NORTH CAROLINA WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH
APPROVED PROCEDURE FOR THE ANALYSIS OF TURBIDITY
According to Standard Methods 2130 B – 2011

This document provides an approved procedure for the analysis of Turbidity for compliance monitoring per 15A NCAC 02H .0805 (a) (7) and (g) (4).

Turbidity is considered a method-defined parameter per the definition in the Code of Federal Regulations, Part 136.6, Section (a) (5). This means that the method may not be modified per Part 136.6, Section (b) (3).

Holding Time:

- Samples must be analyzed within 48 hours of collection (40 CFR Part 136.3 Table II).

- Samples must be stored above freezing and ≤6 °C, if not analyzed immediately.

General Information:

- Sample duplicates are not a required quality control element for Field parameters.

- A nephelometer must be used for the analysis of Turbidity.

- Turbidity samples are not to be diluted unless their concentration falls outside the allowable calibration range of the meter.

- Inline turbidimeters must be calibrated, if possible, daily with Primary standards or Secondary sealed standards. Direct calibrations must meet the meter manufacturer's acceptance criteria, if available. If no acceptance criteria are available, the laboratory must establish performance acceptance criteria. If direct calibration is not possible due to the configuration of the meter, daily calibration verification must be performed by analyzing a sample from a bleed line or the outflow with a calibrated hand-held or benchtop turbidimeter and comparing results to the inline reading. Readings must agree within ± 20%. Inline meters used in conjunction with reclaimed water permits shall be calibrated and/or verified according to the procedures above except that the frequency will be annually, instead of daily.

- A post-analysis calibration verification must be performed at the end of the run anytime the meter is transported by vehicle to another location after calibration. It is recommended that a mid-day or a mid-batch calibration verification be performed when samples are analyzed over an extended period of time. The value obtained for the post-analysis calibration verification check-standard must read within 10% of the standard's true value. If the obtained value is outside of the ±10% range, corrective action must be taken. If recalibration is necessary, all samples analyzed since the last acceptable calibration verification must be reanalyzed, if possible. If samples cannot be reanalyzed, the data must be qualified.

Interferences

- Light-absorbing materials such as activated carbon (when present in significant concentrations) or presence of dissolved, color-causing substances can cause a negative interference. Samples with very high turbidity and/or colored samples may benefit from analysis using the Ratio mode that is available on some meters.

- Dirty glassware and air bubbles give false results. Remove air or other entrained gases in the sample before measurement. Preferably degas even if no bubbles are visible. Degas by applying a partial vacuum, adding a nonfoaming-type surfactant, using an ultrasonic bath, or applying heat. In some cases, two or more of these techniques may be combined for more effective bubble removal. For example, it may be necessary to combine addition of a surfactant with use of an ultrasonic bath for some severe conditions. Any of these techniques, if misapplied, can alter sample turbidity; use with care. If degassing cannot be applied, bubble formation will be minimized if the samples are maintained at the temperature and pressure of the water before sampling.

- Keep the sample cell clean when not in use. Handle sample cells only by the top to avoid dirt and fingerprints within the light path. Discard when they become scratched or etched. A light coating of silicon oil may be used to mask minor imperfections in glass tubes.

- Condensation may occur on the outside surface of a sample cell when a cold sample is being measured in a warm, humid environment. This interferes with turbidity measurement. Remove all moisture from the outside of the sample cell before placing the cell in the instrument. If fogging recurs, let sample warm slightly

by letting it stand at room temperature or by partially immersing it in a warm water bath for a short time. Make sure samples are again well mixed.

Definitions:

- A nephelometer is defined as a turbidimeter with scattered-light detectors located at 90° to the incident beam.

- NTU: Nephelometric Turbidity Units

- Primary standards are defined as liquid suspensions prepared from hydrazine sulfate and hexamethylenetetramine or a commercially certified stock formazin suspension.

- Secondary standards are defined as commercially prepared, stabilized, sealed liquid or gel turbidity standards calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymers.

Meter:

To minimize differences in measured values due to instrument design, the following design criteria must be met:

- Tungsten-filament lamp.
- Total distance traversed by incident light and scattered light within the sample tube is not to exceed 10 cm.
- Detector centered at 90° to the incident light path and not to exceed ±30° from 90°. The detector and filter (if used) shall have a spectral peak response between 400 and 600 nm.
- Instruments with an LED or Laser light source are not compliant with the SM 2130 B-2011 method.

Standards:

CAUTION: Hydrazine sulfate is a carcinogen; avoid inhalation, ingestion, and skin contact. Formazin suspensions can contain residual hydrazine sulfate.

Stock Primary standard preparation instructions:

1. Solution 1 – Dissolve 1.000 g hydrazine sulfate, $(NH_2)_2 \cdot H_2SO_4$, in distilled water and dilute to 100 mL in a volumetric flask.

2. Solution 2 – Dissolve 10.00 g hexamethylenetetramine, $(CH_2)_6N_4$, in distilled water and dilute to 100 mL in a volumetric flask.

3. Stock Solution – Mix 5.0 mL Solution 1 and 5.0 mL Solution 2 in a flask. Let stand for 24 hours at 25 ± 3°C. This makes a 4000 NTU suspension. Store in amber glass or other UV-light-blocking container. Stock solution is stable for up to 1 year. Prepare calibration standards from this stock solution for calibration.

   Note: prepare stock standard and calibration standards using reagent water passed through a filter with pore size no larger than 0.1 μm. The usual filter used for bacteriological examinations is not satisfactory for this.

Secondary standards may be used beyond their expiration date only if their concentrations are verified against a primary standard by the date of expiration and quarterly thereafter and are shown to be within 10% of their original concentration. This must be documented and kept on file.

Procedure:

- Allow instrument sufficient time to warm up.
- Calibrate the meter according to manufacturer's instructions, with at least one calibration or verification standard in each instrument range to be used.
- Gently agitate sample to ensure homogeneity and wait for air bubbles to disappear.
- Pour sample into sample vial. Ensure no air bubbles are present.
- Wipe vial with a lint-free cloth or Kimwipe to remove any condensation, moisture or fingerprints. A thin layer of silicon oil may be applied (optional).
- Gently invert the sample to ensure homogeneity and place in turbidimeter.

- Close chamber lid and read turbidity from instrument display at first stable reading.

- Document turbidity measurement from instrument display.

- If sample dilutions are performed, analyze the dilution water to demonstrate a concentration ≤ 0.02 NTU.

- Report turbidity readings, along with qualification if applicable, as follows:

Measured Turbidity Range *NTU*	Report to the Nearest *NTU*
0 – 1.0	0.05
1 – 10	0.1
10 – 40	1
40 – 100	5
100 – 400	10
400 – 1000	50
> 1000	100

Documentation:

The following must be documented in indelible ink whenever sample analysis is performed:

1. Date and time of sample collection
2. Date and time of sample analysis to verify the 48-hour holding time is met
3. Permitted facility name or permit number, and sample site (ID or location)
4. Collector's/analyst's name or initials
5. Meter calibration and meter calibration time(s)
6. True values of the calibration and/or verification standards (determined by the manufacturer's calibration procedure)
7. True value and value obtained for the post-analysis calibration verification(s), when applicable
8. Quality control assessments
9. Indication of when the post-analysis calibration verification was performed (e.g., time of analysis, end-of-day analysis, etc.)
10. Measured value
11. Units of measure (NTU)
12. Traceability for chemicals, reagents, standards and consumables
13. Unique instrument identification (serial number preferred)
14. Parameter analyzed
15. Method reference or Standard Operating Procedure
16. Data qualifiers, when necessary

Refer to http://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies for additional quality assurance and quality control requirements.

Ref: Standard Methods 2130 B – 2011