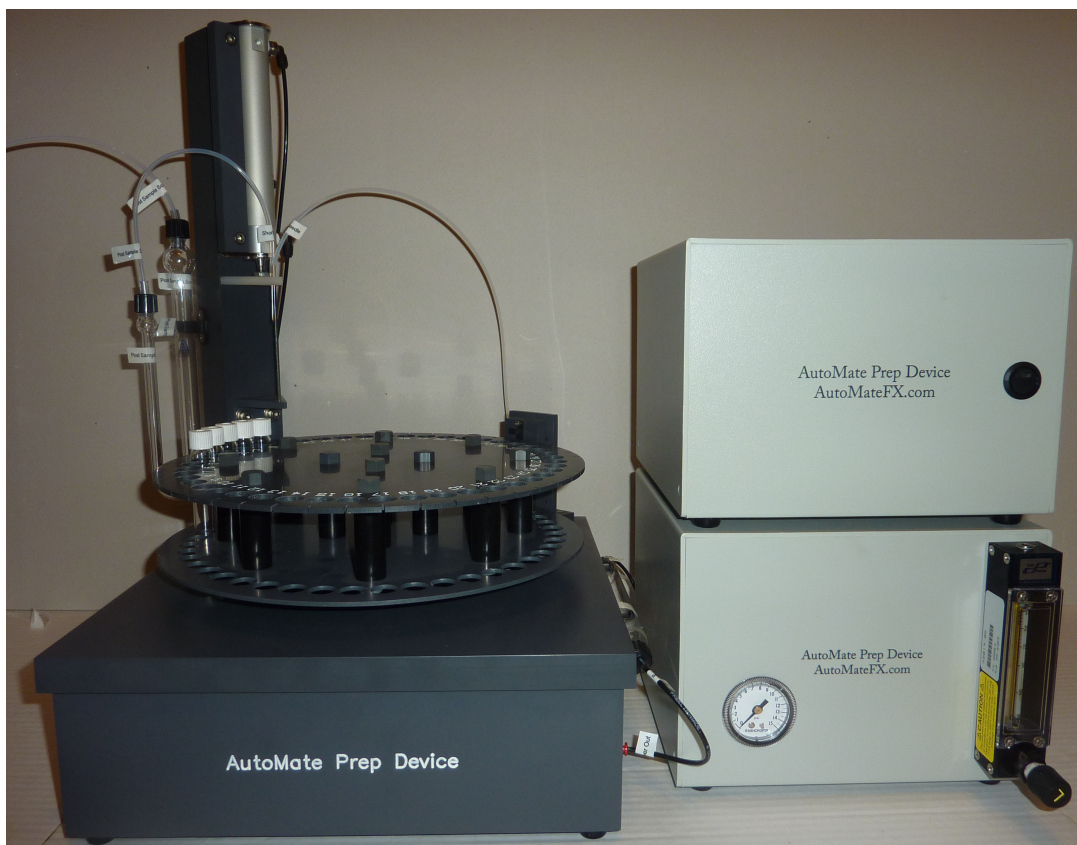
Water = 1 second

Solid carbonate samples

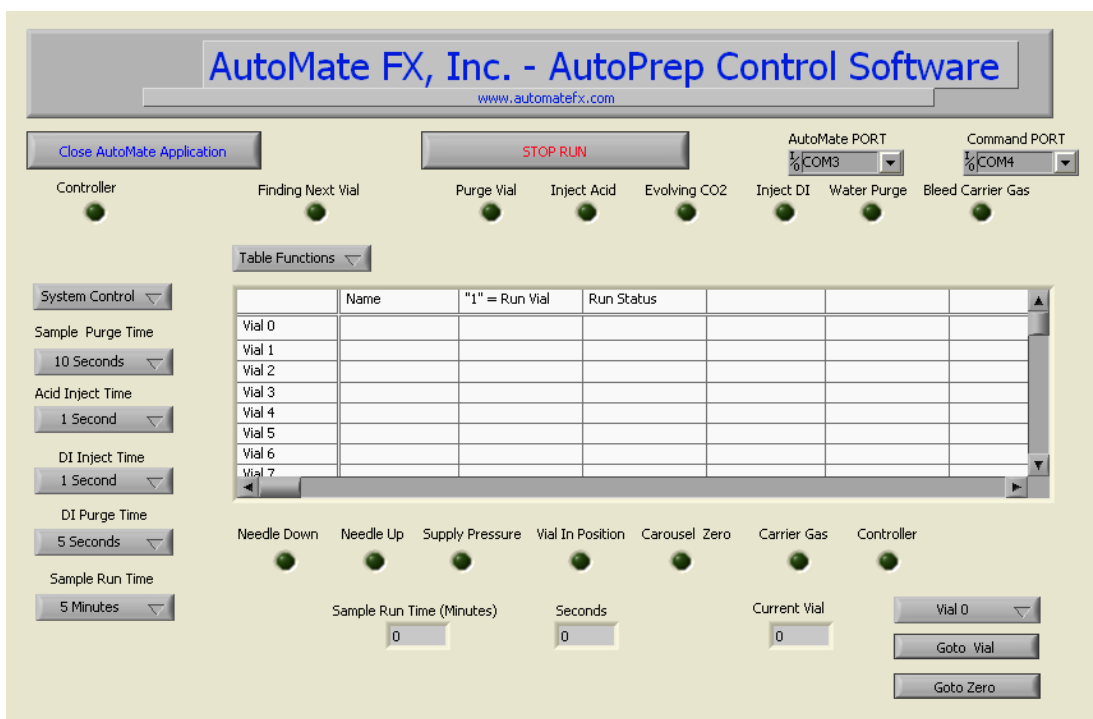
Acid = 3 seconds

Water = 2-4 seconds

All other times use default values.



AutoMate positioning



AutoMate software screenshot

Instructions flushing acid and water prior to shipping

If you need to ship the system you must flush the acid and water out of the system as a safety. To do this empty acid bottle. Then remove 1/8" tube from "Air/liquid to long needle" and replace with disposable 1/8" tubing. Do not lose the 2 ferrules in this fitting. Aim the replacement 1/8" tubing into a beaker. Turn on acid pump (System control, acid pump on). The acid pump will run for a maximum of 7 seconds. Run until acid stops flowing out. Add some DI water to acid bottle and run pump again until water comes out of replacement tube and flows into beaker (again, in 7 second increments). Dump rest of water out of acid bottle. Dump water out of water bottle. Turn on water pump and run until water stops flowing (System control, water pump on) - again in 7 second increments.

Needle replacement instructions

To remove the needle from the needle holder you need to do the following:

1. Turn off compressed gas
2. Turn the carousel to a position where there is no vial
3. Manually push the needle assemble down
4. Remove the tubing from the needles
5. By hand unscrew the the steel pneumatic rod from the needle holder. If you can not move it by hand use a wrench on the lowest portion of the rod where there are 2 flats for grabbing. Do not use pliers on the steel pneumatic rod.
6. Push the steel pneumatic rod up. Then lift the needle holder assembly up and gentle rotate it so that the needle comes out of the needle holder.
7. Once the needle holder with the needles is free measure the length of needle sticking up and the length of needle sticking down. Record this.
8. After recording the stickup and stickdown you can just take a pair of pliers and crush down on the longer portion of the needle you want to remove and then twist the needle out. You sort of wind the needle around the jaws of the pliers as you twist the handles of the pliers.

To install new needle

1. In the supplies that were delivered were 2 "tools" for replacing the needles. The 2 tools are a piece of stainless steel tubing 4" long and a brass rod (together these will give a stickup that works for your system).
2. It is best to find a counter top (or some flat surface) with a small hole in it so that you can place the needle holder flat on the counter with the remaining needle through the hole. You can also do it over the edge of a counter but this is a little more tricky.
3. Use a hammer to gently tap the needle into the holder - only about 1/16". Then slip the stainless steel tube over the needle, hold the stainless steel tube up a little, insert the brass rod into the stainless steel tube, and hammer gently on the brass rod. This will hammer in the needle without bending the needle. Before the rod gets all the way flush to the stainless steel tube stop and measure the stickup and stickdown. The stainless steel tube and the brass rod should be the correct length so that when you have hammered the brass rod flush with the stainless steel tube the needle should be in the correct position. But it is better to stop and check a couple of times.
4. To reinstall the needle assembly you should hold the assembly off to the side and aim the long needle into the smaller hole in the needle guide. When it is started you can then sort of rotate the assembly so that the needles will slide down into position.
5. Then rethread the steel pneumatic rod into the needle holder until the screw on the steel pneumatic rod just is flush with the bottom of the needle holder. Run the nut down to the holder.
6. Reattach the tubing
7. Visually check that the long needle is in the needle guide but not sticking down below the needle guide.
8. Put a vial in the next position, turn to it, plunge needle in, turn on carrier gas, confirm that gas flows to post sample scrubber. If no flow either the tubing is not on tight or, if you changed the shorter needle, maybe the shorter needle is not in far enough.

Troubleshooting

No liquid delivery (either acid or water)

Some users have had problems with getting the pumps to prime (fill with liquid at first usage). There are two possible causes of this problem. (1) a stuck valve (or diaphragm) in the KNF pump or (2) an air block in the tubing between the water bottle and the water pump (most likely between the bulkhead fitting and the pump).

The good thing is that the same procedure should solve either problem. Solution below is outlined for the water system but the acid system is fixed the same way.

To solve it you will need a disposable plastic syringe of about 25 to 50 ml (no needle). One with a locking Luer connection is best but not important.

1. Pull the tube out of the water bottle.
2. Fill syringe with DI water
3. Insert tip of syringe into water tube
4. Turn carousel to a position with an empty Exetainer vial with no cap
5. Move needle down under System
6. Turn on carrier gas under System
7. Turn on water pump under System (you should hear the pump hum and a click)
8. Push on the syringe and gently force water into the tube (you will have to hold the tube onto the syringe)
9. You should see water leave the syringe and go into the tube. Pump should change sound as it starts to pump water (it will slow down a little).
10. Turn off water pump under System or after 7 seconds it will turn off automatically.
11. Replace tubing into water bottle
12. Run the water pump a couple of time to check
13. Turn off carrier gas under System
14. Move needle up

If this does not work try taping (gently hitting) on the tubing inside the wet box while the pump is running. The next sentences tell where to tap. The water bulkhead fitting is attached to a larger diameter tube which goes to a reducing fitting then to a smaller diameter tube and then to the pump. Tap on the reducing fitting and also anywhere the tubing loops up and allows a place an air block could occur. You could also bend the tubing down so that any loops are forced downward to allow air to move.

Troubleshooting continued

No start of AutoMate software when Picarro call for sample.

Background - The Contact Closure interface allows remote start of the AutoMate Prep Device. On the back of the "Control Box" are 2 terminals (red and black) that when crossed with a wire (or a contact closure signal from an analyzer) will start the AutoMate software for one sample. Thus the AutoMate Prep Device can be remotely controlled.

1. You can cross the black and red terminals on the back of the AutoMate control box and determine if the AutoMate is operating correctly. Leave the table blank (no "1" in any vial positions). The "Controller" indicator will go green showing that the contact closure has sensed a start command. Then the finding vial indicator will go green showing that the software is trying to determine which vial to run. It will find no vial to run and abort.

If the test above works then the problem might be that the AutoMate system is not getting the correct signal from the Liaison. Check Liaison cable and contact Picarro and AutoMate.

2. Make sure that both the USB to serial adapters are attached to the computer and the 2 9 pin ports on the back of the control box. One is control communication and one is the start signal from the Liaison.

Generally the Serial to USB cable that connects to the "To Computer" port is attached to the USB port on the left side of the laptop. This is COM3.

Likewise the Serial to USB cable that connects to the "To Computer 2" port is attached to the USB port on the front, right side of the laptop. This is COM4.

If you have trouble with communication with between the laptop and the AutoMate please contact AutoMate.