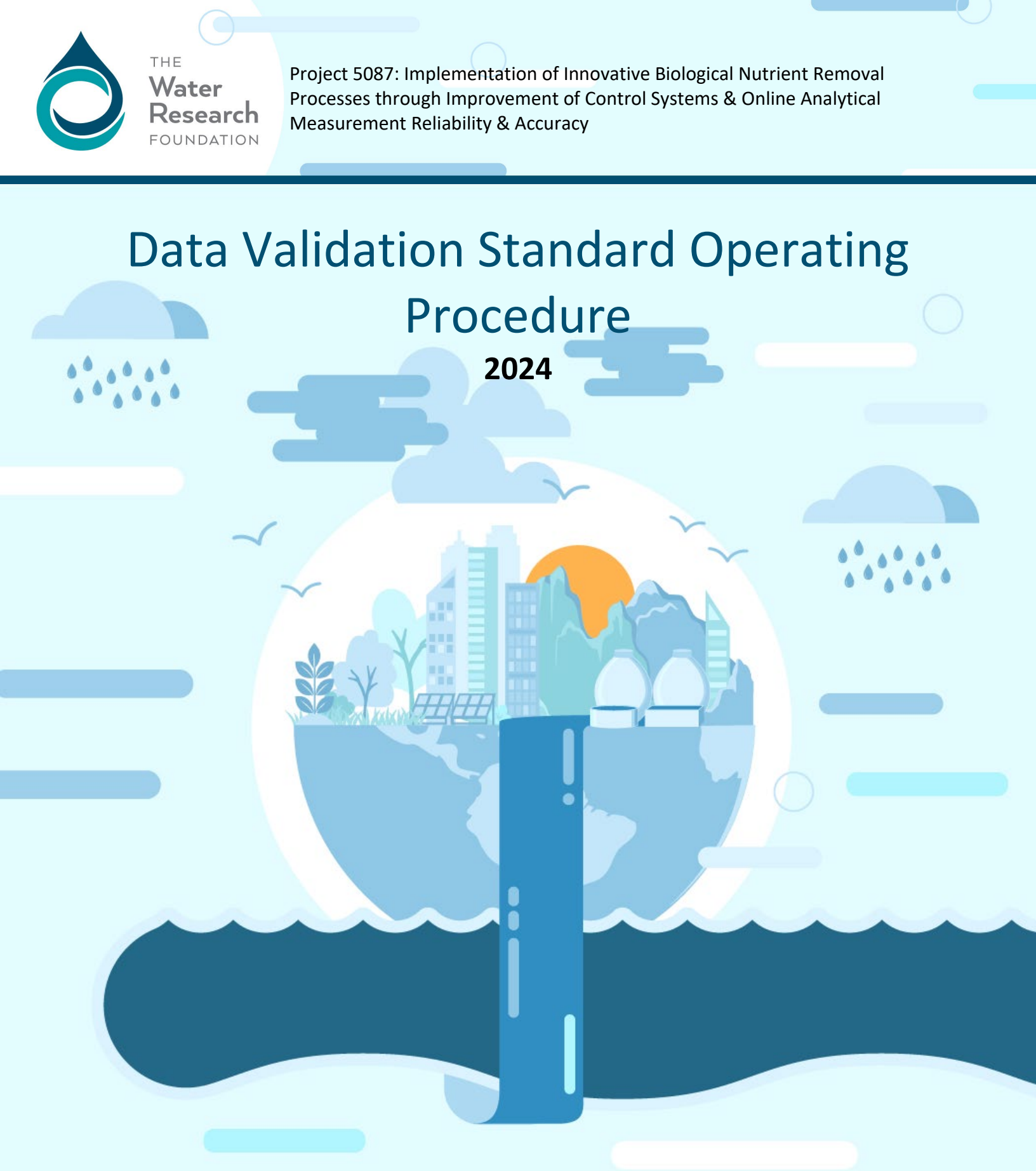
THE Water Research FOUNDATION

Project 5087: Implementation of Innovative Biological Nutrient Removal Processes through Improvement of Control Systems & Online Analytical Measurement Reliability & Accuracy

Data Validation Standard Operating Procedure

2024



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Overview

Data from sensors and analyzers is most valuable when the values are accurate. Field studies at the University of Massachusetts Amherst Water and Energy Testing Center and the Amherst, MA WRRF were conducted to help build a Standard Operating Procedure (SOP) for collection and analysis of samples used to verify sensor data with laboratory data and perform matrix adjustments of the sensors.

In this SOP the term “calibration” refers to a multi-point calibration procedure that is most often performed by a sensor manufacturer in their factory with samples of known concentrations, while “matrix adjustment” refers to a minor adjustment of the sensor reading based on differences between the sensor reading and a laboratory analysis of a sample from the reactor adjacent to the sensor. Factory calibration of sensors is required infrequently and should be performed in accordance with manufacturer recommendations. Operators should observe data trends over time to determine if a matrix adjustment or some other form of sensor or controller correction factor is required. A single erroneous data point from one time point may not necessitate an adjustment, and over-adjustment of sensor readings can result in increasingly inaccurate data.

Sample collection, analysis, and comparison of sensor values with laboratory analyses should be conducted in accordance with sensor manufacturer recommendations. These laboratory analyses are used to confirm reliability of sensor values, and to determine if a matrix adjustment is necessary. Frequency of sensor verification sampling will depend on a variety of factors including the type of instrument, if the instrument is being used for monitoring or control, and operator experience. In the absence of other information, a frequency of once weekly is suggested.

Throughout this SOP, it is assumed that facility staff are familiar with typical procedures for wastewater sample collection (e.g., rinsing a dipper/scoop multiple times prior to collecting a sample), and laboratory analyses. A field log is recommended to record pertinent information such as day/time of sample collection, sensor location and parameter, personnel involved in collecting and analyzing samples, sensor reading and laboratory measurement values, and whether a matrix adjustment was performed.

Step 1: Sample Collection for Data Validation

Step 1A: Collect sample

Collect the sample adjacent to the location of the sensor or analyzer.

1A.i. Clean instrument prior to sampling

Prior to collecting a sample for laboratory data validation, the sensor should be cleaned in accordance with the manufacturer’s directions and then placed back into service for ~15 minutes.

1A.ii. Record sensor value

At the time of sample collection, note the sensor value and/or indicate on the sensor's local control panel that you are collecting a validation sample.

Step 1B: Filter sample immediately

Mixed liquor samples collected for nutrient analyses (ammonia, nitrate, nitrite, and orthophosphate) should be filtered immediately after collection. Delaying filtration of samples, even to transport them to an on-site laboratory can significantly impact results and lead to improper matrix adjustments (see Figure 1).

1B.i. Select filter type

Filtration with a 0.45 µm pore size syringe filter is recommended by some manufacturers. However, a standard coffee filter is also an effective and inexpensive option. One drawback of coffee filters is their lack of structural integrity when pouring the sample. To combat this, some facilities use large diameter filter paper that has been folded into a funnel shape and placed inside a plastic funnel (see Figure 2). It is recommended to maintain a consistent filter type.

1B.ii. Refrigerate samples

If possible, samples should be refrigerated at 4 °C or placed on ice after filtration. However, as seen in Figure 1, temperature has minimal impact on the measured sample concentration for filtered samples that are stored in the range of 12-18 °C. Temperature impacts may be exacerbated at higher temperatures, so it is still suggested to store samples in a cooler, on ice, while transporting them from the collection point to the lab.

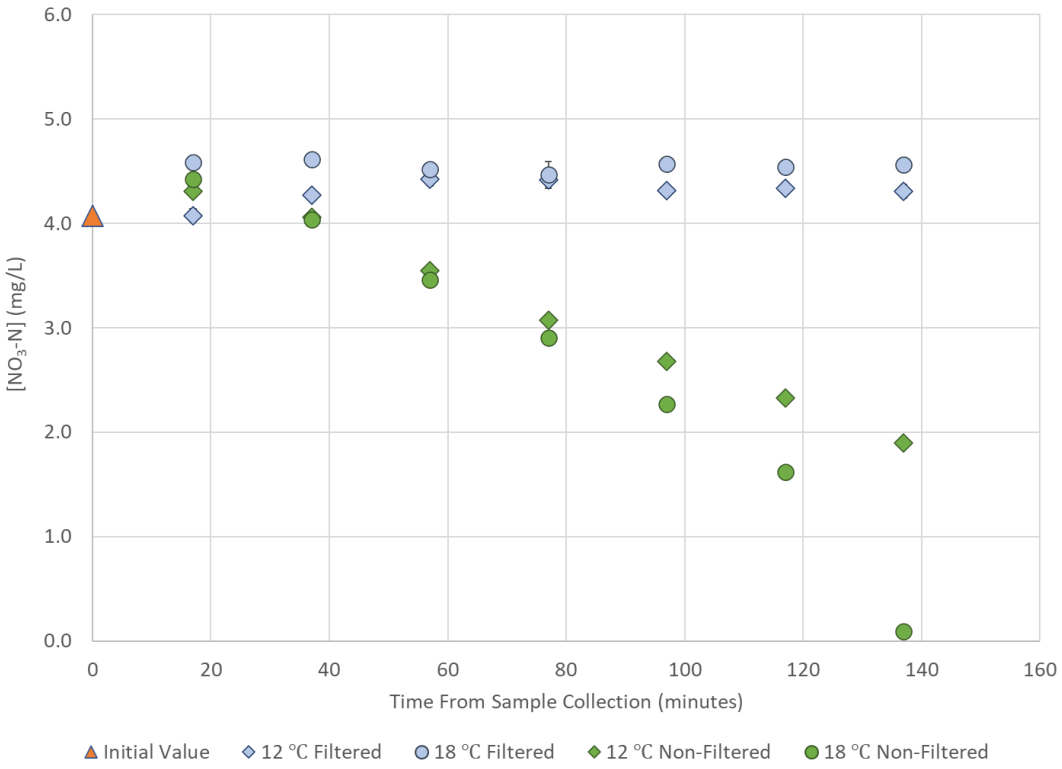

Figure 1. Nitrate concentration as a function of time after sample collection with and without filtration and refrigeration.

Figure 2. Image of a large diameter filter paper that has been folded and placed inside a plastic funnel for sample collection.

After samples are collected, they should be analyzed as soon as possible in accordance with Step 2.

Step 2: Sample Analysis for Data Validation

Samples should be analyzed as soon as practical after collection. The timing of sample collection should be coordinated with lab staff and equipment to ensure that adequate resources are available for testing.

2A: Prepare lab testing supplies

Pre-packaged testing kits are frequently used for these kinds of analyses.

2A.i. Consider ease of method

Based on testing conducted at the University of Massachusetts Amherst Water and Energy Testing Center, and input from survey responses, the accuracy and precision of either Hach Test 'N Tube™ or TNTplus® kits is acceptable for sensor validation testing. However, if the person performing the analysis is less experienced, or performs laboratory analyses infrequently, then a more user-friendly test kit like Hach TNTplus® is recommended.

2A.ii. Confirm concentration range is appropriate

Note that there are kits with several different concentration ranges available for each analyte of interest. Ensure that the test kit used is appropriate for the concentration of the sample.

2B: Perform test following manufacturer's instructions

Pre-packaged testing kits are frequently used for these kinds of analyses.

2B.i. Single tests are typically adequate

Typically, a single test kit will provide appropriate precision for this application, and duplicate testing is not required. If there is a large discrepancy between the value from the sensor/analyzer and the value measured in the laboratory, a duplicate laboratory analysis may be warranted.

After laboratory testing has been completed, the value should be compared to the value from the sensor/analyzer noted in Step 1A.ii. If the two values differ, then a matrix adjustment may be required as indicated in Step 3.

Step 3: Matrix Adjustments

3A: Assess need for matrix adjustment

Operators should make matrix adjustments only when the sensor and laboratory values differ by more than 10%, or in accordance with the manufacturer's instructions. Note that this 10% value is only a guideline, and it may change based on sensor location, measured concentration, operator experience, and manufacturer's instructions. Operators should observe data trends over time to determine if a matrix adjustment or some other form of sensor or controller correction factor is required. A single erroneous data point from one time point may not necessitate an adjustment, and over-adjustment of sensor readings can result in increasingly

inaccurate data. Collection and analysis of a second sample may be warranted to confirm that a matrix adjustment is required.

3B: Perform matrix adjustments only when necessary

Matrix adjustments should be performed only when they are necessary to ensure accurate data.

3B.i. Over adjusting can lead to drift

Operators are also cautioned against making matrix adjustments too frequently as this can cause the signal to drift over time.

3B.ii. Exercise caution when low concentrations are measured

Additional caution should be exercised if a sensor reading is less than 1 or 2 mg/L, as many sensors are less accurate at low concentrations.

3C: Contact manufacturer if frequent adjustments are required

Additionally, if it appears that frequent (e.g., weekly or bi-weekly) matrix adjustments are required, then there may be an issue with the sensor calibration.

SOP Flowcharts

The attached flow charts illustrate the steps for sample validation:

1. Sample Collection for Data Validation
2. Sample Analysis for Data Validation
3. Matrix Adjustments