Preparation of Soil QC Materials for Analysis Laboratories

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A key to a good laboratory quality control / quality assurance program is creating and maintaining high quality reference soil samples. The use of a well prepared QC soil helps a laboratory track and improve analytical performance. Soils used in the NAPT Program are pulverized and blended using a Patterson-Kelly V Shell blender. Development of an in-house laboratory QC soil has two requirements: (1) chemical and physical parameters which reflect typical ranges encountered during daily analytical operation; (2) a well homogenized material in which the mean and variance can be well characterized. Quality control soils should bracket the laboratory's working analytical range. Typically, a low and medium range soils are more important than a high range soil because of their agronomic significance. High range QC soils may have greater importance for environmental issues, specifically soils receiving animal wastes. When collecting a QC soil it's important to locate a site where material can easily be collected with sufficient quantity to be utilized over 1-2 years.

Collection, preparation and storage of a QC soil requires specific steps to ensure homogeneity and high quality over a extended period of time. When collecting it's important to collect from a defined area of 200 to 600 square feet in a location where soil type, slope, and crop residue are as uniform as possible. The depth of collection should be limited to 4-8 inches as not to create depressional area in the field. Coarse fragments and crop residue (root crowns, stalks, leaves and etc.) should be discarded. It is suggested that soils should be air dried on large tarp (20 ft x 40 ft) in thin layers 0.25 - 0.50 inches thick. While drying soil homogeneity can be enhanced by pulling on the tarp's corners to form a pile in the center of the tarp and then using a fine rake, redistributing the soil over the tarp surface. This process should be repeated at least three times. Additional raking using a fine tooth (0.30 inch opening) rake can remove medium gravel other crop residue. Occasionally, It may be necessary to crush large soil aggregates (> 0.5 inches diameter). Crushing should occur before the soil is completely dry. Drying the soil below 2-3% moisture increases soil aggregate resilience and increases fine dust during processing.

Standard soil analysis requires soils to be pulverized or crushed to pass a 2.0 mm (10 mesh) screen. Although this is sufficient for routine soil analysis, it is not fine-grained enough for QC reference soils. Coarse textured QC soils (sandy loams, loamy sands) should be pulverized and screened to pass 1.0 mm with medium and fine textured soils (loams, silt loams, clay loams) screened to pass 0.8 mm or finer. Removal of the coarse soil fractions increases soil uniformity and therefore analytical homogeneity. Finer QC soil material (screened to pass 0.50 mm opening) maybe necessary for specific analytical methods utilizing less than one gram of soil material, e. g. total nitrogen, total organic carbon.

The final step after sampling, drying, pulverizing and screening the QC soil is blending. Small QC soil quantities can be prepared using a rotating barrel such as a lapidary tumbler. Larger quantities (> 5 kg) require blending using a cement mixer or a large rotating drum. Rotating barrels, (such as a cement mixer), are prone to stratification of particles. Therefore it is essential to screen the QC soil to prior to final blending when using these types of mixers.

Storage can influence the stability of a QC soil. Many laboratories divide QC soils into 1-5 kilogram quantities and store them in a zip-lock type bag. This keeps particle separation to a minimum. Bags can then be placed into a large storage container such as plastic barrels with lids with lids. These barrels should be stored where humidity and temperature fluctuations are kept at a minimum, usually somewhere in the laboratory. When a "new" bag is taken from the barrel it should be remixed prior to its use in the laboratory.

Development of QC standards for the soil is performed by replicated analysis usually side by side with an already well established QC soil. The prospective QC soil is analyzed at least 30 times over the coarse of several days and based on repeated analysis; the mean and standard deviation of individual analytes are then established. Suggested ranges of RSD (relative standard deviation) ranges for QC soils representing good homogeneity for a limited group of soil analyses is listed in Table 1. QC warning limits are typically established at plus or minus 2.0 times the standard deviation of the mean value ($\bar{X} \pm 2 \times s$).

Generally acceptable homogeneity for, nitrate-nitrogen, extractable soil P (Bray, Mehlich and Olsen methods), extractable K and organic matter is less than 10% of the mean. Acceptable homogeneity for extractable sulfate sulfur, calcium, and micronutrients is 10-15% of the mean. The RSD value for any given QC soil will vary somewhat depending on the analytical range. A QC soil that represents the range near the detection limit will have a much higher RSD than one outside the very low range. For example a 2.0 mg kg^{-1} bicarbonate extractable phosphorus value will vary plus or minus 1.0 mg kg^{-1}, a high RSD but still very useful. Note that RSD values less than 10% are very useful in tracking analytical quality, while higher RSDs have limited usefulness. Laboratories should strive for RSD values less than 10% across all parameters.

Table 1. Suggested acceptable levels of homogeneity for laboratory QC soils.

Soil Analysis	**RSD (%)** [1]
pH (1:1, 1:2)	0.8 - 1.2
Buffer pH (all methods)	0.8 - 1.2
NO_3-N (Cadmium Reduction)	5 - 10
Phosphorus (Bray, Olsen, Mehlich-1 Mehlich-3)	5 - 10
Potassium (NH_4-OAc, Mehlich-3)	5 -10
Calcium & Magnesium (NH_4-Oac, Mehlich-3)	10 - 15
SO_4-S (Calcium-Phosphate, Mehlich-3)	10 - 15
Zn, Mn, Fe & Cu (DTPA and Mehlich-3)	10 - 15
Organic Matter (WB and LOI)	5 - 10
CEC (replacement)	6 - 12
Sand Silt and Clay (hydrometer)	5 - 10

[1] Values based on RSD of 30 replicates.