

Standard Operating Procedures (SOPs)

The 2008 revisions to NR149 contain many new requirements regarding laboratory SOPs. Laboratories must have SOPs (written documents) that detail lab activities and procedures. The code allows a great deal of flexibility in the way the SOPs are designed which leaves it up to the lab to decide. The SOPs may be in electronic form or hard-copy.

The code requires that for each method being performed that the laboratory generate a specific analytical SOP. In addition to the analytical SOPs, the code requires that the lab document their procedures for at least 19 specific subjects. These 19 issues can be documented in separate SOPs, grouped together in one SOP, or documented in the Quality Manual as long as they are all covered.

Analytical SOPS

Analytical SOPs are SOPs that describe exactly how the lab performs the method. There are generally three ways for the lab to create their own analytical SOPs.

Option 1: the analytical SOP may consist entirely of the referenced published analytical method. Keep in mind that this option will only work if the lab follows the method exactly (very unlikely).

Option 2: the lab may reference a published method and include an addendum that details exactly where the lab deviates from the published method. Note that when this option is used, the addendum must include a date of issue or a date of revision. Generally speaking, this option is considered the most practical approach for small wastewater laboratories.

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Option 3: the lab may choose to create their own SOP from "scratch" . If this option is chosen the code specifically requires that the SOP includes each of the following elements:

1. Identification of the test method
2. Applicable analytes
3. Applicable matrices
4. Method sensitivity
5. Potential interferences
6. Equipment and analytical instruments
7. Consumable supplies, reagents and standards
8. Sample preservation, storage and hold time
9. Quality control samples and frequency of their analysis
10. Calibration and standardization

- 11. Procedure for analysis
- 12. Data assessment and acceptance criteria for quality control measures
- 13. Corrective actions and contingencies for handling out of control or unacceptable data

Analytical SOPs created from “scratch” must include a date of issue or a date of revision. These SOPs can be done in any format/style the lab chooses as long as all of the required elements are included and understandable.

Analytical SOPs can be kept in an “analytical methods manual”, can be included as part of the quality manual or can be kept as individual SOPs.

As with all other procedures in the laboratory, SOPs should be reviewed periodically to ensure that they remain current.

General SOPs

The code requires that each laboratory have documented procedures for all of the following subjects:

Calibration	Procedures for calibration, verification and maintenance of major analytical instruments and support equipment.	149.37(3)(f)
Equip. maintenance	Procedures for the maintenance of analytical instruments <u>to prevent contamination or deterioration</u> that may affect reported results.	149.44(4)(b)
QC: Blanks & zeroing	Procedures for zeroing an instrument and the treatment of calibration blanks , when the referenced analytical method used by the lab requires the response of a calibration blank to be part of a calibration function.	149.44(6)(h)
QC: confirmation of organic analytes	Procedures to confirm the results of organic analytes determined by techniques that, unlike mass spectrometry, do not provide a positive unique identification when... <i>1. The history of a sample source does not suggest the likely presence of the detected analyte. 2. A client or approved project plan requires it.)</i>	149.48(9)(a)
Corrective action	Procedures for initiating, following up on and documenting corrective action addressing QA and QC failures, discrepancies or nonconformance.	149.37(3)(h)
QC: LOD & LOQ	Procedures to relate LODs to LOQs.	149.48(2)(f)
QC Samples	Procedures for evaluating QC samples , including, but not limited to, method blanks, LCS, matrix spikes (MS), and replicates.	149.37(3)(g)

QC sample batches	Procedures for identifying and documenting preparation batches that facilitate determining compliance with the frequencies of QC samples.	149.48(1)(d)
Records	Procedures for document retention, control and maintenance.	149.37(3)(b)
Records	Procedures to control and manage all records and documents that form part of its quality system and that are required to demonstrate compliance with this chapter. The procedures shall ensure that documents required to perform analyses and to ensure the quality of generated data are available to lab personnel, and that records and documents are reviewed periodically for continuing suitability and, when necessary, revised to facilitate compliance with the requirements of this chapter.	149.39(1)(a)
Records	Procedures to prevent unauthorized access or amendments to records and documents.	149.39(1)(g) 4.
Reporting	Procedures for reviewing analytical data and reporting analytical results. (Required to be in Quality Manual)	149.37(3)(i)
Reporting	Procedures and rules for reporting results for samples analyzed by dual column and dual detector systems. These procedures must declare: (1) Under what conditions a presumptive identification is confirmed. (2) Under what conditions a presumptive identification is reported. (3) The value that will be reported when the dual systems both provide quantitative confirmed results	149.48(9)(b)
Sample handling	Sample Acceptance Policy. The lab shall have and follow a written policy that clearly outlines the conditions under which samples will be accepted or rejected for analysis, or when reported results will be qualified.	149.46(2)(a)
Sample handling	Procedures for handling samples. (Required to be in Quality Manual)	149.37(3)(d)
Sample handling	Procedures for identifying samples uniquely. (The procedures shall ensure that the identity of samples cannot be confused physically or when referenced in records or other documents.)	149.46(3)(a)
Sample Storage	Procedures and appropriate facilities for avoiding deterioration, contamination, loss or damage of samples during storage.	149.46(6)(a)
Sample Containers	When the lab provides containers and preservatives for sample collection, including bulk sampling containers	149.46(1)(b)

	<p>such as "carboys", the lab shall have SOPs in place which address concerns that the containers are adequately cleaned and not contributing to contamination of samples, do not contain analytes of interest at levels which will affect sample determinations and that the preservatives used are sufficiently pure to maintain the validity of reported results.</p> <p>NOTE: The laboratory should establish procedures to ensure and document that the sample containers it provides do not contribute contaminants before they are used for collecting samples.</p>	
Traceability	<p>Procedures for achieving traceability of standards, reagents, and reference materials used to derive any results or measurements. (Required to be in Quality Manual)</p>	149.37(3)(c)

There is no required format to document these procedures. As with the analytical SOPs be sure to include a date of issue or date of revision with each of these procedures and to review them periodically.

Option 2 Example

USGS Method I-3765-85 is approved for determination of TSS in wastewater samples. By generating a brief addendum (see next page) that can be attached to the referenced method, a lab can satisfy the SOP requirements in NR 149.

Solids, residue at 105°C, suspended, gravimetric

Parameter and Code:

Solids, residue at 105°C, suspended, I-3765-85 (mg/L): 00530

1. Application

This method may be used to determine the suspended-solids concentration of any natural or treated water or industrial waste.

2. Summary of method

2.1 Suspended solids are those that are retained on a glass-fiber filter. The determined value is fairly representative of the sample but does not accurately represent the suspended sediment concentration of a stream; suspended solids values should not be confused with sediment concentration, which is the more accurate measure of material in suspension.

2.2 The unfiltered sample is mixed thoroughly and an appropriate volume is rapidly poured into a graduated cylinder. The suspended solids are collected on a glass-fiber filter, and the insoluble residue is dried and weighed.

3. Interferences

Precipitation in the sample during storage, such as iron, will produce erroneously high results.

4. Apparatus

4.1 *Desiccator*, charged with indicating silica gel or other efficient desiccant.

4.2 *Filtration apparatus*, consisting of suction flask, gooch crucible, glass-fiber filter disk, and suitable holder.

4.3 *Oven*, 105°C, uniform temperature throughout.

6. Procedure

6.1 Shake the sample bottle vigorously and rapidly pour a suitable volume into a graduated cylinder. Record the volume.

6.2 Quantitatively collect the suspended material from the sample on a tared glass-fiber filter disk. A blank should be determined with each set of samples.

6.3 Wash the suspended material on the filter sparingly with demineralized water.

6.4 Dry the residue and filter disk overnight at 105°C.

6.5 Cool in a desiccator and weigh the filter disk containing the dry residue to the nearest 0.1 mg. Record the weight.

7. Calculations

7.1 Apply a correction for any loss shown by the blank.

7.2 Determine suspended solids in milligrams per liter as follows:

Suspended solids, mg/L =

$$\frac{1000}{\text{mL sample}} \times \text{mg residue}$$

8. Report

Report solids, residue at 105°C, suspended (00530), concentrations as follows: less than 1,000 mg/L, whole numbers; 1,000 mg/L and above, three significant figures.

9. Precision

Precision data are not available for this method.

Reference

Guy, H. P., 1969, Laboratory theory and methods for sediment analysis: Techniques of Water-Resources Investigations of the U.S. Geological Survey, book 5, chapter C1, 58 p.

Addendum to USGS Method I-3765-85

ACME SOP # TSS- I-3765-85

Revision 0, July 2008

ACME Labs follows USGS method I-3765-85 for the analysis of TSS with the exception of the following modifications or clarifications where the method presents flexibility or options.

- 4.2 – Instead of Gooch crucibles, use Millipore filter funnel, 45 mm filters.
- 4.3, 6.4 - Oven temperature is allowed to be 103-105 °C (104 ± 1 °C).
- 6.1 - Generally use 500 mL for effluent, 25 mL for influent. Sample is stirred continuously with magnetic stir bar until sample aliquot is removed.
- 6.2 - Blanks are not analyzed, an exemption allowed in ch. NR 149.14(3)(d)
- 6.4- Samples are dried overnight (at least 8 hours). The laboratory performs a verification of drying effectiveness quarterly as per DNR letter (May 2001).
- 7.1- No correction for blank results is allowed by NR 149.
- NOTE 1. Sample preservation is not to exceed 6 °C and not to be frozen.
- NOTE 2. Filters from NCL, deemed to be equivalent to method specs.
- NOTE 3. Use 3 x 25-mL portions of reagent water to wash filters.
- NOTE 4. Filters undergo 3X final wash of 20, 20, and 10 mL of reagent water.