AutoMate Instructions 1.8
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Initial set up of AutoMate
The AutoMate is composed of 4 parts:
One box with the electronics (called the Controller or Electronics box)
One box with the pumps, valves, flow controls (called the Wet box)
The carousel
The computer (supplied by customer) with the control software

Positioning
(see AutoMate positioning image)
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
(see AutoMate tubing image)

Note all tubing and fittings are labeled so that assembly is quick and easy.

Compressed tank gas (HP $N_2$) runs through 1/8" black nylon tubing from regulator to the back of the carousel. One branch runs the pneumatics that move the needle assembly. The other branch into a solid $CO_2$ scrubber (Ascarite, NaOH on a media). Following removal of CO2, this is now considered the carrier gas. The carrier gas runs through 1/8" black nylon tubing to the 1/8" quick fitting labeled "Carrier Gas In" on the Wet box.

New note as of April 2010: Use of UHP $N_2$ avoids the need to scrub CO2 from the supply gas. Then the Ascarite trap can be bypassed and gives one fewer consumable to worry about.

The other 2 inputs to the Wet box are "Acid In" and "DI Water In". The fittings are such that you can not reverse the bottles. (Once you get the whole thing set up and acid and water in the bottles you will need to purge out the acid line and pump and valve. It was flushed with water for shipping. See next paragraph)

Pump priming instructions (will make sense once the section on the software is read)
Fill water and acid bottles. Put caps on bottles (caps must not be not tightened so that air can get in otherwise a vacuum forms). Put Exetainer vials 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. Repeat about 3-5 times until acid flows after the pump turns off. Make sure that you do not overfill the Exetainer. If vial gets full do the following - turn carrier off and put needle up and move to next vial, put needle down,

carrier gas on. Then repeat for water using “water on for 2 seconds” button. This only has to be done when first filling the bottles, unless you let the bottle go dry.)

The output from the Wet box is through a 1/8" Teflon tube that runs to the longer needle. This just slips over the needle. We have never had leaks at this slip junction. 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. (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, Inc. for replacement 1/8" Teflon tubing). Do not attach the tube from the Wet box to the shorter needle or the liquid in the sample vial will end up in the scrubber and eventually in the coulometer cell.

When delivered the AutoMate will have the Teflon tubing attached to the longer needle already. The other end needs to be attached to the Teflon bulkhead fitting on the back of the wet box. Loosen the fitting and slip the tube in and tighten. Try not to take the fitting totally apart. There are 2 loose ferrules in the fitting and they are easy to lose!

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 or all the liquid in the scrubber will end up in the coulometer cell.

The scrubber is necessary for two reasons. First, to collect any acid vapors or acid drops and keep them from getting to the coulometer cell - this requires simply water or a silver nitrate solution (the coulometer cell is basically titrating CO2 acidity so acid from reaction cell really messes things up). Second, to collect any $SO_2$ that might be released from samples -- this requires a silver nitrate solution (3% silver nitrate in DI – see coulometer manual).

The output from the scrubber travels through a 1/8" Teflon tubing. Again, this tubing is also 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 downstream end of this tube goes into a one-way valve and then into the cathode side of the coulometer cell.

<u>Cables</u>

There are 4 cables, 1 USB to RS232 adapter cable, and one electrical cord for the AutoMate. Each connection is labeled and they can not 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) A 9 pin cable connects the controller to the USB to RS232 adapter and then to computer and (4) another 9 pin cable connects the controller and the coulometer.

The connections for the UIC 5012 and the 5011 coulometers are different. The 5012 connects simply with a 9 pin cable into the "Serial" port on the back. The 5011 connects

with a 25 pin cable. The 5011 top must be removed by taking out the hex head screws. The 25 pin connection is at the back of the topmost board. The 5011 needs the 9 to 25 pin optional adapter cable. Attach it to the 9 pin cable and attach the appropriate 25 pin connector to the coulometer.

Coulometer communication set up

(see communication switches images on page 12)

The software communicates with the coulometer at 9600 baud. This must be set using dip switches inside the coulometer. This is very easy on the 5011. Remove top and the uppermost board has the dip switches. Find the red dip switches with 4 switches. Set 1 and 4 to 0 (zero) and 2 and 3 to 1 (one). (see 5011 communication switches image). replace top. For the 5012 it is a little more in depth. First you must remove the right side cover (right hand cover as you face the front of the instrument). There are 6 Phillips head screws, 3 on the back and 3 on the side. Once the cover is off you are looking at several boards. The one that you want is the uppermost one (see 5011 communication switches image). You can not see it very well due to the mounting design. The board is the same one as in the 5011. On the edge of the board facing you is the same red 4 dip switch fitting. The settings are the same as for the 5011. Set 1 and 4 to 0 (zero) and 2 and 3 to 1 (one). (see 5011 communication switches image) Once the dip switches are set correctly I suggest trying communications with the AutoMate system prior to putting the top or right side cover back on. (see software sections for checking coulometer communications)

Also on the coulometer set the time to 50 minutes and the roller switch to 1 or ug C.

UIC 5014 and 5015 $CO_2$ coulometers need a new firmware chip to communicate with the AutoMate system. These are available directly from UIC, Inc. Contact AutoMate FX for part numbers.

Compressed Gas

Carrier gas can be a variety of gases. The best is ultra high purity $N_2$. high purity $N_2$ also works well, industrial $N_2$ works also but there is some $CO_2$ in it and the Ascarite scrubber will get clogged more quickly. $CO_2$ free air also works but it might be more expensive than HP $N_2$. We do not recommend compressed air because the Ascarite scrubber will clog very fast. We don't recommend a liquid NaOH scrubber like used on manual UIC TIC systems because they require daily maintenance.

Software installation

Installer included on CD.
Windows XP or 7 - any speed. Vista will not work. We are working on Windows 8 installer. Older laptops work well.
Open CD. There is a folder "AutoMate Installer" and one file called AutomateXX.exe.
Copy both to the desktop (or C:).
Open Installerfiles.
Run setup.exe.

Agree to license and location for installation. Once you have clicked on all the dialog boxes the install will take about 15 minutes.
Once it is installed restart the computer as suggested. In control panels, Display, set screen resolution to 1024 by 768.
Then double click on AutoMateXX.exe to run.

AutoMate control software
(see Screenshot image on page 13)
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. Likewise, "Coulometer Test" check communication with the coulometer.

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 of the functions of the automate. See below for specifics of "System Control." 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 next button down is "Run Control" which starts autoruns. The rest of the buttons (7 in total) control parameters during autoruns. See below for specifics of these autorun parameter buttons.

System control pull down buttons
Most 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 (see below)
"Test Controller Communications" is somewhat repetitive as this is also done in "Get System Status" above but it is also useful other times.
"Test Coulometer Communications" talks with the coulometer and gets the current value from the readout from the front of the coulometer. This is displayed in the box at the bottom center of the screen "Data From Coulometer"

Autorun Parameters
Autorun parameter buttons have pre-set value that should work for most situations. They can be modified as needed. If you find that you need different values AutoMate FX can easily re-compile the software with your specific values so that whenever the software starts you specific values come up.
"% Difference" = This is part of the stability equation that determines if a sample analysis is complete. This equation is based on the rate of increase in the counts of micrograms of

C versus the blank. It is basically the same formula that UIC has in their manual for the coulometer
"Sample Run Time" = Minimum length of time for each sample. Will run longer based on the stability equation. (Dirty cell or poorly operating cell will lead to long runtimes)
"Sample Purge Time" = Time to purge atmosphere from vial prior to coulometer reset and acid injection.
"Minimum Purge Counts" = Micrograms of C that must be recorded during the above purge. If this minimum is not met then the run will stop with an error. This is a safety to ensure that a vial is in place, the needles pierced the septa, the cap and septa on the vial are tight, that the connections to the scrubber and the coulometer are tight, etc. If any of these problems exist the run stops and does not waste samples or pump acid and water all over the place.
"Acid Inject Time" = Time to inject acid. The main requirement here is that the level of the acid in the vial, combined with the water manually added to the sample during loading, 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
"Set Sample Rate" = Frequency of data collection. 1 minute works well

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, save a table, save as, or open an existing table.

Easiest way to get data into the file is to copy the cells with sample ID's and masses from Excel and paste it into the correct cells. Don't paste into the Blank row – It will not allow it. Also you have to use the paste function in the "Table Functions" pull down – right clicking is not enabled.

The long box to the right shows the location of where the data will be stored on the hard drive. Default is a folder called AutoMate data in My Documents. I suggest leaving it there and creating a shortcut to the folder on the desktop. Sample name and weight can be entered directly into the table. Data is exported as .dat files and can be directly imported into spreadsheet programs like Microsoft Excel.

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 (carousel will not turn)
Needle up = needle assembly is in the full up position
Supply pressure = carrier gas pressure is above 5 psi
Vial in position = carousel is lined up directly under the needle assembly
Carousel zero = carousel position 0 is lined up directly under the needle assembly

Carrier gas = carrier gas is flowing
Controller = Automate software is communicating with the Controller
Coulometer = Automate software is communicating with the coulometer

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

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 weight column from the top and finds the first row with a weight in the cell. Then it looks to see if the total counts cell has any data. If there is no data it runs that sample. If there is data in the total counts cell it assumes that the sample has been run and looks for a row with a weight but no data in the total count cell. It will then run that cell. Once all the rows with weights have associated data in the total count cell the autorun ends. During an autorun data is stored into the table and the export file at each sample reading (normally 1 minute intervals).

Solid 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/

Vials can be ordered, or replacement caps and septa or just septa.

Standards are usually 6-10 mg. Samples are usually 10-15mg unless the %C is really low in which case more material can be used. Sample weighing is one of the most important steps to getting good results. Samples can be weighed out in a couple of ways:

1. Use a macrobalance (reads to 0.0 mg) and place the vials upright on the balance and then tare the balance. Powder is the placed in the bottom of the vial until the correct weight is reached. Record weight.

2. Use a microbalance (0.0mg) or a microbalance (0.000mg). 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.

I prefer the second method using a microbalance. Results are much better.

Then cap each vial. The tops 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 raise 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 pen. 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).

Solid samples can be weighed out prior to analysis and stored for a long time as needed.

DIC of water samples

The AutoMate Prep Device can also be used to measure total DIC in waters. 5ml is the recommended volume. Contact Automate FX for more information.

Total CO2 in headspace

The AutoMate Prep Device can also be used to measure total CO2 in headspace for soil incubations. Contact Automate FX for more information.

Supplies

The AutoMate requires three main consumables that must be replenished on a regular basis. These are DI water, acid (nitric, hydrochloric, or phosphoric are all compatible with the materials in the AutoMate - do not use perchloric acid), and a $CO_2$ free gas (ultra high purity $N_2$ or $CO_2$ free air).

I recommend trying 10% phosphoric acid and seeing how it works. 1N nitric and 1N hydrochloric acid also work but see the needle will not last as long (see consumable section below).

Water and Acid are stored in 1 liter bottles with the tubing out through the top shoulder. Water goes in the bottle with the blue tubing and acid goes in the bottle with the red tube. One filling of bottles should last about 6 to 8 samples runs (45 total vials per run). 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 quick connect on the back of the Wet box is not valved. If it is unplugged the contents of the acid bottle will flow out. I fill it with a beaker and a funnel. If you need to remove the bottle it is best to cap it and turn upside down and then disconnect the fitting and wipe up the little acid that leaks. (the water connection is valved) Second, do not tightly cap the water and acid bottles when running the AutoMate. This will cause a vacuum to form and eventually no liquids will flow.

Other more long lasting / durable consumables are Ascarite for the $CO_2$ scrubber, 3% silver nitrate solution for the post sample scrubber, needles, and septa top Exetainer vials. The Ascarite in the trap will last a long time if the input gas is low in $CO_2$. The silver nitrate scrubber will last several runs depending on the amount of sulfur in the samples. 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 acid, 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 (the run will automatically stop). 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 the longer needle to the correct depth in the needle holder assembly (an extra long and short needle are included).

Specially coated needles are available for use with more aggressive acids.

Running samples on the AutoMate

The carousel holds 45 vials total. Vial zero is always reserved for a blank. I suggest 100% calcium carbonate standards in 1,2,3,22,44.

Place samples in carousel. You will have to turn carousel to get samples into the positions right around the needle guide. Use the vial button and Goto vial button.

Enter sample names and weights into the table. Or better yet, cut and paste from excel spreadsheet. Use the "Table Functions" pull down and save the table (or allow the automatic save prompt after run is started).

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 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° 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 (3% silver nitrate scrubber) and connect into flow path.

Prepare coulometer cell and titrate to 29% transmittance. Connect AutoMate post sample scrubber to coulometer cell with Teflon tubing.

Check system status lights (the following should be lit - needle up, supply pressure, vial in position, controller)

Check coulometer communications in system control. Light should go on indicating communication with the coulometer.

Go to "Run Control" and select "Run samples"

Once the needle goes into vial 0 and starts the purge check that the carrier gas flow rate is about 100 ml / min on the front of the wet box. Check that the Blank is in the range of 2 to 6 micrograms of C and the standards are within your acceptable range.

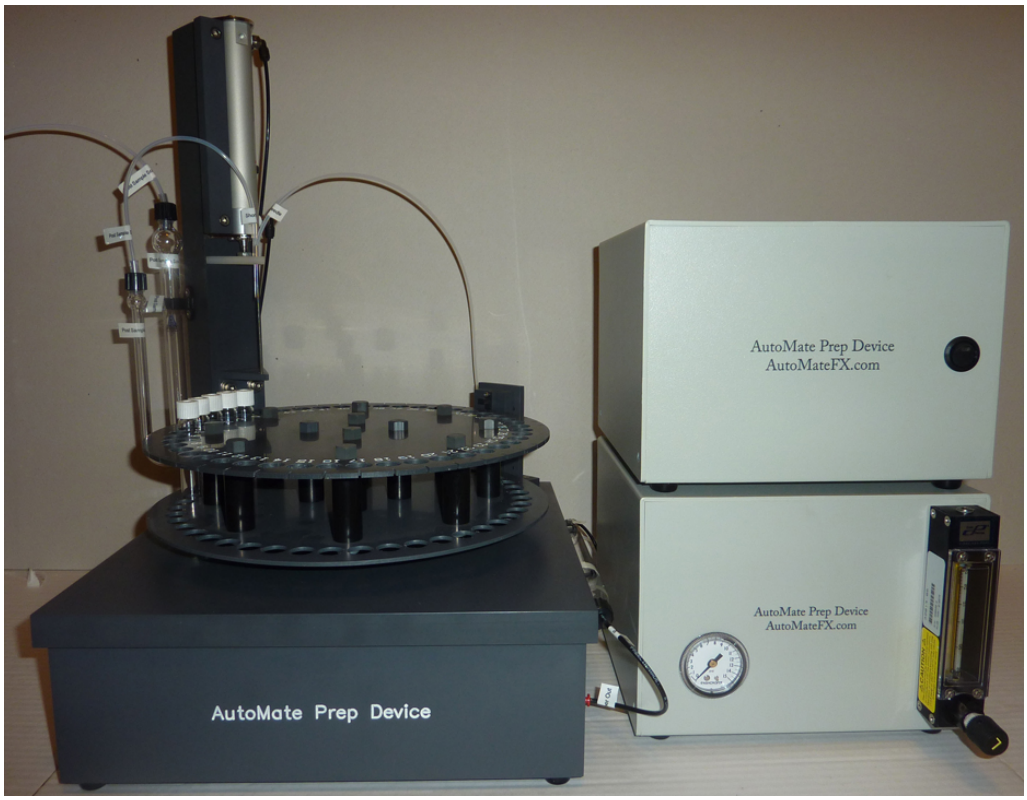
Very short suggestions for setting up the coulometer.
Additional info can be found in the manuals for the Coulometer.

Setting up 5011 and 5012 coulometer

1. Turn main power on to Coulometer (upper switch, not cell current switch)
2. Get clean coulometer cell and stir bar and platinum electrode and silver anode
3. In hood, with gloves and eye protection, fill larger portion of cell (cathode) with Carbon Cathode solution (larger gallon bottle) to between 125 and 150ml.
4. Using a funnel place about 1/4" of Potassium Iodide in smaller portion of the cell (anode)
5. Fill smaller portion of the cell with Carbon Anode Solution to same level as larger side
6. Place platinum electrode in cathode side with the electrode and tube away from the anode and perpendicular to the long axis of the cell
7. Place silver anode in cell (avoid silver touching KI or glass)
8. Gently place cell in coulometer (confirm stir bar)
9. Plug in wires to same colors and attach inlet tubing
10. Rotate cell to get maximum transmittance (%T), then adjust transmittance to 100% Make sure Pt electrode and tube are as far back from light path as possible. Do not tilt rubber stopper backward as this will put the Pt electrode and tube forward.
11. Reset coulometer and turn cell current on
12. After a few minutes transmittance will drop to 29%, color will change and total μgC should be about 1200-5500μgC (depends on the age of the Cathode Solution).

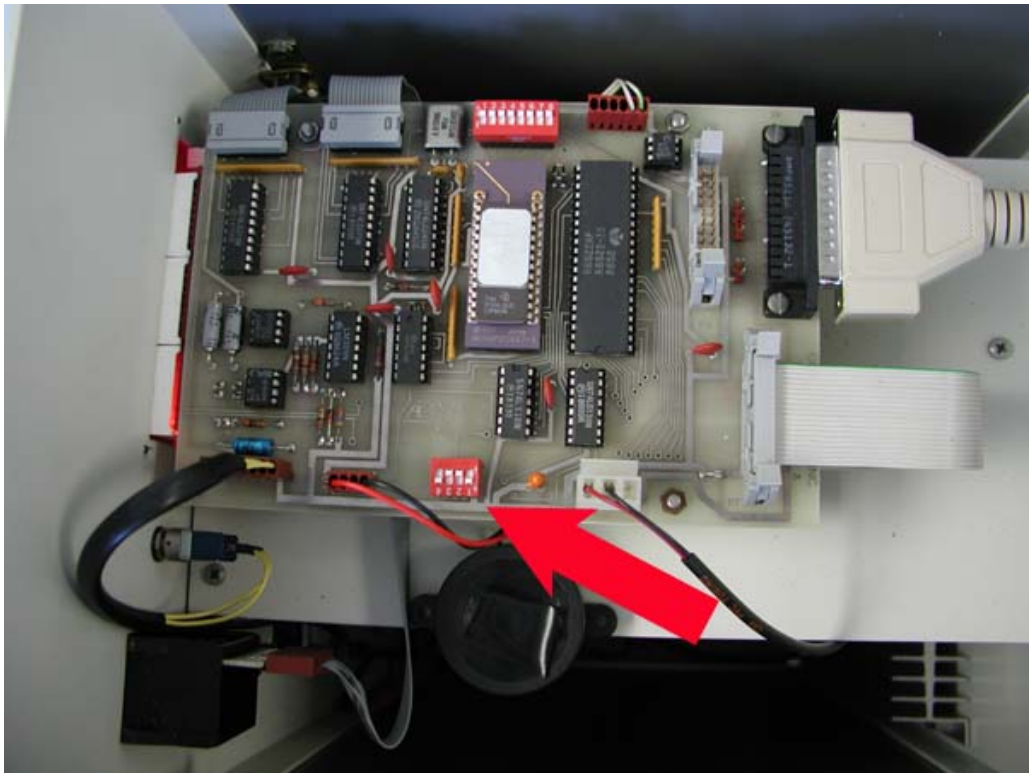
Post run

1. You should be able to do at least 2 complete runs on one set of coulometer chemicals
3. Turn off cell current
4. Unplug wires and tubing from cell
5. Remove cell from coulometer
6. At sink, remove cathode electrode and anode electrode and rinse each with DI water, use metal bottle brush handle to get stir bar out
7. Take #4 stopper and plug anode side and dump cathode solution in to cathode solution waste bottle in hazardous waste collection site. Rinse cathode side of the cell with DI water . Then place stopper in sink.
8. Dump anode solution and KI in to anode solution waste bottle in hazardous waste collection site
9. Rinse stopper and cell in sink with DI water. Use brush and soap to clean cell and stir bar. Rinse again and place stir bar in cell and place both in oven to dry.
10. If cathode electrode is not shiny, place in 12N nitric acid for a few minute. Then rinse with DI.

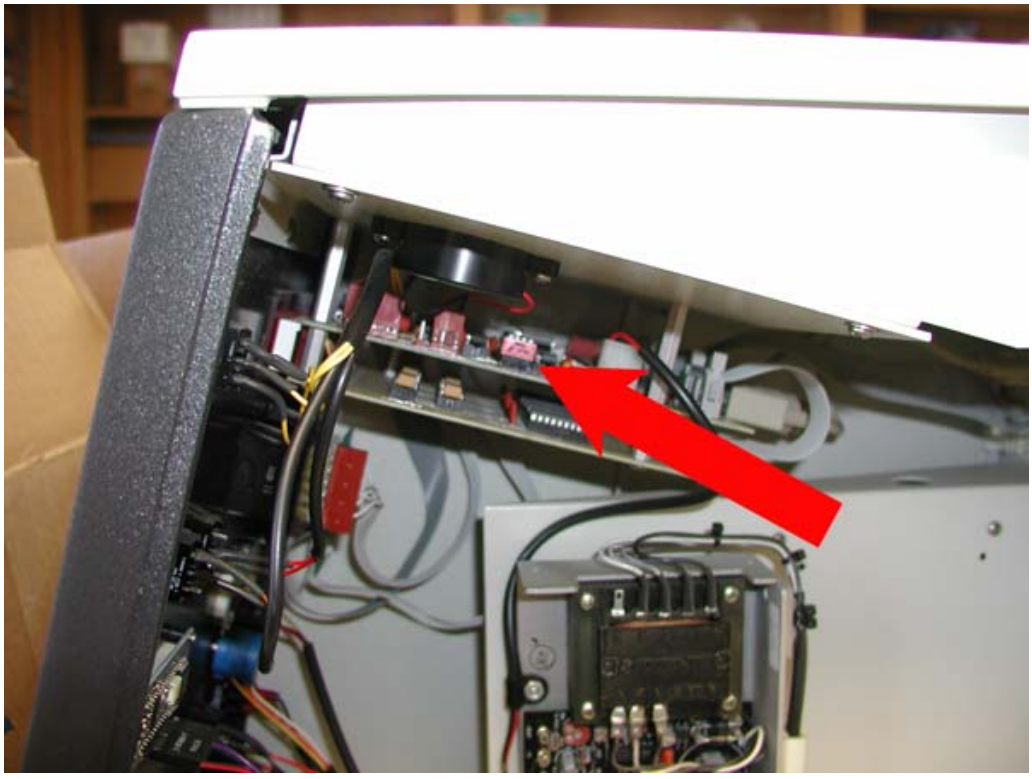



AutoMate positioning

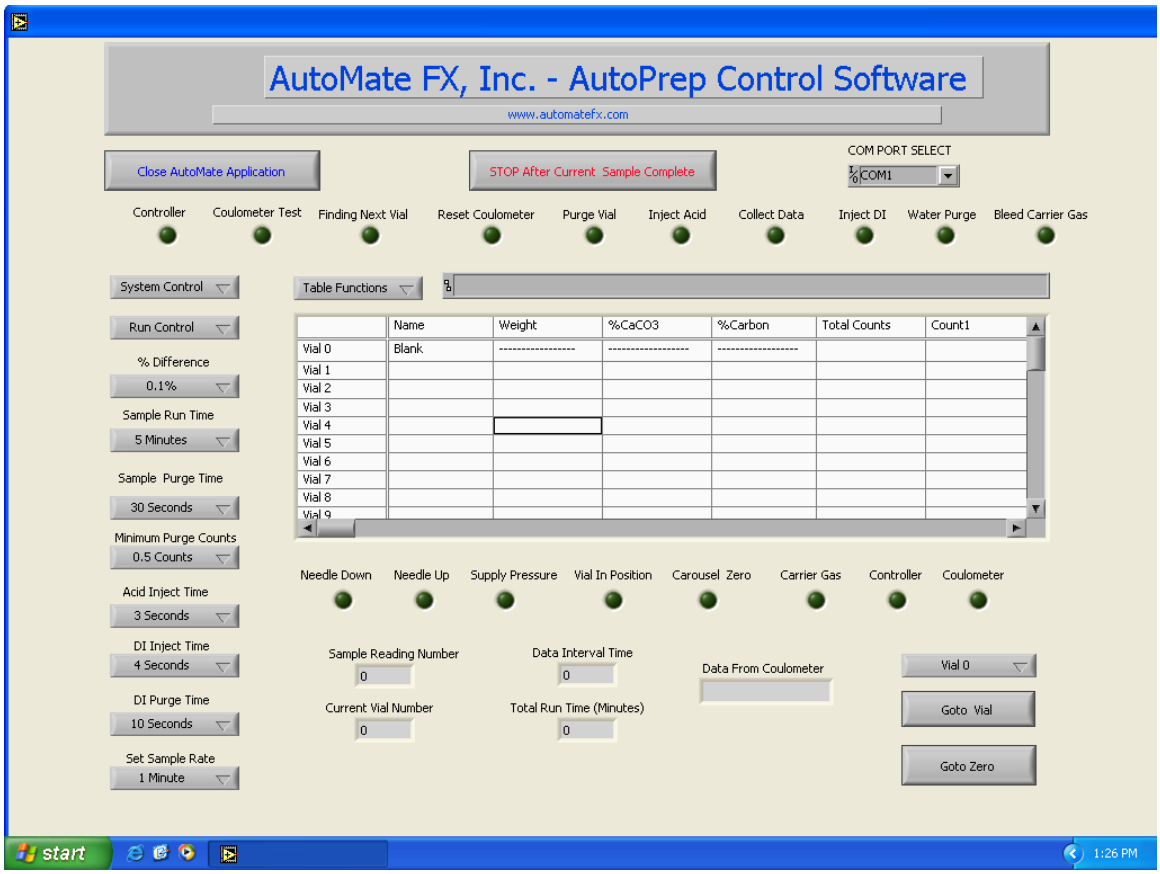

AutoMate tubing (Old version of Wet Box and Control Box shown)



5011 communication switches



5012 communication switches



AutoMate Software Screenshot

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 I sent to you 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.