OSB 446 is the room with NACHO

Elemental Analyzer NC 2500

* Controlled dynamic flash combustion
  + Tin sample is moved to autosampler drum, which is de-aerated to remove atmospheric nitrogen.
  + Then it is introduced into a vertical quartz tube heated at 1000 degrees C with a constant flow of Helium (carrier gas) stream.
  + A few seconds before the sample drops into the combustion tube, the Helium stream is enriched with a measured amount of high purity Oxygen to achieve a strong oxidizing environment, which guarantees almost complete combustion/oxidation.
* Oxidation
  + The combustion gas mixture is driven through an oxidation catalyst (Cr2O3) zone.
* Reduction
  + Then, through a subsequent one of copper which reduces nitrogen oxides, eventually formed during combustion or catalyst oxidation, to elemental nitrogen and retains the oxygen excess.
* Adsorption trap retains water
  + The gas mixture (N2, CO2, H2O) meets a trap containing anhydrone that absorbs water.
* Gas mixture coming out of the adsorption trap is swept into chromatographic column…

To run a sequence,

1. Email myself the sequence spreadsheet
2. Make sure the MS has all green lights showing (all on, all green) and EA is on (ready, normal lights)
3. Check completed run from previous day
   1. In Workspace, file sizes 341 kb for blanks, 350 kb for samples, around 200kb? File size for excel
   2. Blanks: lines should be around 10 to 15 units (not counting m/z 30 line), around 100 the computer will actually integrate it and it will mess with sample values, so when it starts to creep up, the ash trap will need to be changed
   3. Check the Std standards' and blank’s CO2 curves. If they are tailing more on the right as it approaches the cut-off, it can have an effect on the C reference peak, thus affecting sample readings. Therefore, be careful with higher end of C mass.
   4. Check the m/z 30 Nitrogen (NO) curve. This represents the gas that is not being reduced to N2 by the copper reaction tube, so if that peak gets too high it means that the copper reaction tube is failing and needs to be replaced.
4. Create new entry in:
   1. Paper log notebook
   2. Spreadsheet: 2021\_NACHO\_RUN\_LOG.xlsx
5. Create a new sequence by starting with an old one of mine, using "Save as", then making edits
   1. New name
   2. Copy and paste from seq spreadsheet Identifier 1 and mass in the Comment and Amount columns
   3. Make sure all rows have checkmark on the left (re: adjusting to the max peak...)
   4. If you add new rows, add checkmark and add method to match
   5. Hit "save"
6. Kind-of check: Conflow IV diagnostics
   1. On sticky note
   2. Will definitely have to check this if the program has been re-started or if my run is following someone doing a different method (not EA).
7. Load the samples
   1. Line up the carousel with "1" by carefully pushing down on it and turning (don't rotate whole top thing or tube will break underneath)
   2. 2 lids on top
   3. 0 == 50
   4. Load samples using the tray top underneath just in case
   5. Double check that every hole has one sample at the end
8. Press the start button
   1. Make sure either: no rows are selected or the "measure only selection" flag is unchecked.
   2. Make sure my name/ID is correct (will be if I'm going from an old run)
   3. Click okay
9. You'll see the sample combust below the metal circle thing when it glows orange. Then the metal plate turns 1 space.
10. Make sure it's running (hear the noise, see the orange light)
11. Watch for the dummy
    1. Should see sample peaks. If it's bad for some reason, stop the run so it doesn't ruin the samples
    2. ~ 200 sec the N peak of the sample will start
12. Watch for the blank
    1. Should be flat line

* A sample run as a N2 reference peak at each beginning (square shape), CO2 reference peak at the end
* Timing
  + One sample run = 10 min
  + 2 runs/day or backload
  + To backload, wait for 10 min window when the sample isn't being dropped or combusted (set timer if needed). Wait until sample drop, combustion, and wheel rotation.
* Computer programs
  + Isodat Acquisition (where runs are conducted)
    - Method = EA-3.met (always this for my samples)
  + Isodat Workspace (look at previous runs)
* Checking precision of standards
  + Run std dev on d15N and d13C for all standards (QTY and Std)
  + 0.3-0.4 on the edge, >0.4 not okay. 0.1-0.2 great
  + N is usually more consistent than C

Other notes:

* Sample curve should ideally be gaussian curve. When mass is really high, it becomes more leptokurtic (flat on left and wide tail on the right), which results in lower precision. CO2 peaks are normally leptokurtic
* When starting to analyze results, keep the raw data sheet separate (don't mess with it, make a separate copy to sort etc).
* Helium cylinder
  + Terry orders a new one when it's around 1000 psi so there's enough time for it to arrive.
  + It's 99.999% ultra pure He
  + Cylinder is around $230 and lasts ~1 month
* Oxygen cylinder won't run out quickly
* Backloading vs. Multiple runs:
  + If run drift is significant, long run would be bad
  + Linear drift is easy to explain, sinusoidal drift is not so easy to explain (temp, pressure possible reasons. MS sensitive to temp)
  + Backfilling saves machine time, standards
  + Beka has backfilled up to 70-75
* EA -> Conflow IV -> MS
  + Conflow controls flow rates, proper amount of gas
  + MS is continuous flow using He "carrier gas"
    - Vs. Duel inlet (more precise, no mixed gases)
  + MS
    - Faraday ion cups measuring voltage
      * Stronger magnet -> smaller ion tighter turn
      * Weaker magnet -> larger ion
    - V = IR voltage = resistor x current
    - F = m\*v^2/R (v^2 = charged particle in a magnetic field)
  + He2 has mass 4, makes tight turn. H hard to get good measures

Example screenshots of Isodat Acquisition and Isodat Workspace

* See Conflo IV Diagnostics selection (left side of Isodat Acquisition screen)

A picture containing text, screenshot, computer

Description automatically generated

A picture containing text, screenshot, computer

Description automatically generated

Problem chromatographs:

* NO m/z 30 peak is too high: I think once it’s higher than the sample N2 peaks (like below), then it’s time to change the copper reactor before running another sequence.

Graphical user interface, application

Description automatically generated

* NO m/z 30 peak is super high: overwhelming the sample N2 reading. This is an example from a run where the N15 data was unusable.

Graphical user interface, application

Description automatically generated

* The tail of the CO2 sample peak is getting too long and might eventually be messing with the CO2 reference peak so it’s time to change the ash trap.

Graphical user interface, application

Description automatically generated