

Ammonia/Fluoride Distillation Policy (NC WW/GW LCB 10/9/2024)

It is the responsibility of the permittee to ensure that monitoring is conducted according to test procedures approved under 40 CFR Part 136 even if the permittee does not operate the laboratory that performs the analytical testing on the waste stream. In terms of Ammonia Nitrogen and Fluoride monitoring, this means that the permittee must ensure that their effluent is appropriately characterized as to whether distillation is required regardless of what laboratory performs the analysis. Methods that specifically state that distillation is not required may only require distillation to resolve controversies. Additionally, manual distillation may not be required if comparability data on representative samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. A comparison study may be performed in-house or contracted to another certified laboratory. Permittees that do not perform the analyses in-house and contract the analyses or the distillation study to another NC WW/GW LCB certified laboratory must obtain a copy of the initial comparison data and all subsequent comparison data, keep it on file at their facility and make these records available to the Department upon request. Commercial laboratories must follow method requirements for distillation as applicable unless a study is on file for each client's sample(s) showing that distillation is not necessary.

Samples must be spiked according to the NC WW/GW LCB Matrix Spike Policy, in duplicate, to allow for a meaningful statistical comparison. It is recommended that samples are spiked to yield a value within the verified calibration range so that sample dilution is not needed. Comparisons between the matrix spike and matrix spike duplicate, as well as between distilled and undistilled spiked samples must meet a 20% RPD acceptance criterion. Per 15A NCAC 02H .0805 (e) (2), it would be permissible to have another certified laboratory perform the distillation study. When a permitted facility contracts a commercial laboratory to perform the study only, both the distilled and undistilled spiked samples must be analyzed using the same method technology used by the permitted facility to report compliance data. When a commercial laboratory performs a study to characterize samples routinely submitted by a client for analysis, distilled samples must be analyzed using each parameter method technology that will be used.

The following frequencies are required:

Initially, compare a minimum of 9 samples from each matrix (e.g., effluent, influent, stream, etc.), spiked in duplicate, both with and without the distillation step (a total of 36 samples), to evaluate the need for distillation. These 9 samples may be spread out over a 12-month period. However, at least three samples from each matrix would be required to obtain initial certification.

If the characteristics of any permitted matrix change (e.g., contributing industries are added or lost, major change in plant processes, etc.), or if the laboratory changes to another analytical method that requires the comparison, a minimum of two additional samples must be spiked in duplicate and analyzed, both with and without the distillation step, to demonstrate that that distillation is still not required.