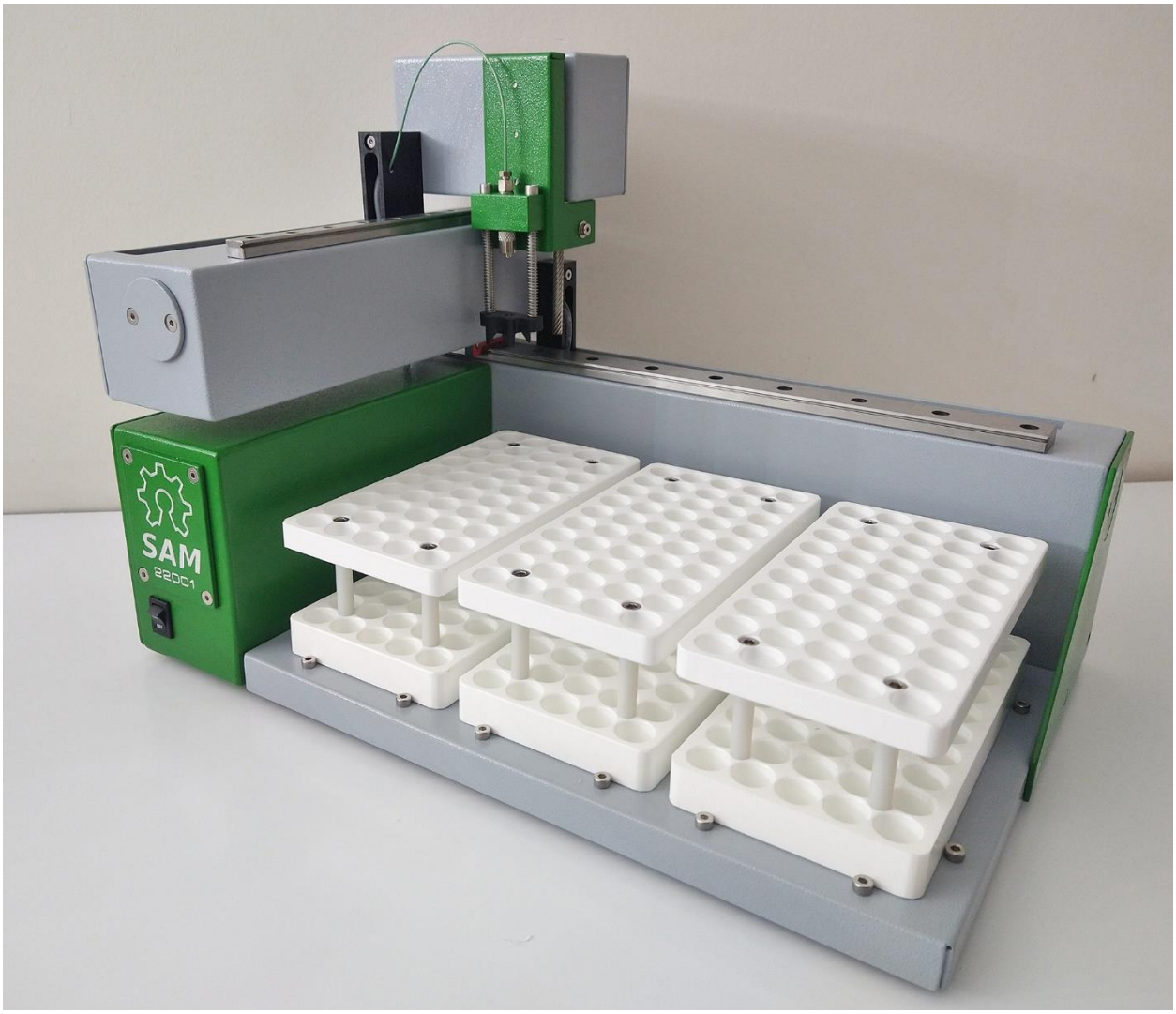
OpenAutoSampler

SAM for Picarro isotopic gas analyzers

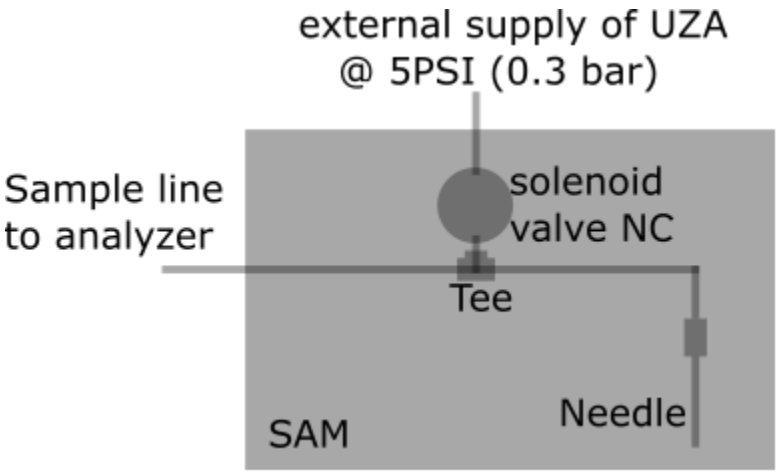
SAM is designed as an affordable and fully open source autosampler for use with Picarro isotopic gas analyzers in combination with Labco 12ml Exetainer vials (5.9ml optional). The electronics and software are based on open source 3d printers.

SAM can be used instead of the SSIM or SSIM2 by Picarro. SAM is tested with Picarro's G2201-I, G5131-I or G5102-I analyzers, but SAM should be compatible with any Picarro analyzer that is compatible with the SSIM or SSIM2.



Basics of SAM

SAM is basically just a needle attached to a piece of tubing. A tee is installed between the needle and the analyzer. A solenoid valve is attached to the tee and supplies Ultra Zero Air.



Analysis of a sample follows these steps:

1. The needle moves to a position above the vial to be analyzed. The needle is not inserted in the vial yet.
2. Pre-Flushing: A solenoid valve is opened and Ultra Zero Air (UZA) flows towards the analyzer and out of the needle. This flushing cleans the sample line and flushes all gas of interest (CO2/CH4/N2O) out of the analyzer. Recommended time is 120-180 seconds.
3. Sample introduction: The solenoid valve supplying UZA closes. The needle is immediately inserted in the vial. Gas from the vial flows into the analyzer. Sampling time can be set to any length of time (5 to 15 minutes is recommended).
4. Post-Flushing: The needle is retracted from the vial and air from the laboratory flows to the analyzer. The atmospheric air with the CO2, N2O and CH4 flushes the sample out of the analyzer. This step is important to give the analyzer time to tune the lasers. As a side-effect this also reduces carry-over between samples. Recommended time is 90 seconds.

Pre-install checks

Leak check your analyzer before connecting SAM! It is possible that the analyzer has developed leaks during shipping or previous operation. SAM (or the SSIM2) can only operate with an analyzer that does not have significant leaks!

A small leak in the analyzer might have gone unnoticed if the analyzer was used with large samples stored in gas bags or pressurized cylinders.

Install a shut-off valve at the inlet of your analyzer. Connect a gasbag or tank (regulated to less than 5PSI or 0.3BAR) filled with UZA (or N2) to the shutoff valve.

(insert picture of a suitable setup)

Let gas flow though your analyzer and wait until the concentrations of CO2, CH4 or N2O stabilize. (a minimum of 5 minutes).

Slowly (over a minimum of 30 seconds) close the shut-off valve at the inlet of your analyzer. Monitor the concentration of CO2, CH4 or N2O. Monitor the CO2/CH4 or N2O concentration. The concentration should not significantly rise by more than 10ppm CO2 or 10ppb N2O over the next 10-20 minutes.

There is a leak if the concentration keeps rising towards ambient**. Find the leak before tightening any fittings! (See troubleshooting below)**

Install of SAM

Minimum install

A minimum install only requires a tank of Ultra Zero Air. If you plan to use an Ultra-Zero Air generator, make certain that it removes all gasses of interest. Most UZA generators only remove hydrocarbons and leave CO2 and N2O in the gas stream. **The pressure of UZA needs to be regulated to 5PSI or 0.3BAR**! A higher pressure can damage the analyzer. A 0-100PSI regulator is not suitable, it is much safer to find a 0-20PSI regulator. The UZA supply is connected using 1/8" tubing to SAM.

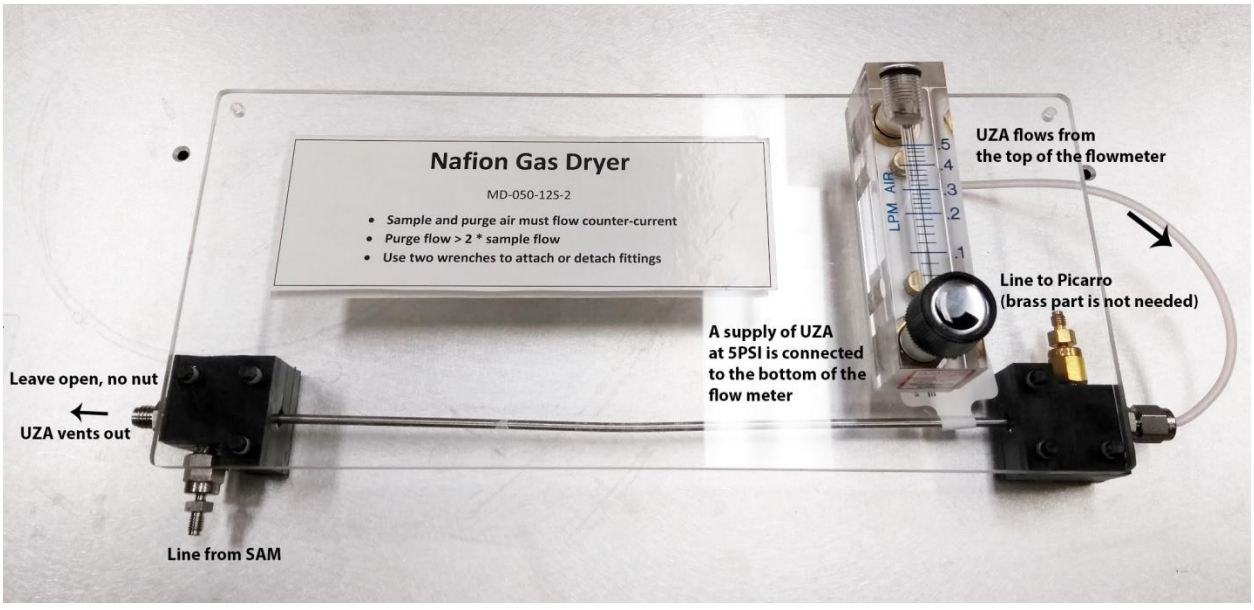
The 1/16" sample line from SAM is connected to the analyzer using a Swagelok adapters.

Recommended install for 2201-I (CO2 + CH4) and 5102-I (N2O)

The Picarro instruments can be damaged by water (H2O) condensation in the instrument. This can occur if there is a sudden power-outage when the analyzer is filled with sample that contains a high concentration of H2O.

There also is a possibility of degraded accuracy when samples with high concentrations of H2O are analyzed.

H2O can be removed from the sample by using a membrane dryer (Nafion tube) in between SAM and the analyzer. A tested Nafion dryer is MD-050-12-2 by PermaPure. The Nafion membrane needs a flow of dry gas in the opposite direction of the sample flow of about 100ml/min. (see figures below)

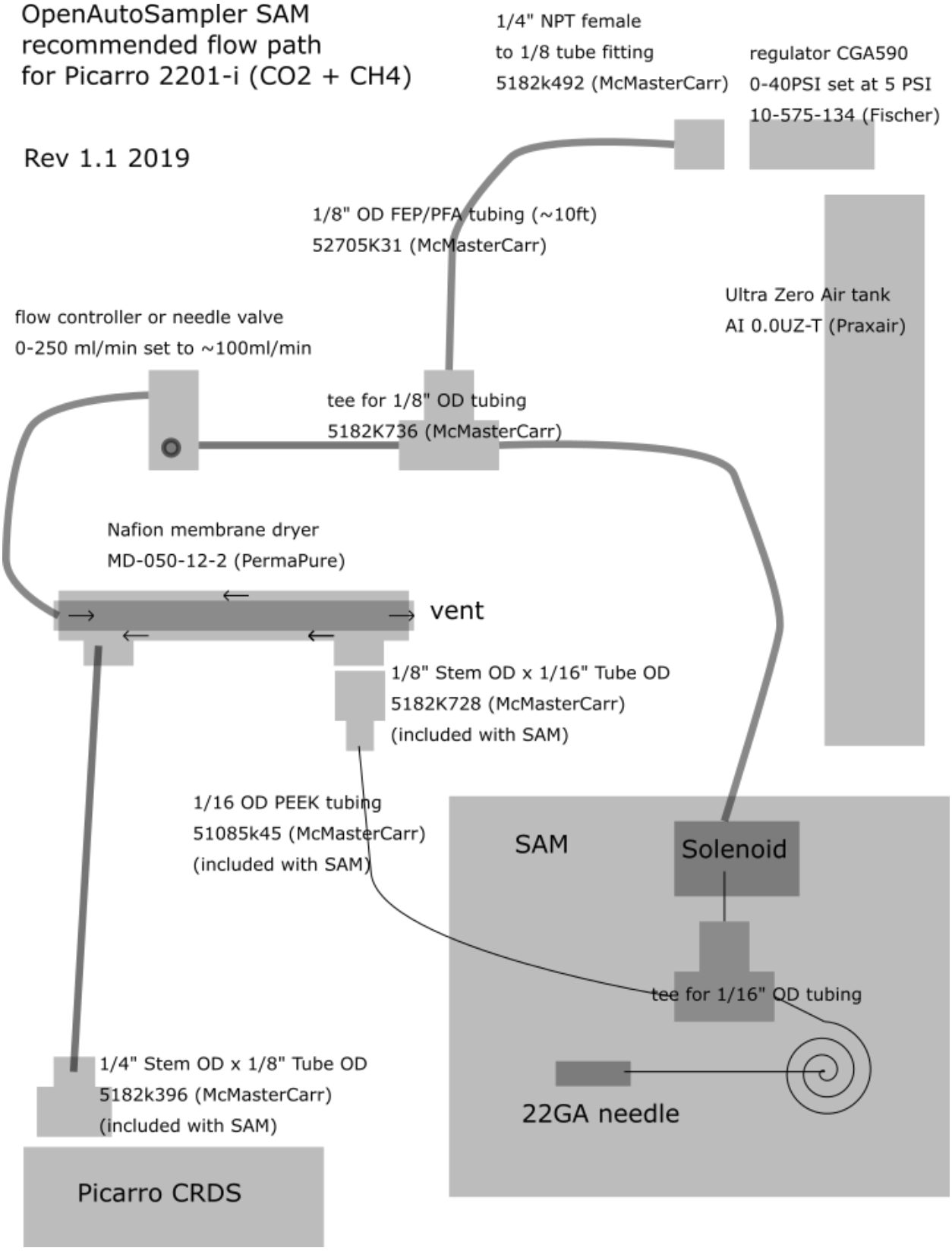


(the Nafion in this picture is mirrored from the one in the schematic)

OpenAutoSampler

OpenAutoSampler SAM recommended flow path for Picarro 2201-i (CO2 + CH4)

Rev 1.1 2019



Recommended install for 5131-I (N2O with d18O) and 5102-I (N2O without d18O)

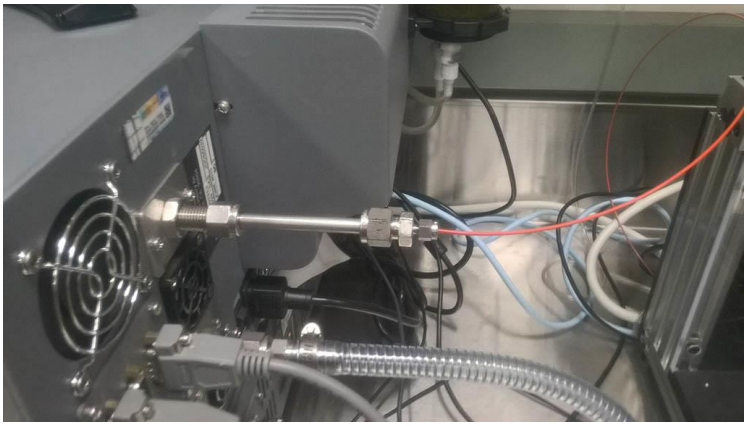
The newer Picarro 5131-I analyzer has a built-in Nafion dryer. No external Nafion dryer is needed. The older 5102-I analyzer has no Nafion dryer built-in, follow the schematic above to add one.

The N2O analyzers suffer from interference from:

- High concentrations of CO2 (1000ppm and above)
- CO at any concentration above ambient. CO is released by all rubber septa tested. The effects are potentially massive!
- H2O (the built-in Nafion in the new analyzers removes H2O)
- Acetylene (no solution yet)

I recommend using a filter to remove all interfering compounds. An effective filter can be created by filling a 10cm (4in) long piece of stainless steel tubing (1/4" OD, 3/16" ID) with 4 pellets of 1% platina on alumina (Sigma Aldrich # 232114) and filling the remaining space with Soda Lime.
The platina converts the CO to CO2, the Soda Lime removes all CO2. The filter is installed directly at the inlet of the analyzer. This filter lasts about 48hours.





Software installation

Driver

SAM uses a Einsy RAMBO control board (https://reprap.org/wiki/EinsyRambo). The driver is available through https://ultimachine.com/products/einsy-rambo-1-1 .

- Download driver and unzip in known location
- Go to Device Manager in Windows and look at the list of devices
- Plug SAM into any USB port on the Picarro analyzer
- Look at which new device pops up
- Right-click on the new “unknown device”
- Select manually browse for driver, select the location where the driver is stored
- When the driver is installed the RAMBO board should show up as a COM port .
- Write down the COM port (“COM9” for example)

Control Software

- The control software should be compatible with the Python version installed on the analyzer.
- Copy the contents of the folder “CONTROL_SOFTWARE” on the USB-stick to C:/SAM/
- SAM_auto_control.py should now be in C:/SAM/SAM_auto_control.py
- Open SAM_auto_control.py in notepad++
- Change the setting for COM port to the right COM port (from Device Manager)
- The control software should now work, test by double clicking C:/SAM/SAM_auto_control.py
- If the software doesn’t open a user-interface:
    - Start a command-prompt: Start-> run -> cmd.exe
    - Browse to c:/SAM/
        - c:
        - cd SAM
    - Try starting the script by typing it’s name: “SAM_auto_control.py”
    - Look at the error messages…

Example data handling scripts

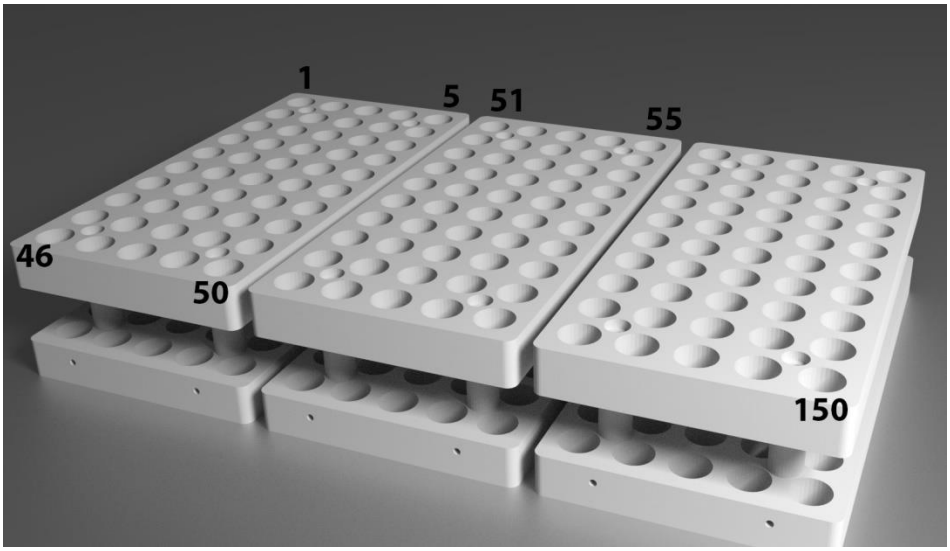
A script is used to extract the sample data from data files stored by the analyzer. It should be located in c:/SAM/DATATOOLS

Analyzing samples

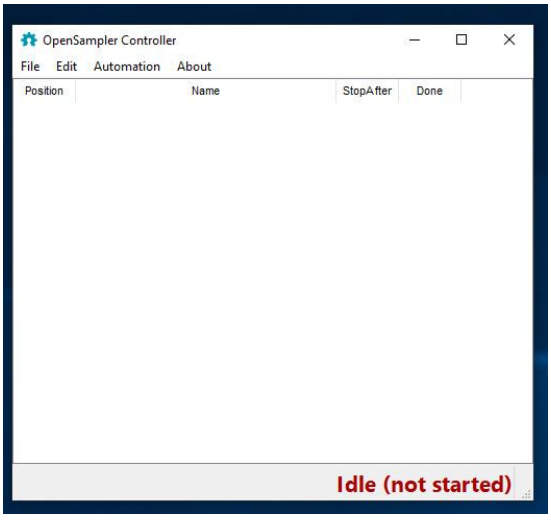
SAM is designed for use with 12ml Exetainer vials. See https://www.labco.co.uk/products/double-wadded-exetainer-cap/product/586-exetainer-12ml-d-w-white-capped-x-1000/category_pathway-39.

The 12ml vials can be filled with 20-35ml gas sample.

The vials are loaded in the supplied trays. The default numbering is as shown below.



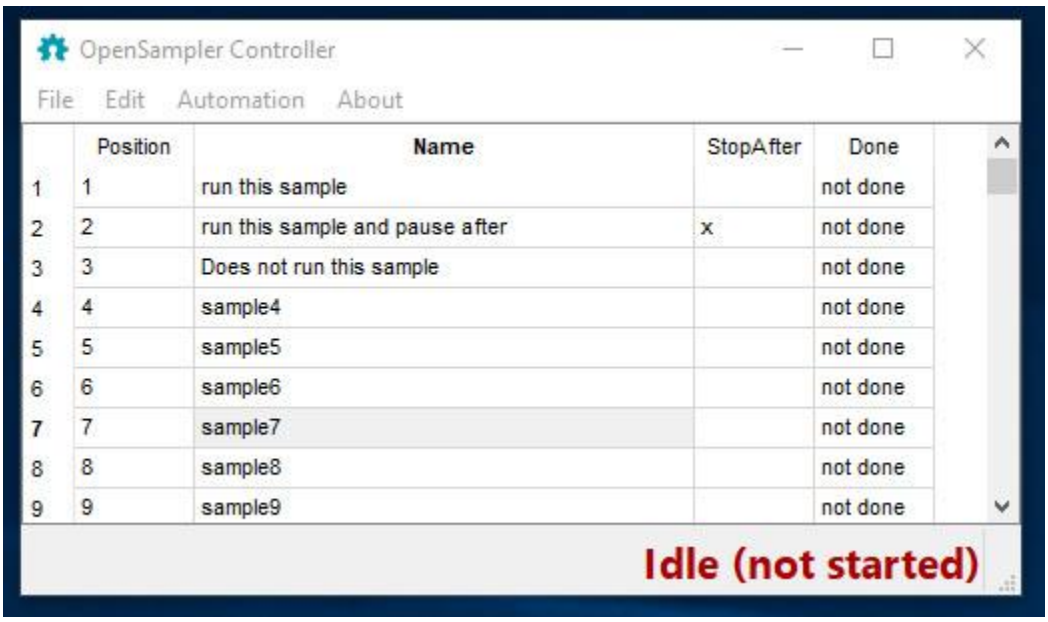
Start the AUTO software:



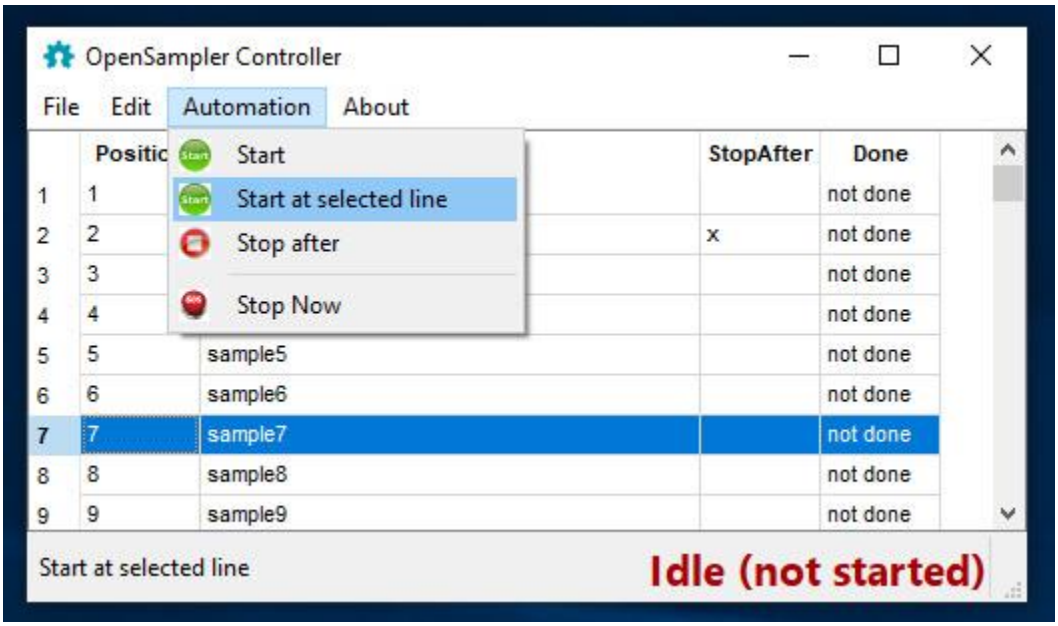
Create a sample list: File->New and type how many samples you want to start with.

Samples can be analyzed in any order. Samples can be changed, added and removed when SAM is running. There is a column “StopAfter”, if anything is typed in this column SAM will pause after that

sample has been analyzed (see figure).



A samplelist can be started or re-started at any position by selecting the line to start at and clicking Automation-> Start at selected line. In the figure below SAM will start at line 7.

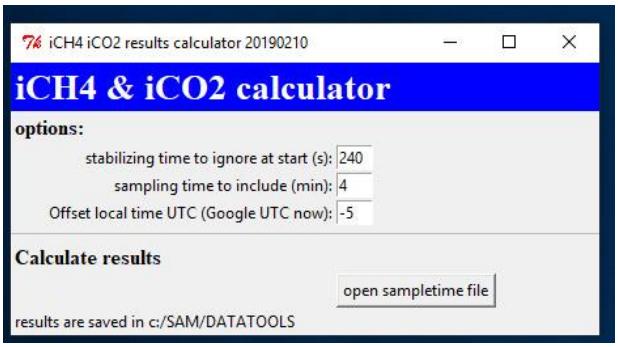


SAM will stop when the last sample of the list is analyzed or when StopAfter is activated for a certain sampleline.

Summarizing results

SAM comes with “example” data handling scripts (written in Python 2.7). This means that it cannot be guaranteed that these scripts work “out of the box”. Each analyzer is setup slightly different. Data that is logged can be customized for each user. Support can be given if remote access to the analyzer can be provided.

Start the results calculator located in C:/ SAM/Datatools



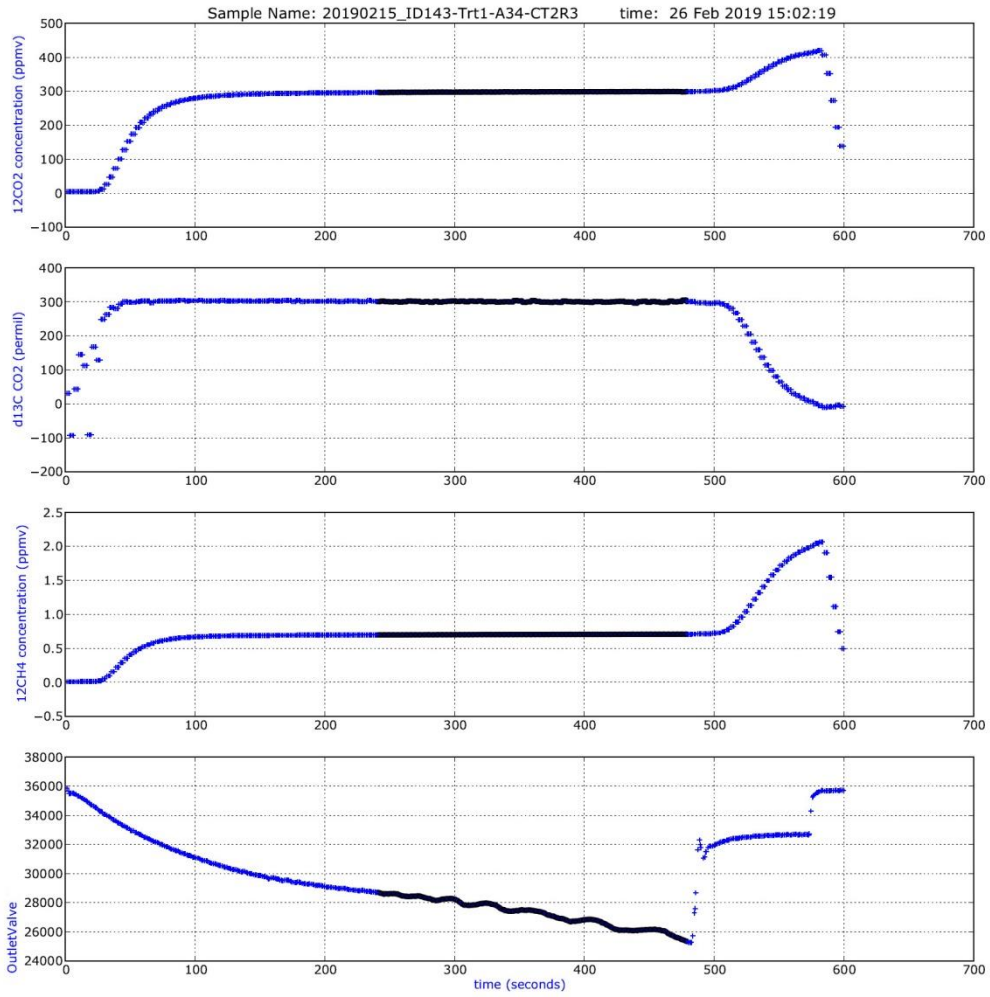
- Picarro analyzers save the data with a timestamp in UTC (universal time). So set your computer time offset compared to UTC.
- Select how much time to ignore at the start of the sample. (240s)
- Select how much time to include in the calculations (4-30min)

Click the button and select the file of the day you would like to get the results from.

The calculator will try to find the .DAT datafiles in c:/UserData and extract the part where the samples were measured. Four result files will be generated:

- Cleaned up raw measurements covering the timespan of all selected samples: 20190227_230231_merged_results.txt
- Charts for each individual sample, including pre-flushing and post-flushing: 20190227_230231_charts_whole_run.pdf
- Charts with only the points included in the results calculation (excluding flushing and stabilizing time): 20190227_230231_charts_sample_only.pdf
- The summarized results for each sample in a .csv file: 20190227_230231_results.csv

20190227_230231_charts_whole_run.pdf:



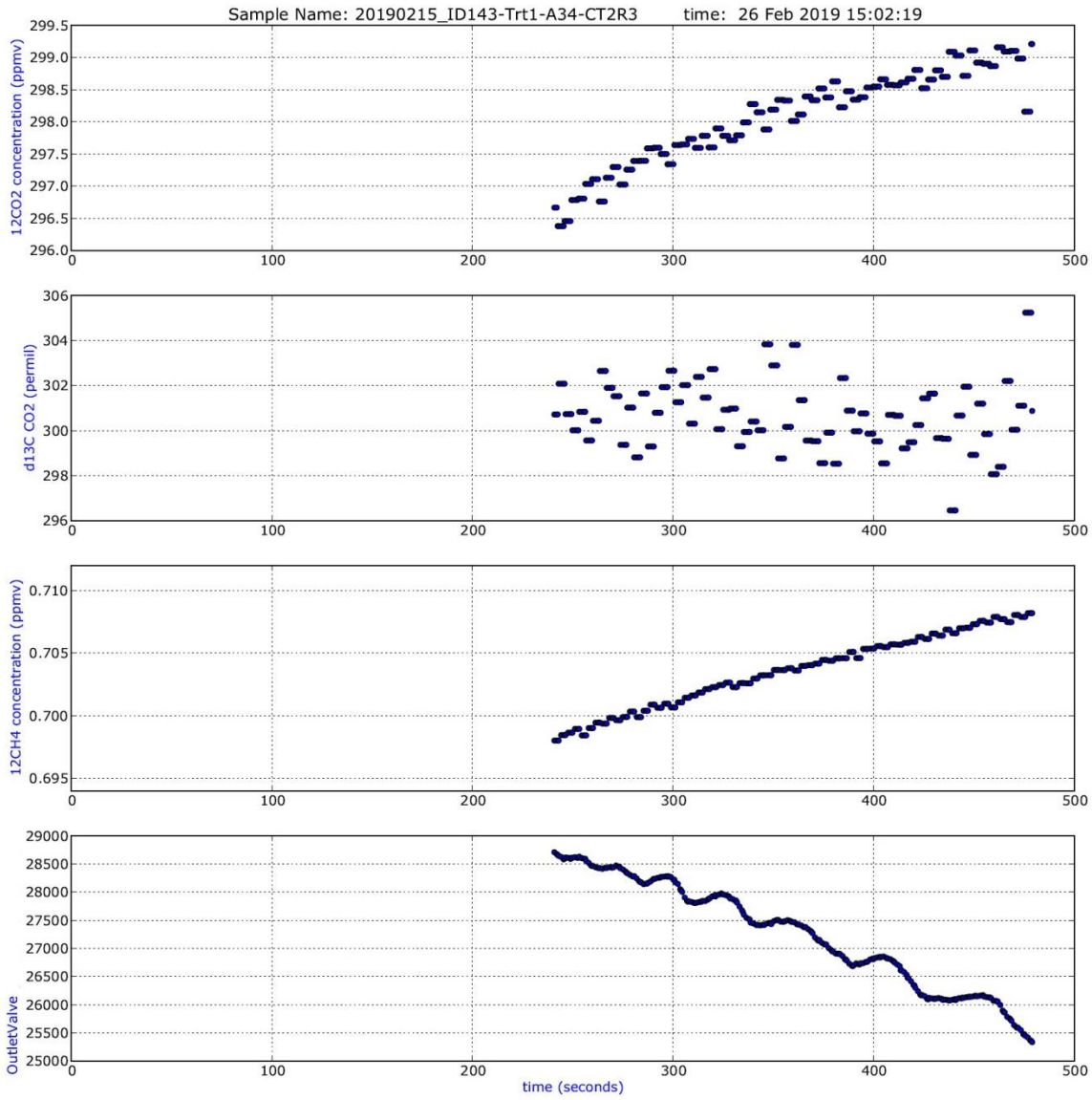
These charts show all data recorded from the moment the needle was inserted in the vial. The dark area is the time segment that is used for the calculations.

At the top 12CO2 concentration is shown. It can be seen that the concentration starts at ~0ppm because the analyzer was flushed with UZA. After 20 seconds the sample reaches the cavity and the concentration increases while the sample is filling the cavity. At time 240s the concentration is considered to be stable and the data is now used for the calculations. At time is 500s the needle is retracted from the vial and air from the lab starts to fill the cavity.

The bottom chart shows the position of the OutletValve (a proportional solenoid valve between the cavity and the vacuum pump). OutletValve value is an indicator for the pressure at the inlet of the analyzer. This value is used for troubleshooting. If OutletValve for one vial is much higher than for other vials it means that this vial does not hold vacuum (vial is leaking). If OutletValve is much lower than normal it probably means that there was no sample in the vial.

OpenAutoSampler

20190227_230231_charts_sample_only.pdf



These charts show only the data points being used in the calculations.

In the top chart it can be seen that the 12CO2 concentration is not completely stable during analysis. In this case about 25ml sample was injected in a 12ml Exetainer vial. 20-30ml of gas in the vial is not quite enough to completely flush and fill the cavity of these Picarro instruments. The measured concentration will be 2-3% below actual concentration (comparable to the SSIM2 by Picarro).

20190227_230231_results.csv

Will show mean values for data of interest. It will also show slope of the measurements. If the slope of one particular sample is significantly different from other samples it could indicate a problem (leaks or interference!)

Modifying analysis settings

SAM uses Python 2 scripts for control. Before editing the scripts always make a backup copy. The script can be edited using Notepad++.

Modifying data summarizing scripts

SAM comes with “example data handling scripts”: these scripts might work for your research, or they might need to be changed to summarize the data keys you are interested in. Before editing the scripts always make a backup copy. The scripts can be edited using Notepad++.

Basic support on these scripts is provided by email. Extensive customization of the scripts will involve programming fees.

Troubleshooting

Test the vacuum in the vials: the bubble test

It is important to test the vacuum in the vials that will be used. An easy way of testing the vacuum is the bubble test:

- Take 10 evacuated Exetainers
- Take a needle (no syringe) (22Ga works)
- Hold the Exetainer under water, and put the needle in the septum
- Wait for the vial to fill with water
- Repeat for the other Exetainers.
- Look at the size of the bubble left in the Exetainers
- The bubble should be no larger than a pea, and equal in size for all Exetainers
- See attached a picture as a reference (by Nicolas Belanger lab @ Teluq)

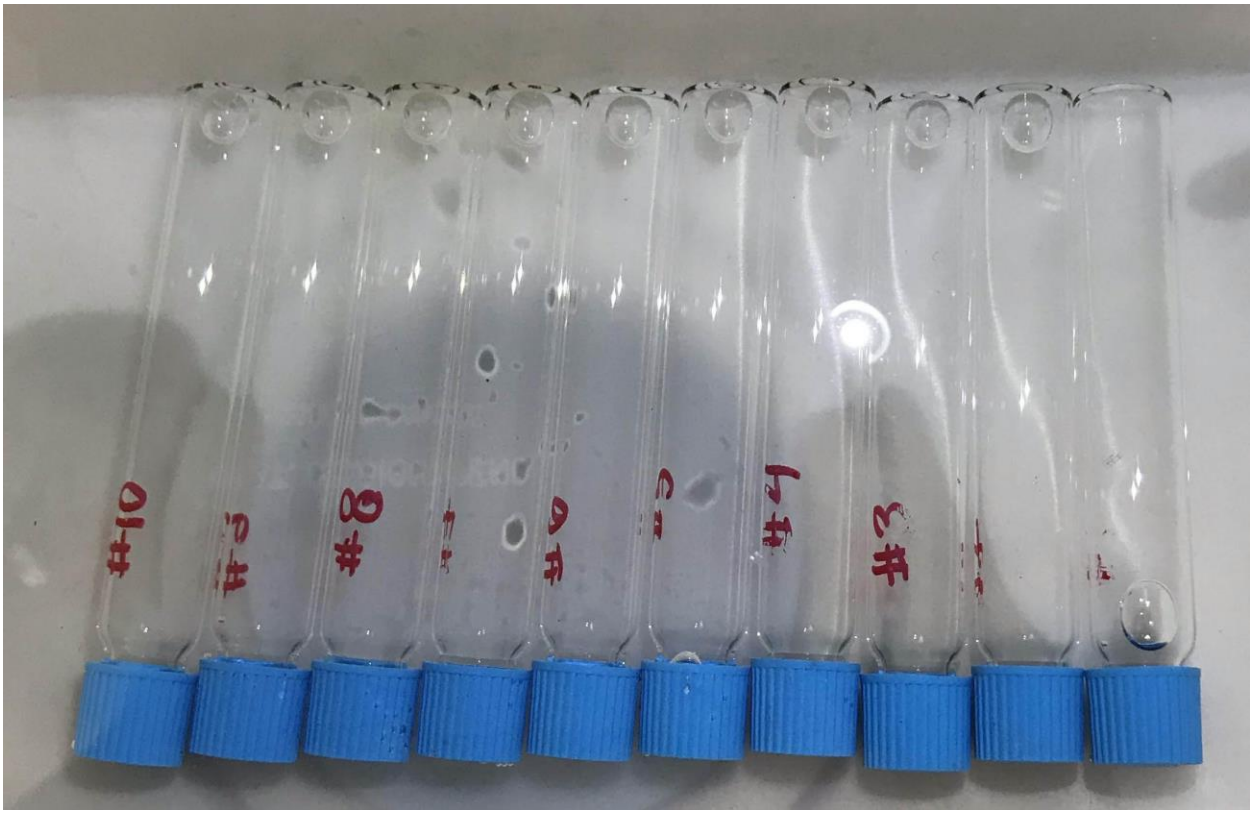


Figure: vials filled with water, showing equal small bubbles-> good vials (credit Nicolas Belanger)

Caps and septa

We recommend using double septa in the caps: double wadded caps. In our experience this gives a better vacuum in the vials and repeatability seems better.

It is very important not to over-tighten the caps. The butyl rubber septum will be pushed into the vial. Holes from previous piercings of the septa will open up. It is likely that the vial will leak. It is also possible that the needle of SAM will bend when the caps are too tight.

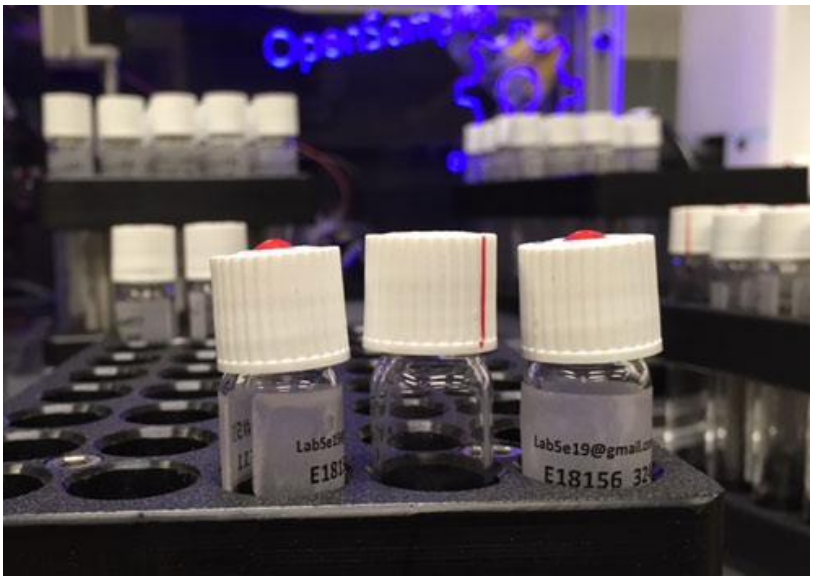


Figure: left and right vial have over-tight caps, middle vial is fine. When over-tight the top septum bulges up, the bottom septum get pushed into the glass vial.



Figure: holes in bottom septum (from previous piercings) open up when cap is over-tight.

Leak tests

Fill an evacuated vial with UZA. Place in rack and analyze with SAM. The concentration should stay below 20ppm during the whole analysis. (It will never be 0PPM, the vials always contains some CO2. As well there will be some ad/de sorption of CO2 from the sample system). For N2O the concentration should stay below ~20ppb.

If the concentration increases too much, there is a leak in the system. First try to tighten the needle (pliers).

You can use high concentration CO2/CH4/N2O as a tracer and let it flow for at least 20 seconds over each fitting to find the leak (do this while the needle is inserted in a vacuum vial or solid piece of rubber).

(add figure as example of leaks).

Finding a leak in SAM, tubing towards the analyzer, and the analyzer itself

(For N2O analyzers: in CRDS Data Viewer click Settings->Change GUI mode from Standard to Service. The password is “picarro”. Select “co2_raw (ppm)” as one of the data keys to chart.)

Find a drinking straw or a piece of tubing. Starting at the fitting/connection furthest away from the analyzer. Slowly blow your breath over the fitting for about 20 seconds. Wait and monitor the CO2 concentration displayed by the analyzer for at least 20 more seconds. If the CO2 shoots up to 2000-5000ppm, you have found the leak. If the CO2 just goes up by a few hundred ppm the leak is close by. Work your way past all the fittings to find the point where the CO2 response is highest.

If the leak seems to be right close to the analyzer: use the straw to blow a lot of CO2 right in the housing of the analyzer. Do this close to the inlet of the analyzer. If there is a very large response in measured CO2, there is a leak right in the analyzer. Contact Picarro Support for further steps.

Maintenance

- Clean the linear guides every 6 months using a lint-free rag
- Lubricate the guides after cleaning with a few drops of light oil (sewing machine oil or similar)
- Replace the needle after every 1000-5000 samples. Leak-check the system after replacement of the needle!

Safety precautions

Pinch hazard: Keep hands clear of SAM at any time when SAM is powered.



SAM uses a supply of compressed gas. Compressed gas is dangerous! Follow all local regulations.



Do not use SAM for analysis of flammable or explosive gas mixtures



SAM is not suitable for use with samples that are corrosive or contain liquid acids. (Besides SAM, the Picarro analyzer might be damaged as well!)



SAM uses a needle to take gas out of vials. Used needles are a biohazard! Dispose of needles responsibly! Follow your local regulations

