

*A guide for running Spock..*

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*Overview*

Spock is a Micromass dual inlet mass spectrometer that measures the stable isotopes of carbon and oxygen in atmospheric CO2. Spock primarily runs samples from flasks and calibration tanks on the NOAA carbon pathway, or the CMDL Cooperative Air Sampling Network. Spock is designed to measure the isotopic ratios of 45/44 and 46/44 of CO2 in samples. Spock measures three masses: Mass 44 with Species 12C16O16O, Mass 45 with Species 13C16O16O and Mass 46 with species 12C18O16O. Pure CO2 must be extracted cryogenically from the air stream to isolate the stable isotopes within the air sample. We use a vacuum to pull air through a chilled ethanol bath held at ~-90ºC to freeze out water, and then through a “triple trap”(Figure 2) held at ~-200ºC with liquid nitrogen to freeze out carbon dioxide. Remaining gasses including oxygen and nitrogen are vented away. The triple trap is then heated to -20 ºC and the sample is released into the sample bellows of the mass spectrometer to begin analysis. The reference bellows is filled with CO2 from Niwot Ridge, west of Boulder, at the end of every run. Aliquots of the sample and reference gas are released into the source iteratively, where they are ionized and transported down the flight tube to be separated by their appropriate masses, collected in Farraday cups. The final step is extreme data crunching using excel macros.



# Figure 1. Spock’s extraction system.



**Figure 2.** The triple trap. Air is pulled through the tubing, which is within an open-bottomed cylinder and immersed in liquid nitrogen at -200 C. After the extraction, a vent at the top of the cylinder closes, the trap is heated by the wiring wrapped around it, and the vaporized nitrogen displaces the liquid nitrogen. The sample is warmed to -20 C, and is released to the sample bellows.

*Starting from a finished run..*

1. The run is finished when the message log reads “Finished Autorun”.
2. Run a bellows refill before you start the next run (should take about an hour). Go to Program>Run>**TKBelRefill2**>Run.
3. Turn off the liquid nitrogen (saves LN2 and allows the doer to defrost, may need to make sure it’s not frozen if humid out) (**Figure 2.**).
4. Restart the computer to clear the memory. Press shut down and when the message pops up on the screen press cntr. Alt. delete to restart.

*Transferring the run to NOAA database*

1. Close the mass spec software on the upper left hand corner by double clicking and saying ‘yes’, you wish to exit.
2. Open the Data file and right click on the SPOCK.TXT file, click Settings>General>Change the name to the current run number ###.TXT.
3. Left click on the file you just renamed and click Copy>A-drive.
4. Wait until the green light on the floppy disk drive turns off then it is safe to take out the floppy disk.
5. Insert the floppy disk in Carbonito and copy the TXT file from the floppy disk to the TXT files folder on Carbonito.
6. Open Spock CO2 crunch 1.3 and crunch the current run by pressing ‘Spock CO2 crunch’ and typing in the appropriate run number when in the macros prompt.
7. Click do not save to all questions except when the program asks if you would like to save in the format, then you click ‘save’.
8. Be sure that all standard deviations between reference, trap and internal and external calibration tanks are below reference limit (0.04 for 13C and 0.08 for 18O), along with flask pair differences (same as on Amos and Tpol).
9. Be sure to check the performance file and that the 13C 18O data is congruent with past data. You will need to **copy the performance data** from the current crunch and **paste to the performance file** and save. If these values are off there could be issues with the mass spec such as a leak or contamination somewhere down the line.
10. Save the file to your GoogleDrive to transfer the files to the NOAA database.
11. To transfer the data to the NOAA database you must sign into Pulse secure and transfer the o1 and crunched files.
12. Sign into **vortex2.cmdl.noaa.gov** username: **sil** and password on FileZilla. Find the raw file in the directory under **projects/co2c13/flask/o1/transfer**. Drag over the files from the computer on into the transfer folder on the network.

*Preparing the next run*

1. Close all stop-clock valves on the flasks if you have flasks loaded. Close tanks if you are finished running them (we like to run calibration tanks ~30-40 times). Be sure the valves are closed on the software and then you may unload the flasks and send them on their way to the appropriate location along the carbon pathway (VOCs, CH4C13, C14, H2, back to NOAA).
2. Setting up a new run>**Session Builder** <http://om.cmdl.noaa.gov/sb/> online>**username:sil password:20020118**, always start a run with 4 Reference tanks, in this case the reference tank for Spock is CARR, then you can have some arrangement of a maximum of 28 samples in between the first set of four references and the second set of four references. Following the second set of four references you must add four trap tanks, in this case, MOLL. Following that can a maximum of 28 more samples and concluding with ref set of four ref tanks. Be sure that the port number matches with the flask port that is specific to each flask or tank. Be sure the port names are capitalized when you enter them into session builder.

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| 4 Reference tanks: CARR |
| Maximum of 28 samples, flasks and/or tanks |
| 4 Reference tanks: CARR |
| 4 Traps tanks: MOLL |
| Maximum of 28 samples, flasks and/or tanks |
| 4 Reference tanks: CARR |

1. Copy data that pops up in the new tab and paste into any old txt file. Save as the new **runnumber.o1** onto **Disk Drive A** (floppy disk). Wait for the green light to turn off and eject to floppy disk to upload the newest session onto Spock.
2. Open A drive on Spock, copy the newest run by left clicking>Copy>Drives>C drive>NOAA CO2.
3. Open DI version 2.47>OS/2 window and change to the DI2.47 directory by typing ‘cd isotech’, enter, then ‘cd DI2.47’, enter, then ‘make2 “runnumber.o1” and enter. Once it says download successful, you can exit out.

*Tuning*

1. Sign into the application with username **SPOCK**, password **c13o18**.
2. Run a center scan. You can do this with **control C**, or by going to the **Tests** menu, and choosing **Center Scan**. This represents the beam that is recorded across a spectrum of accelerating voltages. The center of the scan will be recorded as the “HT” in the upper right dialog box.
3. Using the **Raw data** option in the **Cursor** menu, record the “time” (x axis) values at each end of the flat peak. The difference is the peak, and you want to maximize (lengthen) that peak.
4. Fine tune the peak by adjusting several parameters: HP, ZV, IR, EX, and EV (explained below). You should tune in this order. Run scans of each by going to the “Run Scan Parameter File” and choosing the appropriate scan. You can change the parameters in the **Tune Source** page (in the **Mass Spec** menu), or by dragging the red line to the place you think is best. Do a center scan after each change to see its effect, and to record the adjusted HT. Save the changes in the tuning page as you go under CURRENT, CO2, AUTORUN, and SILCO2. Under the **Cursor** menu you can also find a **zoom in** function, a **link peaks** function (click the two scans that you want to link), as well as the **Raw data** and a **drag** functions.
   1. HP. *Half plates. These are the first plates that the ions go through as they leave the source.* Zoom into the peaks of the major and minor beam scans and enter their maxima into the source parameter (or drag the red line there). If the peaks are not coincident, find a compromise between them.
   2. Z plate. *The z plates narrow the beam in the y plane, whereas the half plates had narrowed the beam in the x plane.* You only need to examine the major and minor scans, so un-choose the middle 2 by unclicking them in the “lines” menu. Zoom into the peaks of the major and minor beams, and find the spot that maximizes both the best.
   3. IR. *This is the voltage of the repeller, which repels the ions out of the source and down the flight tube.* Find the peak of the major and minor beams in this scan, then go between 1.4 to 1.75 V to the left of it. Currently the IR is approximately 1.7 V below the peak.
   4. EX.Find the peak of major and minor beams in the Extraction Voltage scan and go approximately 0.2 V to the left of it.
   5. EV. The Electron Voltage scan does not always have a clear peak. Make sure that you are not set in a strange slope or dip.
5. Once your peak is as wide as you can get it (shoot for at least 12 V), save the tuning under the four file names CURRENT, CO2, AUTORUN, and SILCO2.
6. Record in the notebook the bellows setting, the HT (accelerating voltage), the peak width, and the Ion Guage (also on the screen). Also, record the name of the water trap and the bellows position.
7. Go to **ID Peak** in the **Tests** menu, and make sure that you are still tuned to mass 45.0. If you are not, change it to 45.0.

*Starting the next run*

1. Pump out any of the tank line or flask valves you have newly loaded. If you just put on a new tank be sure to **check that the tanks line is holding by pumping down** on it (with the tank closed..obviously) then closing the valve, waiting about ten minutes and pumping down on it again, to make sure no air has built up due to a faulty connection in the line.
2. Make sure the **LN2 is on and the controller is on ‘automatic’**. Be sure the **triple** **trap is not iced up**.
3. Put on a **new water trap** by first making sure the valves are shut and then taking off the old, plugging the valves with glass plugs or at once inserting a fresh one out of the oven. Be sure the water trap is properly seated by pumping down on it and making sure that it holds. If **the ethanol is slushy, take off the dewar and replace it with a new one and fresh ethanol.** Then pump down on low vac, if it doesn’t pump down close valves and try reseating the water trap if it still doesn’t pump down get a new one and high vac on it for a while.
4. To start the run go to Analysis>Autorun file>Run#>run.
5. Go through the prompts as applies to the run, do not evacuate manifold and open flasks and tanks after you have begun.
6. Don’t forget to click Yes to ‘Open Flasks?’..Pops up five minutes or so after the initial prompts.

***Aboring the run***

1. Program:Terminate. You might need to do this several times.
2. You will then need to go to **Analysis:Autorun Batch Edit**. Load the run you have just stopped (click on SETUP) and click on Release All.
3. Go to **Analysis:Parameter File Edit.** Load the NOAA Sample Parameters and change the start position to
4. Go to the NOAACO2 folder on the desk top and delete the file SPOCK.txt.

***Baking Spock***

1. Pull out the magnet and remove the head amp, use #5 allen key.
2. Turn off LN2 and the cryocool, remove the water trap and replace it with a metal bridge or glass plugs, take off the TT doer to thaw ice that may have built up inside.
3. Open all valves on the manifold, manually and go to HV (A8 & AP do not need to be opened).
4. Put bakeout tents into place.
5. Go to the Mass Spec tab, click tuning and load **Bakeout2** Temp 120;90, Duration 12;12. Then start!