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USDA LTAR Common Experiment measurement: Best practices for collection, handling, and analyses of water quality samples

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Abstract

Following best practices during sample collection and analysis will produce data of known and defensible quality. This protocol details the best practices for the collection of water samples from croplands and for the following water quality protocols:

- USDA LTAR Common Experiment measurement: Dissolved nitrate (NO_3^-) concentration
- USDA LTAR Common Experiment measurement: Dissolved ammonia (NH_3) concentration
- USDA LTAR Common Experiment measurement: Total nitrogen (TN) and total dissolved nitrogen (TDN) concentration
- USDA LTAR Common Experiment measurement: Total phosphorus (TP) and total dissolved phosphorus (TDP) concentration
- USDA LTAR Common Experiment measurement: Total suspended solids (TSS)

Sample collection and handling for the analysis of primary water quality metrics

1 Measure the primary water quality metrics in water leaving croplands from surface runoff, subsurface flow or drain tile, and leaching or percolating water below the rooting zone.

Note

- The general sample collection methods outlined below precede more specific instructions
within each primary metric protocol.
- Use plastic or glass bottles to collect all samples.

2 Collect the **surface runoff and drain tile** samples manually as single discrete grab samples or using automatic samplers.

3 Collect grab samples at a specific site for a limited duration (seconds to minutes) and to represent a “snapshot” of the measured constituent.

3.1 During a grab sample collection, condition the sampling bottle by filling and discarding the sample three times before retaining the fourth sample.

4 Collect automated samples as a single discrete sample or composite sample over time.

5 Collect runoff and drain tile samples frequently using automated composite sampling through discharge-weighted methods such as equal discharge increment procedures (APHA, 2005).

6 Sample the **leaching or percolating water** using soil water solution sampling lysimeters consisting of porous devices that can extract soil water under a vacuum or access wells to permit sampling beneath a water table. Care must be taken in the selection of lysimeter type with respect to the analytes that are to be measured, as well as in the installation of lysimeters (see reviews by Weihermüller et al. 2007 and Singh et al. 2018).

Note

The volume of sample collected should suffice to ensure a representative sample and allow for duplicate analyses if required.

7 Return samples to the laboratory on ice and filter them on collection day if possible.

7.1 Store the subsamples of filtered and unfiltered samples in separate bottles because preservation techniques and storage times differ between analyses.

Quality assurance and quality control: General procedures

8 For the analytical methods described above, follow standard quality assurance (QA) and quality control (QC) procedures (APHA, 2005).

Note

- Strictly following QA/QC procedures during sample collection and analysis will produce data of known and defensible quality.
- The recommended QA/QC procedures include a written and detailed standard operating procedure (SOP) for use in the laboratory as well as the following items listed below.
- Laboratories are responsible for maintaining accurate performance records defining the quality of the generated data.

9 Perform instrument maintenance according to the manufacturer's instructions and maintain a log of the instrument performance.

10 Determine the method detection limit (MDL; the analyte concentration that produces a signal with a 99% probability of being different from the blank) within site-specific water matrices.

11 Field Blanks: Place deionized water in a sampling container in the laboratory, transport it to the field, and treat it as a sample, including exposure to sampling conditions, sample handling (preservation, filtration, and storage), and sample analysis.

12 Laboratory Blanks: To assess contamination from the laboratory environment, treat deionized water as a sample throughout the analytical process. Values should not exceed the MDL.

13 Prepare a standard calibration curve with four to six analyte concentrations. Check for instrument accuracy and precision.

13.1 Conduct a new calibration at the beginning of each instrument run. Reportable results fall within the range of the standards used in the calibration.

14 Run a certified standard (also termed: instrument performance check standard, calibration check standard, external standard, or standard reference material) with each analytical sequence to verify calibration and method accuracy.

Note

- Certified values should fall between 90-110% recovery.
- The results should not exceed three standard deviations from the accepted average value.
- If the certified standard is outside of calibration limits, recalibrate the instrument.
- Reanalyze all samples analyzed after the last acceptable standard.

15 Laboratory fortified matrix (LFM): To determine whether the sample matrix contributes bias to the analytical results, add a known amount of analyte to a sample. The LFM is analyzed just like a sample.

15.1 Conduct one LFM duplicate per sample set or on 5% of the samples, whichever is more frequent. Acceptance criterion is 90-110% recovery.

16 Analyze the duplicates to assess precision.

16.1 If duplicates differ by more than 10%, reanalyze the sample.

Note

- To ensure data comparability across the LTAR network, laboratories shall participate in an annual common proficiency testing round conducted by an accredited provider.
- Common proficiency testing, or inter-laboratory comparison, involves the analysis of a sample of unknown analyte concentration by a group of laboratories. The results of each participating laboratory are sent to the provider and compared to the known or actual value.

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