** Guide to running Amos**

Amos is a Isoprime dual inlet mass spectrometer that measures the stable isotopes od carbon and oxygen in atmospheric CO2. Amos primarily runs flasks but it equipped to run calibration tanks as well. Almost all of Amos’s work is connected to the NOAA CMDL Cooperative Air Sampling Network. Air samples are collected in 2.5 liter flasksand are measured for concentrations of CO2, CH4, N2O, H2, and various other trace gases at NOAA. Amos is designed to measure the isotopic ratios (45/44 and 46/44) of CO2 in these samples.

Amos measures three masses:

Mass Species

44 12C16O16O (the most abundant)

45 13C16O16O (carbon 13 isotope)

46 12C18O16O (oxygen 18 isotope)

To measure the stable isotope ratios of CO2, pure CO2 must be extracted from the atmospheric sample. This is accomplished cryogenically: a vacuum pulls the air sample through a mass flow controller set at 40 sccm, through a chilled ethanol bath held at -90°C to freeze out water, and into a “triple trap” held at –195.5° C with liquid nitrogen to freeze out carbon dioxide (Figure 1). The other gases, oxygen, nitrogen, etc. are vented away. The triple trap is then heated to –20° C and the sample is released into the sample bellows of the mass spectrometer. The reference bellows is filled with CO2 from a tank of Niwot Ridge air at the end of every run (therefore, it is isotopically quite similar to our sample gas). Aliquots of sample gas and reference gas are released iteratively into the source, where they are ionized, repelled down the flight tube, and separated by isotopic mass. They are collected in Farraday cups, the current is measured, and then a long sequence of data crunching begins!

**The practical stuff: how to run**

1) First things first – check previous nights run, crunch the data

* Look at message log, if run is completed you should see a message ‘*Ending current batch ‘00000.xls’ ‘Kernal stopping inlet script’ ‘…batch report ended’.*
* Open ‘Amos CO2 Crunch’ in C:/ drive. Select *‘Amos CO2 Crunch 1.0.xls’*  (say yes to enable macros). Click button and choose the run number that you want to crunch (i.e. 000108.xls). This is found in C://->IonVantage Projects->Amos (month)->BatchDB. Say ‘**NO’** when asks if want to save changes. Look at crunch and determine if good to go for the day!
  + Are the standard deviations of the refs ~ 0.015/0.03 for d13C and d18O? (columns AK and AL)
  + Is the internal precision (columns L and N) less than 0.01?
  + Is the trap on target?
  + Is there a lot of drift in the “virtual ref?”
  + Did the samples run at the target beam (usually 5 nA)?
* Copy performance data into the performance file. Save when done.
  + Look at the trap tank over time
  + Look at drift over time
  + There is a lot of useful information in here!

2) Remove flasks from previous run

* Close all stop-cock valves on the flasks. Remove carefully and determine the next stop on the flasks journey through carbon cycle world. This is designated by the sample pathway on sample sheet. For example, CH4c13, CH3D, CO2c14, VOC. In general, the stable isotopes of CO2 are measured first, then CH413 then VOC then CO2c14.
* Put samples in appropriate sample boxes and transfer to flask storage room where boxes are placed in the correct pile (labeled in flask storage room).
* Finished samples go into boxes on cart with bubble wrap between layers.

3) Prepping instrument

* Switch liquid nitrogen controller to **off** position. This allows time to melt any excess ice build up.
* Close HV in ‘*Manifold’* tab on inlet page in IonVantage. Open LV, V1, V2 and E1.
* Changing water trap:
  + Make sure valves around water trap are closed (E2 and E3).
  + Get new trap from 110°C oven (use glove) and let cool for a minute before you remove old trap. Gently loosen cajon fitting around trap and pull straight down to remove. Carefully set new trap in by pushing directly upward and tightening cajons.
  + Evacuate water trap by opening E2 and E3; watch and make sure pumps down to ~2.8. Scroll pump will make noise when pumping down. I like to HV on water trap for a couple minutes as well.
* Note the consistency of the EtOH. If slushy, gently remove the EtOH and replace with fresh EtOH from freezer. Note: close valves around water trap when replacing so that if you break the water trap the instrument will not be pumping on air/EthOH vapors. Make sure temperature reads at least -85°C on cryocool before starting run.

4) Loading flasks for new run

* Gather flasks boxes from flask storage room for CO2c13 analysis. Incoming sample boxes are placed on table labeled **‘CO2’.** There are 8 flasks per box (4 pairs). Amos’s manifold can run 40 flasks per run, therefore 5 flask boxes. Glance at sample sheets and make sure you are not getting all of the same site. Try not to run more than 3 pairs per side of the same site in each run.
* Place flasks on the manifold in pairs according to sample sheet. Remove red cap from flask and carefully press glass flask into cajon port. Gently twist to make sure flask is sealed in o-ring.
* Evacuating flasks
  + Close E2 and E3 and open LV. LV, V1, V2 and E1 should all be green.
  + Go to ‘*flask manifold’* tab on inlet and open MV, MA, MB, MC and MD. This evacuates the main tubing leading to manifold.
  + Once pumped down to ~2.8, start opening flask manifold valves, 5 at a time (i.e. A1, A3, A5, A7 and A9).
  + Continue until all valves are open and vacuum is at ~2.8. If not pumping down, check to make sure flask is seated properly in port and that it is closed.

5) Tuning

*We tune the machine daily to get the best possible beam – what we see on the screen as a peak. This requires letting reference gas into the mass spec and watching the peak as we make adjustments. (Note we want the three beams to be coincident at the top of peak, magnet position can affect peak shape)*

* While evacuating flasks, the instrument can be tuned. See manual for specifics on each parameter.
* Open RV and RM. Toggle c/o so that RC and SW are green and SC and RW are red. This allows reference gas that is in reference bellows to enter the source. Squish reference bellows (double click) so that m44 (major peak) reads ~5nA. This is the ideal beam for running. Allow couple minutes to stabilize.
* In tune window, click on three shortcut buttons for the tune page, scan, and center scan (buttons look like line graph and list). Click on ‘*Run Center Scan’.*
* You are looking for a nice flat, square peak.
* Fine-tune the peak by adjusting the following parameters: HP, Z, IR, EX, and EV. Go to *‘Aquire’* in tuning window and run each of these scans. This should not change too much from day to day.
  + 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. If you change the peak center, click save/accept peak center.
  + 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 of them the best.
  + 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 -6 V. [-5;-9]
  + 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. Usually around 70 V. (currently 1.5)
  + 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. Usually around 90 V.
* Once you have a nice center scan, close RV.
* Note tuning parameters down in notebook if anything major changes.
* To find a peak you can go back in the notes and view a previous peak, and adjust to those numbers.

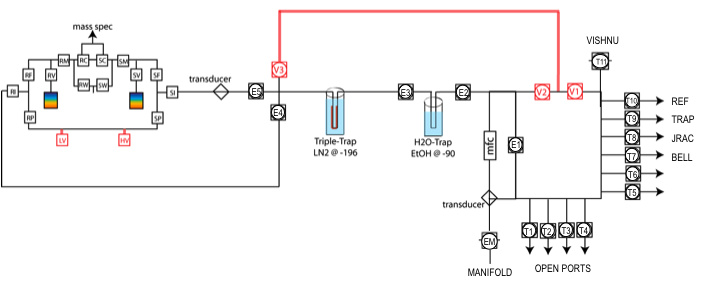
6) Making the session

*This takes the reference tank, flask, and PFP information and neatly puts it into a run sheet for us.*

* On Amos’s computer, go to <http://om.cmdl.noaa.gov/sb/> (should be pinned or start typing in search bar). Username is sil and password is “20020118”. Choose Isoprime Double Trap (Amos).
* The center scroll bar allows you to choose which type of sample you would like to enter. The options for that type appear on the left, and based on your choice, can be appended onto the window on the right. This will become your sample list for that run. To begin, highlight “working reference” on left. Choose the Amos reference (currently JELL). Its information, including sample port, will appear in the center. If you change your choice in the left window, click “ok” for it to update in the center panel. Once you are satisfied with your choice of working reference, click “append” four times so that the first four samples of the run are of that working reference.
* To enter flasks, scroll down to CCGG flask in the middle bar. Enter the flask ID in the left window to find its information. Press ok and it will automatically update the center panel. Append that information into your sample list and check that everything is correct. The ports will automatically begin with 11 (A1) and update sequentially. You will need to change flask port after TRAP is entered. See port-valve identifications on side of Amos computer.
* Hit save, copy the first tab data, open the crunch file, add a sheet at the bottom and paste the sample list.
* Highlight the list and click’ Text to Columns in Excel’, click delimited, next and comma and next.
* Copy and paste this text into IonVantage, make sure there is the proper amount of cells to fit all of the rows for the session.
* On IonVantage session builder, make sure the ‘bottle’ column represents the correct ports for each sample, reference and trap tank.
* Save the IonVantage as the new run number with 3 0’s before the run number.

7) Almost there…

* Check to make sure you have enough liquid nitrogen and that control is on automatic
* Make sure EtOH is cold
* Evacuate all flasks and PFPs and tank lines
* Wait some time … then check to make sure that flasks, water trap, tank lines are holding vacuum.
* Start the run. When prompted, open flasks



# 

# 

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.