MANUAL FOR THE CERTIFICATION OF LABORATORIES ANALYZING ENVIRONMENTAL SAMPLES FOR THE IOWA DEPARTMENT OF NATURAL RESOURCES

Criteria and Procedures
Quality Assurance

Chapter 3. Wastewater and Sewage Sludge

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(Section # 7 Extract).
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7. Quality Assurance.

7.1 **General requirements**. **All quality control information must be available for inspection by IDNR at any time or by UHL during site visits.** A manual of analytical methods and the laboratory's QA plan are also to be available to the analysts.

7.1.1 **QA Plan**. All laboratories analyzing compliance samples must adhere to defined quality assurance procedures. This is to insure routinely generated analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy. **To accomplish these goals, each laboratory must prepare a written description of its quality assurance activities (a QA plan). The following items should be addressed in a QA plan:**

a. Sampling procedures;

b. Sample handling procedures--specify procedures used to maintain integrity of all samples. Samples likely to be the basis for an enforcement action may require Chain-of-Custody procedures.

c. Instrument or equipment calibration procedures and frequency of their use.

d. Analytical procedures.

e. Data reduction, validation and reporting
data reduction: conversion of raw data to final concentrations.
validation: includes insuring accuracy of data transcription and calculations.
reporting: includes procedures and format for reporting data to clients and IDNR

f. Types of quality control (QC) checks and frequency of their use. This may include preparation of calibration curves, instrument calibrations, replicate analyses, use of external QC check samples, and use of QC charts.
g. Preventive maintenance procedures and schedules.
h. Specific routine procedures used to determine data precision and accuracy for each contaminant measured. Precision is based on the results of replicate analyses. Accuracy is normally determined by comparison of results with "known" concentrations in spiked media.
i. Corrective action contingencies. Laboratory response after obtaining unacceptable results from analysis of PE samples and from internal QC checks.
j. Laboratory organization and responsibility. Include a chart or table showing the laboratory organization and line of authority. List the key individuals who are responsible for ensuring the production of valid measurements and the routine assessment of measurement systems for precision and accuracy (e.g., who is responsible for internal audits and reviews of the implementation of the plan and its requirements.

The QA plan may be a separately prepared QA document or may incorporate, by reference, already available standard operating procedures (SOPs) that are approved by the laboratory director and address the listed items. If a particular listed item is not relevant, the QA plan should state this and provide a brief explanation (e.g., some laboratories do not collect samples and thus are not required to describe sampling procedures but should be aware of IDNR requirements for sampling). A laboratory QA plan should be concise but responsive to the above-listed items. Minimizing paperwork while improving dependability and quality of data are the intended goals. The QA Plan should describe how and what the laboratory is actually doing, not theory or suggested practices.

7.1.2 Class S weights or better should be available to make periodic checks on balances. A record of these checks is to be available for inspection. The specific checks and their frequency are to be as prescribed in the laboratory's QA plan and the laboratory's operations manual, if appropriate. This frequency should not exceed annually.

7.2 **Analytical Quality Control**. The following are required for each method:

7.2.1 **The laboratory must perform acceptably on PT samples at least annually by each method/matrix. The proficiency testing program in which the laboratory is enrolled must meet the criteria listed under "Criteria for Proficiency Programs" in this manual.**

7.2.2 **At least once each quarter, the laboratory must analyze a QC sample independent of the materials and preparation of calibration standards**. If the specified limits are exceeded, corrective action is to be taken and documented, and

a follow-up quality control standard analyzed as soon as possible to demonstrate the problem has been corrected.

7.2.3 **Calibration and quality control must be followed as specified in the method.** In general, a standard curve composed of at least a reagent blank and three standards covering the sample concentration range are to be prepared. These standards should be from a different source than the quality control standard used for 7.2.2.

7.2.4 At the beginning of each day that samples are to be analyzed, the standard curve is to be verified by analysis of **at least a reagent blank and one standard** in the expected concentration range of the samples analyzed that day. All checks should be within +/- 20% of the standard curve or the system must be recalibrated.

7.2.5 If the reagent blank specified in 7.2.4 is not carried through the full analytical procedure, **then some other blank** (at least one per day) is to be carried through the entire analytical procedure. Results from reagent blanks should not exceed the laboratory's method reporting limit (see paragraph 7.2.8).

7.2.6 **The laboratory must analyze known spikes and sample duplicates on a regular basis at a frequency of a minimum of 5% of the number of samples analyzed as a group (all samples being analyzed by the same procedure at one time).** If the analyte is normally below the laboratory detection limit duplicate known spikes must be analyzed. The known spike and sample duplicate are to be analyzed through the complete analytical system. Corrective action is to be taken in accordance with the laboratory's QA plan if any duplicate or spike result is out of the laboratory's statistical acceptance range or as stated in the referenced method (generally not to exceed 100 +/- 40% for organic or 100 +/- 20% for inorganic analysis) unless the laboratory can demonstrate matrix effect in the case of matrix spikes. **Statistically based acceptance limits should be established once sufficient data is available.**

7.2.7 **The laboratory must calculate traditional control limits on an on-going basis** for each analyte. The laboratory may use quality control criteria in the sections above more stringent than those stated, if their experience with on-going analytical operations demonstrates such limits to be appropriate for their operations.

7.2.8 It is further recommended the laboratory calculate the MDL at least annually in accordance with the procedure given in 40 CFR Part 136, Appendix B