Summer 2020 Research Project: Methods Comparison

* K2SO4 extractions
* Soil moisture
* Loss on ignition
* pH
* EC (Electrical Conductivity)
* CHNS analysis
* Ion Chromatography (IC) analysis

In this project, a methods comparison was performed in which results and data from Summer 2019 were compared to that obtained in Summer 2020. We are observing the difference in how our results change when procedures are performed in the laboratory vs. in the field. We looked at two different storage conditions for the samples. Half of the samples from the dry and wet plots were stored in dry ice and half of them were stored in a freezer. This helped us calculate the difference between observed data and expected data for samples in a natural setting vs. samples in a controlled environment. We also observed if there were changes in soil content between two different time steps. For the different time steps, we compared the changes in soil content for soil samples that were tested immediately after they were collected vs. after the same soil samples were placed in a -20 Celsius degree freezer for a month. Below is a description of how these methods were performed using different measurements of soil.

The barcode on the samples collected in the field were scanned into a tablet and this data was provided to the data science team. Other data collected in the field included, temperature, TDS, and volumetric water content of both locations. We also clicked pictures of the dry and wet plots from where the samples were collected.

K2SO4 extractions:

* For soil samples collected in the field, 1 Tsp, 2 Tsp, and 1 Tbsp of soil was added into three different 50 ml centrifuge tubes (VWR 89039-656) that contained 30 ml of 0.5 M K2SO4 which were weighed prior to adding the soil.
* For soil samples measured in the laboratory, 4.928 g (1 tsp), 9.857 g (2 tsp), and 14.786 g (1 tbsp) of soil was added to 50 ml centrifuge tubes that contained 30 ml of 0.5 M K2SO4.
* The samples were then placed on the shaker machine at 300 RPM for 30 minutes, and placed in a refrigerator overnight.
* The next day, the K2SO4 samples were removed from the refrigerator and filtered using filter paper (Whatman #3 Cat. No. 1003-110).
	+ Soil filtration racks were placed with funnels and filter paper. Scintillation vials (No. 121043) labeled with the barcodes for the samples were placed below the funnels to collect the K2SO4 extract.
	+ The vials were approximately ¾ full with the extract. These vials were weighed and then placed in the freezer.
* The remaining sample/slurry left behind in the 50 ml tubes was sieved. The stones from the samples were collected in weigh boats/weighing tins, allowed to dry overnight, and weighed the next day. The weight of the stones were subtracted from the weight of the soil that was measured in the tubes before the K2SO4 extraction.

Soil moisture:

* Approximately 15 g of soil was weighed into tins for the oven from four sub-samples (frozen whirlpak – plot#1, dry ice – plot#1, frozen whirlpak – plot#2, and dry ice – plot#2).
* The tins were placed in the oven at 70 degrees Celsius for three days, and weighed for the change in soil moisture.

Loss on ignition:

* Approximately 2 grams of soil was weighed into four crucibles, and were placed into the muffle furnace (Carbolite 301) at 100 degree Celsius for 8 hours on the first day.
* The samples were allowed to cool down and weighed on the second day. On the second day, these samples were put back into the muffle furnace at 550 degree Celsius for 4 hours.
* The samples were allowed to cool down and weighed on the third day. On the third day, these samples were put back into the muffle furnace at 1000 degree Celsius for 2 hours.
* The samples were allowed to cool down and weighed on the fourth day.

pH:

* The 15 g of soil that was dried and weighed in the step for “Soil moisture” was transferred into 50 ml centrifuge tubes (VWR 89039-656) to measure pH. It is helpful to use 15 g of soil sample because the same amount will be used for the EC (Electrical Conductivity) reading.
* 30 ml of ultrapure water was added to these tubes, and placed on the shaker machine for 30 minutes at 300 RPM.
* These tubes were then placed in the refrigerator overnight.
* The samples were taken out of the refrigerator the next morning and brought to room temperature before measuring their pH to avoid incorrect pH readings.

Using the pH probe in Lab#115:

* Turn on the Oakton instrument, and press “Mode” until the screen reads “pH”.
* Remove the small plastic covering on the tip of the pH probe, and clean the tip with D.I/ultrapure water.
* Wipe the probe tip with a clean kimwipe, and place the tip into the 7.0 pH buffer (yellow colored buffer kept in the hood).

Note: if the pH reading if off by more than 0.1 units, it needs to be recalibrated.

* If the reading appears to look correct, clean the probe tip with D.I./ultrapure water and wipe it with a clean kimwipe.
* Place the clean probe tip into the soil slurry and stir it around keeping in mind that pushing the probe tip too hard against the soil sample will damage the tip.
* Wait till the instrument flashes “ready” on the screen. This indicates that you have obtained the pH reading of your sample.
* After obtaining each reading for the samples, carefully clean the probe with D.I/ultrapure water and wipe it with a kimwipe.
* The pH probe needs to be stored in KOH solution.

Note: the pH probe needs to be calibrated after every 50 samples or after every 3 months, whichever comes first.

EC (Electrical Conductivity):

* The same sample tubes that were prepared to measure pH (15 g of soil in 30 ml of D.I./ultrapure water) were used to measure EC.

Note: 15 g of soil sample in 30 ml of ultrapure water is used for this step because the EC probe has two silver rings on it that need to be completely submerged into the soil slurry.

Using the EC (Electrical Conductivity) probe in Lab#115:

* + Clean the plastic yellow covering and the EC probe with D.I./ultrapure water, and wipe them with a clean kimwipe.
	+ Turn on the Oakton instrument, and press “Mode” until the screen reads “uS”.
	+ Place the clean probe along with its plastic covering into the soil slurry.

Note: do not remove the plastic yellow covering from the probe while measuring the EC. It should be taken off only to be cleaned between each measurement.

* + The screen will flash “ready” when the instrument has obtained a reading.
	+ After obtaining each reading for the samples, carefully clean the probe and its plastic covering with D.I/ultrapure water and wipe them with a kimwipe.

CHNS analysis:

* Approximately 1 g of the soil samples dried in the oven for three days from the four sub-sample sites (frozen whirlpak – plot#1, dry ice – plot#1, frozen whirlpak – plot#2, and dry ice – plot#2) were put into safelock tubes along with 3 mm tungsten beads and ground up into fine powder using the mixer mill in Lab#115.
* About 7-10 mg of these samples were weighed into aluminum tins for the CHNS analysis. Triplicates were measured from each of the four sites.

Ion Chromatography (IC) analysis:

* The 36 samples measured in the lab, 6 samples measured in the field, and the blanks that were processed for the K2SO4 extraction were used for this step. There were an additional 42 samples that were measured for the second time-step and processed for the K2SO4 extraction. All of these samples were run on the I.C. in Lab#115.
* The vials collected from the K2SO4 extraction were taken out of the freezer and allowed to thaw for approximately 1 hour in a lukewarm water bath.
* 100 uL of sample was mixed with 900 uL of D.I./ultrapure water into the tubes that are used for the IC machine.
* The analytes measured for the anion content were, fluoride, chloride, nitrite, nitrate, and phosphate. The analytes measured for the cation content were, lithium, ammonium, sodium, magnesium, and calcium.

References

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