

Division of Consolidated Laboratory Services (DCLS)
Virginia Environmental Laboratory Accreditation Program (VELAP)

Technical Assistance Document

**Procedures for Application for Variance Regarding Manual Distillation:
Determinative Methods Approved in 40 CFR 136.3 Table 1B**

This is a Technical Assistance Document prepared by the Laboratory Certification Group of the Virginia Division of Consolidated Laboratory Services. This document was prepared to provide laboratories with technical assistance in achieving compliance with the required regulation.

Regulatory Requirement:

40 CFR 136.3 Table 1B, for Ammonia (as N), mg/L, requires distillation or gas diffusion at pH>11 followed by specified approved determinative technologies using certain approved methods listed on the table. The table contains a Footnote 6 regarding distillation, providing the supplemental information. [Note that Footnote 6 may also apply to other analytes as designated in Table 1B; e.g., see Fluoride.]

⁶ “Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies. In general, the analytical method should be consulted regarding the need for distillation. If the method is not clear, the laboratory may compare a minimum of 9 different sample matrices to evaluate the need for distillation. For each matrix, a matrix spike and matrix spike duplicate are analyzed both with and without the distillation step. (A total of 36 samples, assuming 9 matrices). If results are comparable, the laboratory may dispense with the distillation step for future analysis. Comparable is defined as <20% RPD for all tested matrices). Alternatively the two populations of spike recovery percentages may be compared using a recognized statistical test.”

Supplemental Information:

The following information is provided to laboratories as clarification regarding performance and evaluation of a comparability study acceptable to Virginia Department of Environmental Quality (DEQ) for the purpose of determining the necessity of routine distillation (hereafter, “distillation study”):

1. Based on language in the methods, the following ammonia methods *do not require distillation*: Electrode Methods: SM 4500-NH3 D-1997, SM 4500-NH3 E-1997, and ASTM D1426-08 (B); Ion Chromatography Method: ASTM D6919-09. No distillation study is required to omit distillation for these methods.
2. Approval for omission of routine distillation:
 - a. is given per each permitted outfall (also called “matrix”), when ammonia is required by the outfall permit;
 - b. is given based on a comparison study done by a VELAP-accredited laboratory;
 - c. is method-specific;

- d. is not laboratory-specific. As such, the distillation study may be subcontracted to a VELAP-accredited commercial laboratory performing the same method for which exemption is sought.
3. The distillation portion of sample processing may be subcontracted to a VELAP-accredited commercial laboratory that holds accreditation for an approved ammonia method requiring distillation for the purpose of completing a distillation study or resolving a matrix issue on routine samples.
4. A written approval from VELAP for the omission of distillation for a specific permitted outfall is required before distillation of samples for analysis of Ammonia (as N) in compliance samples may be omitted.
5. Distillation study data submitted to VELAP for review must be presented in a fashion as described in Footnote 6, to include the laboratory's raw data and %RPD calculations and sampling information sufficient to demonstrate that the requirements for evaluation of data have been met.
6. Use form DGS-35-596, Distillation Variance Application, or a comparable substitute with information organized similarly, to submit summarized data for VELAP review. The form is available for download on the VELAP page at www.dgs.virginia.gov/dcls.
7. Associated raw data must be made available upon request.
8. Distillation study data will be reviewed by VELAP for acceptability according to the requirements of 40 CFR 136.3 Table 1B Footnote 6.
9. VELAP may seek DEQ's assistance in review of data.
10. VELAP will maintain records of the data review for at least the minimum VELAP record retention time [5 years] or longer upon request by DEQ.
11. DEQ may request access or review of VELAP records on distillation studies at any time.
12. The 40 CFR 136.3 Table 1B footnote 6 provisions for omission of distillation studies require that distillation be used to "resolve any controversies". An example of a "controversy" is matrix spike failure. A lab that does not have distillation capabilities will be required to subcontract any controversial analyses, or specifically any samples in which the matrix spike does not meet quality control evaluation requirements.
- 13. A DISTILLATION STUDY WILL CONTAIN, PER REGULATED OUTFALL:**
 - Identifying information on the regulated outfall (name, address, permit number, contact information);
 - 9 matrix samples collected in the same manner as permit requirements (e.g., 8 hr composite; 24 hr composite; grab), with sufficient volume for 4 analyses. This sample type information will be recorded on the data summary;
 - Grab samples, if submitted, collected on separate days. The sample date/time will be recorded in the data summary. The laboratory will maintain records demonstrating that preservation and hold time requirements were met;
 - Analysis of a matrix spike and a matrix spike duplicate, both with distillation and without distillation, from each of the 9 matrix samples. The spiked sample concentrations must be between the lowest and highest calibration standards to be considered part of the study; and
 - Calculation of Relative Percent Difference (RPD) for each comparison pair (i.e., with distillation / without distillation).
 - A complete distillation study will include 36 analyses (9 matrix samples x 4 analyses per matrix = 36 analyses).

14. Additional consideration for COMMERCIAL LABORATORIES: **A commercial laboratory opting to omit distillation must demonstrate its documented procedure and process for assuring that distillation is omitted only for samples associated with an approved variance given by VELAP.**
15. An approval given by VELAP for omission of distillation applies to the effluent as tested in the study.
16. **Any change or potential change to the characteristic of the effluent requires a re-application for variance and a repeat of the distillation study.**