

Ammonia/Fluoride Distillation (NC WW/GW LC Policy 04/09/2019)

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. 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 LC 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.

Samples must be spiked according to the NC WW/GW LC 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. Both the distilled and undistilled spiked samples must be analyzed using the same method technology used to report compliance data for the permitted facility. It is recommended that both the distilled and undistilled portions of the spiked sample be analyzed by the same laboratory. Per 15A NCAC 2H .0805 (e) (2), it would be permissible to have another certified laboratory distill the samples and send the distillates back to the permittee for analysis within the prescribed holding time. It is the responsibility of the permittee to ensure that when only the distillation portion is contracted, that samples are distilled into the proper receiving solution required by the analytical method to be used. That means when the titration method will be employed, samples must be distilled into a boric acid solution. When the phenate method will be employed, samples must be distilled into a sulfuric acid solution.

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.