OSU CSS SOIL HEALTH LABORATORY QUALITY ASSURANCE PLAN

Sample intake

As soon as samples are submitted to SHL, a group number is assigned (e.g. 219001). A group is defined as a set of samples of the same type (soil, plant, solution, etc.) from one customer, submitted at the same time, which will all have the same set of analyses performed. If there are any questions about the submission, the lab manager will contact the customer for clarification. A new order is made for the group in the SHL Access database and an Excel sheet is created from the appropriate template. Each sample is given a SHL ID#, which is the group number plus a sequential identifier (e.g. 219001-1, 219001-2, etc.). The ID# will be written on the original sample container in permanent marker. A key is made in the Excel results sheet to link the customer's ID to the SHL ID. An email is sent to the customer with their group number informing them that their samples have been received. Samples are either immediately prepared for analysis or they are stored in the appropriate location. The location of the samples is recorded in the Work Orders spreadsheet.

Sample handling procedures:

- Every sample is stored in a bag that has the complete SHL ID number on it. Shorthand, such as "x102-1" is not allowed. Putting the group number on a box and only the individual number on the bag is not allowed. Printing labels with the group number and sample number and sticking those labels on the sample bags is allowed.
- Samples should be kept together with their group.
- Samples where all tasks have been completed for the group should be stored in the archive until the appropriate disposal date.
- Samples should be stored for at least one year unless the customer returns to pick up the samples or requests that the samples be disposed of.
- If a sample ID is rubbing off of a bag, it should be rewritten immediately.
- If wet samples are received, but cannot have the intake/drying process completed before the end of the day, they should be stored in the refrigerator. An email should be sent to the lab manager and a note should be left of the "shift change" board so those samples can be processed as soon as possible.

Sample weighing procedures:

- Samples should be weighed into a vessel of appropriate size. Each vessel should be visually inspected for contamination before weighing a sample into it.
- Weights should be recorded to the last decimal place on the balance being used.
- A balance should be used where the smallest difference in weight is less than 1% of the value being weighed
    - E.g. If a balance only can go to one decimal place, the smallest difference that can be measured is 0.1 g. This balance should not be used for samples weighing less than 10 g.
- Whether a sample should be weighed directly into the container or onto a sheet of weighing paper will depend on the properties of the substance, the vessel being used, the mass being weighed out, and the capabilities of the balance. Ask for help from the lab manager if you are not sure what to do.

Washing dishes
All labware that comes in contact with soil or plant material should be washed before its next use. All mass soil should be rinsed into the "soil bucket" in the sink. Once that is done, it should be quickly rinsed with tap water. It can then be washed in the "clean bucket" with tap water and dilute Micro 90 detergent. Everything should be scrubbed with a brush or sponge, and then triple rinsed with DI water. All open-type vessels should be placed upside down to dry overnight. Labware should not be put away wet and should not be dried with paper towels. If a piece of glass or plasticware needs to be dried quickly, it can be rinsed with ethanol to displace the water and dried with lab air from a spigot. Glassware may need acid washing. In this case, glassware should be submerged in 10% HNO_3 for 24 hours.

QA/QC samples:
For all analyses, an in-house laboratory control standard is run. For soils, this is called the SHL Std soil. For plants, this is a composite tree fruit sample (CTFS). The results of these LCS samples are recorded immediately in the QA/QC tracking sheet (saved in R: drive, accessible from all lab computers). If the results for the LCS sample are outside of the action limit for an analyte, the results from that run are not accepted. If the results for the LCS sample are outside of the warning limits but inside the action limits, results are accepted if it is not part of an unacceptable warning limit pattern (see Lab Quality ConTroll).

For all analyses, a method blank is run. If the results from the method blank are different from the calibration blank, results for the unknown samples are adjusted accordingly.

For all automated equipment, samples should be run that check the calibration of the instrument during the run. In general, one of these samples should be run every 10-20 samples. These are referred to as calibration control samples (CCV).

Depending on the needs of the specific analysis, other QA/QC samples may be run. For example, standard addition, spike samples, second source, and replicates may be needed.

Proficiency testing
SHL participates in the North American Proficiency Testing Program (NAPT), a round robin program where five unknown soil samples and three unknown plant samples are analyzed every quarter. Results are sent to NAPT, which compiles the results from all participating laboratories. SHL receives a report detailing the mean values for each analysis, the median absolute deviations, and SHL's reported results. Any results that are further from the mean than acceptable are flagged. When that happens, SHL will look into that method to determine why our numbers deviate from other labs', and take appropriate steps to correct the issue.

Instrument maintenance
Instrument maintenance is performed on a schedule for every instrument. Documentation of this maintenance is recorded in the instrument's maintenance logbook. Use of the instrument is also documented in the logbook.

Brief routine maintenance guidelines and QA/QC procedures for instruments:

Agilent ICP-OES:

- The instrument is rinsed with 5% nitric acid for ten minutes at the end of every day that the machine is used
- Peristaltic pump tubing is changed every month or when sample RSDs go above 2%
- The torch, nebulizer, and spray chamber are cleaned according to the manufacturer's instructions every two months
- WaveCal performance check is performed at least every three months, or whenever the peaks have noticeably shifted.
- If any mold or growth is observed in the rinse lines, they need to be changed.

Lachat FIA:

- Pump tubing should be switched out when it no longer feels round to the touch.
- If mold/growth is observed in any sample or waste lines, or if they have hardened, they should be changed. Extra tubing is stored above the instrument.
- Reagent solutions should not be kept for more than one month.

Elementar Vario Macro Cube

- When any tube or part is replaced, everything associated with those connections should be checked. All o-rings should be inspected. If any flaws are observed, it should be replaced.
- A combustion tube that has been removed from the instrument will be too weak to used again, and should be disposed of after removing the reusable components (support tube, sheath tube, possibly ash crucible)
- A reduction tube that was easily emptied of its oxidized copper filling can be used again as a reduction tube. If the drilling tool was needed to remove the copper, it is likely scratched and cannot be used again effectively.
- The ball valve should be taken apart and cleaned according to the manufacturer's instructions every six weeks, regardless of machine use.
- The machine should only be used if, in standby mode, the pressure is >1100, He flow is 590-610, and TCD flow is 590-610.
- A full calibration using sulfanilamide and/or phenylalanine should be performed once every six months.
- Standard check samples should be used to check the calibration before samples are run every time that the machine starts from a sleeping, off, or error state. If the check samples are not in the acceptable range, check all tubes and gases. Do not run a new calibration if the check samples are wrong: generally, the calibration is fine, but there is a problem somewhere else.
- Do not adjust the O2 or He regulators except under the express supervision of the lab manager.
- Do not ever use the regulators as coat hooks – this can alter the flow to the machine and change the results.
- Always wear clean, fresh gloves when wrapping or touching samples for the Elementar – skin oils can get on the foil and analyzed along with the sample.