DETERMINATION OF TURBIDITY IN WATER USING THE HACH TL2300 TURBIDIMETER

TABLE OF CONTENTS:

Page

1. SCOPE AND APPLICATION 2
2. SUMMARY OF THE METHOD 2
3. EQUIPMENT AND SUPPLIES 2
4. REAGENTS AND STANDARDS 3
5. FORMS 4
6. SAMPLE PREPARATION, PRESERVATION, AND STORAGE 4
7. SAMPLE ANALYSIS/QUANTITATION 4
8. POST RUN PROCEDURE 11
9. DATA ARCHIVAL 12
10. QUALITY CONTROL AND CORRECTIVE ACTIONS 13
11. MAINTENANCE 15
12. SAFETY/HAZARDOUS WASTE/POLLUTION PREVENTION 16
13. REFERENCES 16

APPENDIX OF SIGNIFICANT CHANGES 17

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1. SCOPE AND APPLICATION

1.1. This method is applicable to drinking, surface and saline waters in the turbidity range from 0.10 to 40 nephelometric turbidity units (NTU). Higher sample values may be measured by quantitative sample dilution. The test covered by this SOP is TURBIDITY.

Key Parameters	
Method Detection Limit (MDL)	0.10 NTU
Practical Quantitation Limit (PQL)	1.0 NTU
LFB/ALTQC	10 NTU

2. SUMMARY OF THE METHOD

2.1. This SOP is based on EPA Method 180.1, Rev. 2.0 (1993), as approved at 40 CFR part 136. The Quarterly Initial Calibration and Calibration Verification are performed per manufacturer's instructions manual.

2.2. This method compares the intensity of light scattered at 90 degrees by the sample with the intensity of light scattered by a standard (STD) reference suspension under defined conditions. The higher the intensity of scattered light, the higher the turbidity.

2.3. Interferences: The presence of floating debris and coarse sediments which settle out rapidly will result in variable readings. Positive interference may be caused by finely divided air bubbles. The presence of true color, i.e. the color of water due to dissolved substances which absorb light, can interfere with turbidity readings.

3. EQUIPMENT AND SUPPLIES

3.1. Instrumentation

Instrument Unique ID	Component	Manufacturer	Model	Software
HACH T-1	Turbidimeter	Hach	TL-2300	6.3.6
HACH T-2	Turbidimeter	Hach	TL-2300	6.3.6

3.2. Equipment

Equipment	Manufacturer/Vendor[1]	Model/Catalog Number
Volumetric Pipette, Class A, Assorted	Fisher (SR)	13-690-110; 13-681-50
Glass Turbidity Cells	Hach	2084900
Volumetric Flask, Class A, Assorted	Fisher (SR)	10-212B; 10-210-5D; 10-212E; 10-212-5L; 10-208M
Graduated Cylinder, Class A, Assorted	Fisher (SR)	08-553A; 08-548D
Glass Beaker, Assorted	Fisher (SR)	02-540E; 02-540G; 02-540K; 02-555-20E
Oiling Cloth	Hach	4707600
USB Flash Drive	SanDisk/Office Depot	801187
Or Equivalent [1]**Note:** (SR) designates equipment is typically ordered through the in-house stockroom		

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3.3. Consumables and frequently replaced supplies

Supplies/Consumables	Vendor[1]	Catalog Number
Kimwipes X-Lg	Fisher (SR)	06-666C
Silicone Oil	Hach	126936
Wash Bottle	Fisher (SR)	03-409-22C
Reagent Bottle, Polyethylene	Fisher (SR)	05-719-88
Parafilm	Fisher (SR)	13-374-12
Compressed Air	Office Depot	911220
Or Equivalent		

[1]**Note:** (SR) designates supply is typically ordered through the in-house stockroom

4. REAGENTS AND STANDARDS (SEE DEP SOPS NU-041 and NU-042)

Note: Prior to preparing standards or running samples, read the safety notes in the safety/hazardous waste section of this SOP and the MSDS for the chemicals used in this analysis.

Note: Purchased and prepared standards must be documented in the Laboratory Information Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for standards are generated in the module and documented in each analysis folder. Follow preparation and labelling instructions in DEP SOP NU-042. Certificates of Analysis (CoAs) for purchased standards are maintained in a folder for Turbidity analysis on the Nutrients drive at: \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\MetaData\Turbidity\CertificatesofAnalysis\. All containers holding standards, i.e. each standard, must be labeled with their unique ID and expiration date.

4.1. Chemicals

	Chemical	Alias	Manufacturer	Catalog Number	Purity	Storage	Notes
4.1.1.	StablCal® Turbidity Standard Kit, 0-4000 NTU	TUR-CAL SET	Hach	2662105	See COA	Room Temp	Unique IDs generated for each separately in the Standard Preparation Tracker.
4.1.2.	StablCal® Turbidity Standard, 1.0 NTU	TURBIDITY-STD-1.0 NTU	Hach	2659853	See COA	Room Temp	Poured fresh or kept sealed and stored in the dark, as needed. Follow preparation instructions on bottle.
4.1.3.	AmcoClear 10 NTU Turbidity Standard	10 NTU-ALTQC	Fisher (GFS Chemical)	NC187655 9 (87277)	See COA	Room Temp	Poured fresh or kept sealed and stored in the dark, as needed. Follow preparation instructions on bottle.
4.1.4.	Gelex Stray Light Turbidity Standard	GELEX STRAY LIGHT	Hach	2589100	See COA	Room Temp	Serves to measure the drift of the instrument for maintenance purposes. Stored in the dark.
4.1.5.	DI Water	H2O	In house	N/A	N/A	N/A	Used for all blanks (BLK, MBLK, CCB). Poured fresh each use.
4.1.6.	StablCal® Turbidity Standard, 10 NTU	10 NTU LFB	Hach	2659902	See COA	Room Temp	Sealed vial.
Or Equivalent							

Deionized water is replaced daily. All other purchased chemicals are given the expiration

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date from the CoA and/or chemical container. If there is not an expiration date listed, the expiration date is designated by following the protocol in SOPs NU-041 and NU-042.

5. FORMS

5.1. There are four digital forms referenced in this SOP and associated with the operations of Hach T-1 (HT) and Hach T-2 (TT) Turbidimeters.

- Form **NU-TURBIDCOVER**
- Form **NU-TURBSHEET**
- Form **NU-TURB-CAL**
- Form **NU-PIPETTE-Turbidity**

The forms are maintained in the Templates folder located at: \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\metaData\Turbidity\Forms\.

Note: Always ensure that most recent version of the digital forms are used to document and store data.

6. SAMPLE PREPARATION, PRESERVATION, AND STORAGE (SEE DEP SOP NU-033)

6.1. Upon receipt, samples are stored in the Nutrient refrigerators in the receiving room of the DEP laboratory at ≤ 6°C (not frozen). **Samples must be analyzed for turbidity within 48 hours of collection or results will be Q qualified.**

6.2. Remove the samples from the refrigerator following the proper protocols to preserve the samples' chain of custody. Allow samples to come to room temperature or use a warm water bath. Condensation on the cells will affect results.

6.3. Invert the sample bottle well to thoroughly disperse any solids. Visually inspect a cell for scratches or other defects which can cause false readings. Set aside damaged vials to be tested later. To condition the vial, fill it approximately one third full with sample and carefully rotate the vial while discarding it into a waste beaker. Fill the cell with sample to a level above the line on the cell. Pour and perform one sample for analysis at a time.

6.4. Cap the cell and wipe with a Kimwipe to remove water spots and fingerprints. Wait for tiny air bubbles to disperse and rotate as needed if any bubbles have settled on the glass. Invert gently before putting into instrument. Align the arrow on the vial to the corner of the holder.

7. SAMPLE ANALYSIS/QUANTITATION (SEE DEP SOP NU-033)

7.1. **Instrument settings for calibration and analysis runs on HACHT-1 and HACHT-2:**

These settings should be verified prior to the quarterly calibration and each analysis run by pushing Options > Reading Setup at the main reading screen.

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- Reading Mode: Single, Signal Avg, Time 15 s
- Unit: NTU
- Ratio: On
- Bubble Reject: On
- Data log Set Setup:
    - Auto Store: On
    - Comments: On
    - Send Data Format: CSV
    - Auto Send: Off

7.2. Verify current quarterly calibration is valid by checking the calibration sticker on the instrument. Proceed to Sample Analysis (7.6.) if calibration is valid. Proceed to the Quarterly Calibration of Turbidimeter (7.3.) if calibration is due or needed.

7.3. **Quarterly Calibration of Turbidimeter:**

Note: This is carried out according to the manufacturers' instructions as specified in the Turbidimeter user manual.

Note: Observe every measurement reading to catch any error messages. If an error message is observed during calibration, then the calibration needs to be restarted.

7.3.1. Clean the USEPA filter assembly with a Kimwipe and compressed air before calibration (11.2.).

7.3.2. Make sure the Turbidimeter has been on for at least one hour before calibration.

7.3.3. Open Form **NU-TURB-CAL** (refer to Section 5). Choose the appropriate instrument (HachT-1 or HachT-2) on the form, fill all required information including file name "**YYMMDDXX_Calibration**" (**YY-Year, MM-Month, DD-Day, XX(HT or TT)-instrument's name**), analyst's name, date/time, and all standard serial numbers, and expiration dates.

7.3.4. Remove the <0.1 NTU standard from the case containing the set of Hach StablCal® Primary standards (4.1.1). Then, invert the case containing the standards 20 times. If the <0.1 NTU standard is shaken, let it sit for at least 15 minutes due to interference caused by finely divided air bubbles.

Calibration (StablCal®) Standards Sequence
<0.1 STD-Blank
20 NTU STD (CCV)
200 NTU STD
1000 NTU STD
4000 NTU STD

7.3.5. Push the Calibration icon at the right sidebar menu on the display screen. The first calibration standard to be measured is automatically highlighted in the software screen.

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7.3.6. If necessary, oil the standard cells. Clean the outside of the standard cell with a Kimwipe to remove excess oil/debris. Place the standard in the turbidimeter aligning the arrow marks. Press Read, wait for the instrument to complete the stable reading (blue bar). Record the NTU value for the <0.1 STD-Blank on the **NU-TURB-CAL** "Calibration Curve" table in the Raw (NTU) column.

7.3.7. Repeat step 7.3.6 for the remaining StablCal® standards.

7.3.8. Once the calibration has finished, click View Graph and document the correlation coefficient (R^2) in the **NU-TURB-CAL** "Calibration Curve" table in the R^2 row.

7.3.9. Verify calibration curve meets the passing criteria (refer to 10.1.) in the "Calibration Curve" table of **NU-TURB-CAL** form. All standard and R^2 values must be acceptable. Press Store to save the calibration then proceed immediately to the quarterly calibration verification (7.4.)

Note: Repeat calibration in case of failure. Often the second calibration passes. Always report additional failure to the supervisor.

7.4. **Quarterly Calibration Verification of the Turbidimeter:**

7.4.1. **The quarterly calibration verification of the instrument must be performed immediately after a new calibration and before samples are analyzed. Before the quarterly calibration verification, execute the instrument verification by pressing the "Verification" button on the instrument panel and measuring the 20 NTU Standard. Press store.**

7.4.2. Insert the USB in the turbidimeter. Start by pushing the Sample ID icon at the right sidebar menu on the display screen of the turbidimeter and selecting Options on the bottom menu. In the dialog box that opens, select Import Sample ID List → Delete? → Yes → CalVerification Import.csv → OK.

7.4.3. Click on the first line that reads BLK then press Select. Pour DI water (4.1.5.) into a sample cell and cap it. Insert the cell, close the cover, and press Read. Record NTU value in the NU-TURB-CAL "Calibration Verification" table Raw (NTU) column.

7.4.4. Next measure the <0.1 NTU StablCal® Standard. Select Sample ID → <0.1 NTU → Select. Insert the cell, close the cover, and press Read. Record NTU value in the NU-TURB-CAL "Calibration Verification" table Raw (NTU) column.

7.4.5. Repeat the same steps as 7.4.4. for the remaining calibration verification sequence: 20 NTU, 200 NTU, 1000 NTU, 4000 NTU, 1.0 NTU, 10 NTU ALTQC, 10 NTU LFB, and Gelex Stray Light. Record NTU value in the NU-TURB-CAL "Calibration Verification" table Raw (NTU) column. Follow manufacturer's instructions for standards. If using a saved vial of the 1.0 NTU, invert gently about 5-10 times. If using a saved vial of the 10

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NTU ALTQC, do not invert.

Note: Gelex Stray Light "STD Value" is from the previous calibrations Stray Light Calibration Verification Value.

7.4.6. Verify Quarterly Calibration Verification meets acceptance criteria (refer to 10.2. and 10.3.) in the "Calibration Verification" table of form NU-TURB-CAL by ensuring all standard values are acceptable. Print and attach a label to the turbidimeter containing the instrument ID, calibration date, calibration expiration (three months from calibration date), and analyst's initials.

7.4.7. Proceed to the electronic transfer of the quarterly calibration and verification (7.5).

7.5. **Electronic Transfer of Quarterly Calibration and Verification:**

7.5.1. To view and transfer the calibration data to the USB flash drive, press Data log → Calibration Log → Filter → Off. Select the calibration just completed. Press Options → Send Data → Single Point → OK.

7.5.2. To transfer the calibration verification data to the USB flash drive, press Data Log → Reading Log → Click Filter → On → Time Interval → Set the date range → Press OK three times. Then hit Options → Send Data → Filtered Data → OK.

7.5.3. Unplug the USB from the turbidimeter and insert it into a laboratory PC.

7.5.4. In the USB logger folder, rename the calibration file to "**YYMMDDXX_ CalibrationLog**". Then rename the calibration verification file to "**YYMMDDXX_VerificationLog**".

7.5.5. Create a new folder with the Calibration's name "**YYMMDDXX_Calibration**" (**YY-Year, MM-Month, DD-Day, XX(HT or TT)-instrument's name**) in the appropriate instrument and year's calibration folder in: \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\AnalysisFolders\Turbidity\.

Note: All files associated with this calibration are saved and placed in the newly created calibration folder.

7.5.6. Save the Calibration and Verification log files in the newly created folder (7.5.5.). Periodically, delete the files from the USB drive after saving. At the top of the excel files, add 'Analyst:' and 'Reviewer:' fields, and the file names to separate cells in the files for e-signatures on the pdf versions of these files.

7.5.7. Confirm the standard raw (NTU) data entered in the **NU-TURB-CAL** form during the calibration/verification process matches the values in Calibration and Verification logs, and all values are "Acceptable" (10.1. and 10.2.).

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7.5.8. Record any maintenance performed during or prior to calibration, including filter cleaning in the **NU-TURB-CAL** form.

7.5.9. Save the **Calibration log**, **Verification log**, and the **NU-TURB-CAL** form (the form should be named matching the File Name **YYMMDDXX_Calibration**) to the folder created in step 7.5.5.

7.5.10. Re-save all files with the same names as PDF format and follow instructions in 9.2. for e-signing all documents.

7.6. **Daily Run – Sample(s) Analysis:**

7.6.1. Turn the turbidimeter on and allow it to warm up for at least one hour prior to start of analysis.

7.6.2. Create a new folder with the run's name "**YYMMDDXX#**" (**YY-Year, MM-Month, DD-Day, XX(HT or TT)-instrument's name, #-Run Number**) in the appropriate instrument, year, and month folders in: \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\AnalysisFolders\Turbidity\.

Note: All files associated with this analysis run are saved and placed in the newly created run folder.

7.6.3. Create a new sequence to import into the turbidimeter using the following procedure:

7.6.3.1. Generate the backlog in the Excel (.CSV) format using LIMS by right clicking the backlog → Save → Excel table (CSV) → OK. Save the backlog with the following name: "**YYMMDDXX#_backlog**" (**YY-Year, MM-Month, DD-Day, XX (HT or TT)-instrument's name, #-Run Number_backlog**). At the top of the file, add 'Analyst:', 'Reviewer:' fields, and file name in separate cells for the e-signatures on the pdf versions of this file.

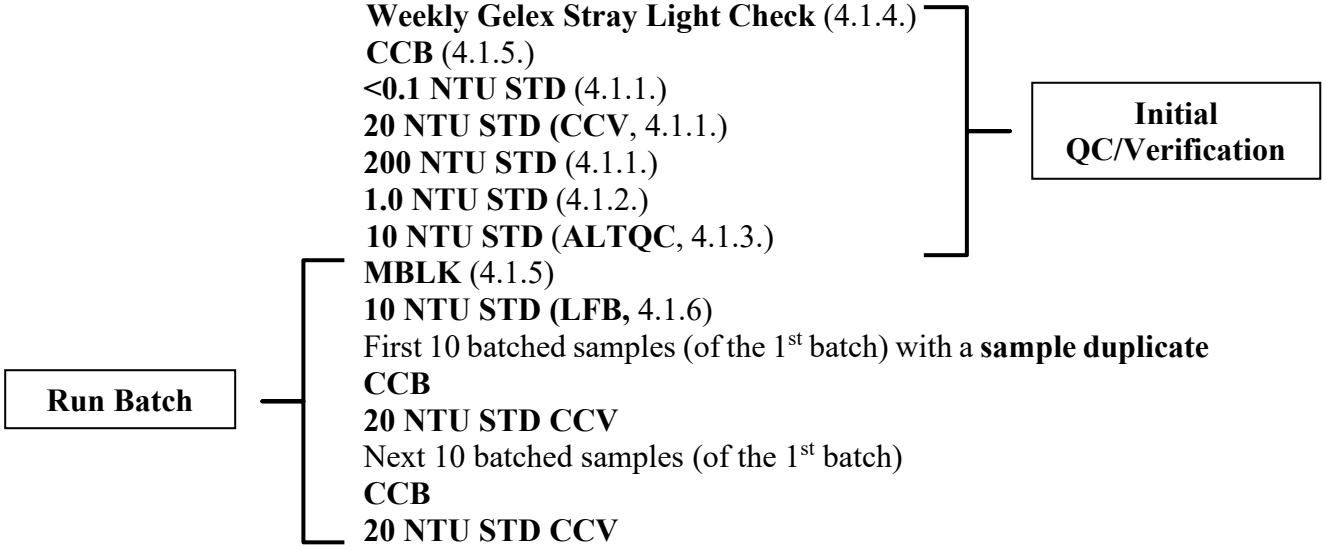
7.6.3.2. Insert the USB flash drive into a computer and open the **SampleID Import** file. Transfer the Sample IDs from the saved backlog (7.6.3.1.) into the #SampleID column starting after the initial Run and Batch QCs (CCB through 10 NTU LFB).

7.6.3.3. Save the updated **Sample ID Import** file on the USB flash drive. Insert the USB flash drive into the turbidimeter. To import the sequence, press Sample ID → Options → Import Sample ID List → Delete previously imported Sample IDs? → Yes → SampleID Import.csv → OK.

7.6.4. Open Form **NU-TURBSHEET** (refer to Section 5) on a computer. Fill-in only the file name (Analyst and Reviewer fields will be filled later) in the appropriate cell at the top of the form "**YYMMDDXX#**" (**YY-Year, MM-Month, DD-Day, XX (HT or TT)-instrument's name, #-Run Number**).

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7.6.5. On the **NU-TURBSHEET** form, create run batches beginning after the set of Initial QC (Gelex through 10 NTU ALTQC) by filling the form's Sample ID field based on the generated backlog (7.6.3.1.). Ensure each run batch (total of 20 samples) has the proper batch QC including sample duplicates (10.7.) – Follow the example run sequence below:



Note: Samples or QC with the same name, date, or time will be considered as duplicates by the turbidimeter and not transferred. An individual sample or QC, however; can be run multiple times and the multiple data points will be transferred in the correct order.

7.6.6. Weekly Gelex Stray Light Check

7.6.6.1. At the beginning of each week, the calibration must be verified with a Gelex Stray Light standard. To do this, go to the "Sample ID" tab on the instrument and select Weekly Gelex Stray Light – if Gelex Stray Light has been performed for the week (see Note below) skip to the step 7.6.7. Otherwise, wipe and insert the Gelex Stray Light cell (4.1.4.) into the meter. Ensure the arrow on the cell is aligned with instrument, close the cover, and press read. Observe and wait for the meter to automatically read and store data.

7.6.6.2. The Gelex Stray Light standard must be ± 5.0% percent of the previous Gelex Stray Light Weekly check. (10.4.) Input the Gelex Stray Light value into NU-TURBIDCOVER to verify acceptability.

7.6.6.3. The NTU value and date/time should be recorded from the reading log to the first **NU-TURBSHEET** of the week. Ensure the gelex data is archived in a reading log weekly (8.1.1.). It can be combined into a daily reading log or exported as a stand-alone reading log file named "**YYMMDDXX#_Gelex**". If already verified and archived for the week, document the date with a comment on **NU-TURBSHEET**.

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7.6.7. Complete the daily verification of calibration by running the remaining Initial QCs as listed on the **NU-TURBSHEET** form. Press SampleID on turbidimeter digital screen, select the appropriate QC, Kimwipe vial then insert the cell into the meter ensuring the arrow on the cell is aligned with the instrument. Close cover, and press read. Observe and wait for the meter to automatically read and store data. This procedure is repeated for each QC.

Note: All verification QC and Standards must meet criteria. In case of failure, repeat the verification process. Report repeated failure to supervisor.

7.6.8. Once the Initial QC is completed, follow the run batch sequence on the **NU-TURBSHEET** to initiate sample analysis. See section 6 for sample preparation details.

7.6.8.1. Press SampleID on turbidimeter digital screen then select the appropriate sample number.

7.6.8.2. Samples should be at room temperature before starting analysis. Invert sample bottle well, pour into vial, cap and wipe with a Kimwipe. **Note: Pour and perform one sample analysis at a time.** Insert the cell into the meter ensuring the arrow on the cell is aligned with the instrument. Close cover, and press read. Observe and wait for the meter to automatically read and store data.

7.6.8.3. When it is time to run batch QC, scroll up to beginning of SampleID list and select the appropriate QC.

Note: If an unstable error message is observed during sample analysis, reinvert the sample and wait for 60 seconds prior to reanalyzing the sample. When an unstable error occurs, the instrument does not record a data point.

7.6.9. Note any unusual observations of the sample, errors during the analysis, and/or reanalysis on **NU-TURBSHEET**. Check "✓" the boxes on the sheet as each sample and QC are analyzed.

Note: If the sample selected for duplicate has a turbidity value of less than 1.0 NTU or greater than 40 NTU, or exhibits unstable reading, another sample can be selected as a duplicate from the batch.

7.6.10. Equipment and/or Field blanks should have a turbidity of **LESS THAN** 0.10 NTU. If the measured turbidity is not less than 0.10 NTU, rerun the blank for confirmation on the same run. Just as the <0.1 NTU Standard, MBLK, and CCB, these samples are not inverted as this can cause a positive interference of finely divided air bubbles.

7.6.11. The precision value (relative percent difference or RPD) for the field duplicates must be less than 20 %, provided the sample value is ≥1.0 NTU (PQL). If the RPD is greater than or equal to 20%, both field duplicates should be rerun for confirmation on the same run. Note in the comment section of the digital bench sheet any visible differences between the field

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duplicates.

7.6.12. Samples with turbidities exceeding 40 NTU need to be diluted and re-analyzed. Document using form **NU-PIPETTE-Turbidity**.

7.6.12.1. Dilute the sample following the dilution factors outlined in the form using a graduated cylinder. Fill all the required fields in the form.

7.6.12.2. Select or add (see 7.6.13.) the sample ID that required dilution on the instrument panel and analyze the diluted sample. Make sure to denote the dilution factor in the Reading Log file. Save the dilution form "**YYMMDDXX#_Dilution**" (**YY-Year, MM-Month, DD-Day, XX (HT or TT)-instrument's name, #-Run number**). The file will be saved with other forms in the run's folder.

7.6.13. Imported sample IDs cannot be altered. Additional samples, dilutions or QC can be manually added to the turbidimeter by following these steps: Select Sample ID → Options → New → Type the new Sample ID → OK → Check Add Date/Time → Check Add Number → OK. Make sure that the Sample ID is not already in the sequence.

7.6.14. Once the run is complete, proceed to Post Run Procedure section.

8. POST RUN PROCEDURE

8.1. Electronic Transfer of Daily Analysis Raw Data:

8.1.1. To transfer the run reading log to the USB flash drive, select Data Log → Reading Log → Filter → On → Time Interval → Set the date range → Press OK 3 times. Then hit Options → Send Data → Filtered Data → OK.

Note: If the filtered data doesn't correctly show after choosing the time interval, double click the OK twice rapidly after setting the date or extend the date range.

8.1.2. Unplug the USB from the turbidimeter and insert it into a computer.

8.1.3. Before editing and saving the reading log file in the analysis folder, first save the raw data file in the appropriate instrument and year folder at \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RawData\Turbidimeters\.

8.1.4. The reading log will then be renamed as "**YYMMDDXX#_Reading Log**" and saved to the daily run folder. Periodically, delete the file from the USB after saving.

8.1.4.1. The log will then need some adjustments to align with the **NU-TURBSHEET**. Format the sample numbers in ascending order. Edit sample IDs as necessary to denote duplicates, re-runs, and dilutions.

8.1.4.2. At the top of the reading log file, add "Analyst:", "Reviewer:" ,

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and the "Run Name" (7.6.2.) fields to separate cells. The run name should be entered into the appropriate field but the analyst and reviewer fields will remain empty for e-signatures on the pdf versions of this file.

8.1.5. In the open digital bench sheet (**NU-TURBSHEET**) on the computer, copy and paste the "Result" column (raw data) into the 'NTU' column from the Reading Log. Make sure all sample IDs and QC match in both files. Copy and paste the Date/Time column as well. Open the newly saved file, resave it as "CSV" format with the same name.

8.1.6. Convert the CSV (Comma Separated) file from step 8.1.5. into QC Manager. Choose the appropriate instrument (GenChem: HachT-1 or HachT-2). Check that the number of significant figures has been set correctly.

8.2. Calculations:

8.2.1. Check that any sample dilution factors are uploaded and calculated correctly in QC Manager.

8.2.2. Reported data is automatically rounded in accordance with EPA Method 180.1, Rev. 2.0 (1993) rounding rules seen below.

NTU Range	0.10-1.0	1-10	10-40	40-100	100-400	400-1000	>1000
Report to Nearest	0.05	0.1	1	5	10	50	100

9. DATA ARCHIVAL

9.1. An electronic report folder (7.6.2) is created for each analytical run and saved in the appropriate instrument, year, and month folder located at: \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RunData\AnalysisFolders\Turbidity\.

The folder is labelled using **YYMMDDXX#** of the analysis run. Each folder should contain the following **in both Excel and PDF format**:

- Turbidity Cover Sheet – (YYMMDDXX#_Cover, NU-TURBIDCOVER)
- Digital Bench Sheet – NU-TURBSHEET (7.6.4.)
- Reading Log (8.1.3.)
- A sample Backlog (7.6.3.1.)
- QC Assessment uploaded from QC Manager to LIMS at the time of test authorization
- NU-PIPETTE-Turbidity form, if needed (7.6.12.)

9.2. Once all data in the Excel file formats are updated and verified for correctness, save the excel files as in PDF format. All PDFs must have electronic signature of the analyst and reviewer. Select Tools → Certificates. Digitally Sign the PDF document in the corresponding spot at the bottom of all pages or in appropriate

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field when applicable.

Note: When first signing the PDF, the changes will be saved by overwriting the existing PDF that was generated from Excel. This is the only time the file can be overwritten after changes have been made. If an error is spotted, corrections must be made in the Excel file and then saved with the file name format: **YYMMDDXX#_<Form Name>_Corrected** as both an excel and PDF file. **Add comments explaining the correction, then digitally sign the PDF.** Both the corrected Excel file and the original file with the error must be preserved and stored in the "Archive" folder (9.3.).

9.3. After the authorization of the run is completed, the authorizer must lock all the Excel files with the appropriate password. Authorizer shall create an "Archive" folder within the run folder and store all the Excel and CSV files in the newly created folder.

9.4. Method validation data is on file with the supervisor. Digital copy is also stored on the network, under an appropriate sub-folder in: \\floridadep\data\labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

9.5. About once a month or when the instrument displays a storage warning , the entire instrument reading log should be exported to a folder named "Reading Log Archive" in the appropriate Hach-T# folder in the Nutrients drive as a backup at \\floridadep\data\labs\Lab3\Chem_Data\Inorganic\Nutrients\RawData\Turbidimeters\. After saving, the data stored in the turbidimeter can be deleted. Do not delete the most current calibration log.

10. QUALITY CONTROL AND CORRECTIVE ACTIONS (SEE DEP SOPS NU-038 and NU-043)

10.1. Quarterly Initial Calibration:

Standard ID	STD Value (NTU)	Acceptable Results (NTU)	Acceptance Limits
Standard 1	<0.10	<0.10	N/A
Standard 2	20	19 ≤ x ≤ 21	95% – 105%
Standard 3	200	190 ≤ x ≤ 210	95% – 105%
Standard 4	1000	950 ≤ x ≤ 1050	95% – 105%
Standard 5	4000	3800 ≤ x ≤ 4200	95% – 105%
R^2	≥ 0.995		

Calibration is performed on a quarterly (3 months) basis per manufacturer's instructions manual using the STABLCAL® TURBIDITY CALIBRATION KIT (4.1.1.) that includes the standards outlined in the table above. Acceptable results and Acceptance Limits obtained through the calibration process are outlined in the table above. The correlation coefficient of the calibration range must be equal

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to or larger than 0.995.

10.2. Quarterly Calibration Verification:

Sample ID	STD Value (NTU)	Acceptance Results (NTU)	Recovery (%)
BLK	<0.10	<0.10	N/A
Standard 1	<0.10	<0.10	N/A
Standard 2	20	19 ≤ x ≤ 21	95% – 105%
Standard 3	200	190 ≤ x ≤ 210	95% – 105%
Standard 4	1000	950 ≤ x ≤ 1050	95% – 105%
Standard 5	4000	3800 ≤ x ≤ 4200	95% – 105%
1.0 NTU	1.0	0.90 ≤ x ≤ 1.10	90% – 110%
10 NTU ALTQC	10	9.0 ≤ x ≤ 11.0	90% – 110%
10 NTU LFB	10	9.0 ≤ x ≤ 11.0	90% – 110%
Gelex Stray Light	See 10.3.	See 10.3.	See 10.3.

Calibration Verification is performed immediately after the Quarterly Initial Calibration. In addition to the same standards (4.1.1.) analyzed during the Quarterly Initial Calibration, a BLK(CCB) (4.1.5.), 1.0 NTU (4.1.2.), 10 NTU ALTQC (standard obtained from a secondary source) (4.1.3.), and 10 NTU LFB (4.1.6.) are analyzed to verify the calibration values and ensure that the instrument is functioning properly. The acceptance limits for the recovery of all standards are outlined in the table above.

Note: The Relative Error %(s) for the calibration are assessed through the calculation of the recovery% during the Calibration Verification process.

10.3. Additionally, The Gelex Stray Light Standard (4.1.4.) is analyzed as part of the Quarterly Calibration Verification step. During the Calibration Verification, the standard should be within ± 0.02 NTU of the previous Calibration Verification value. If the Gelex Stray Light measurement does not satisfy the acceptance criteria, contact Hach Customer Service; the integrity of the optical system is at stake.

10.4. At the beginning of each run and prior to the start of analysis, the turbidimeter calibration is verified with a blank (4.1.5.), <0.1 NTU standard (4.1.1.), 20 NTU standard (4.1.1.), 200 NTU standard (4.1.1.), and 1.0 NTU standard (4.1.2.). The Acceptance Limits for all the standards are outlined in Table 10.2.

The Gelex Stray Light Standard (4.1.4.) is also run and verified on a weekly basis. If the measured value for the Gelex Stray Light standard is not within ± 5% NTU of the last Gelex Stray Light weekly check, the instrument must be recalibrated.

10.5. The continuing calibration verification (CCV, 20 NTU standard (4.1.1.)), and CCB (4.1.5.) are analyzed at the beginning, after every 10 samples, and at the end of each batch. Acceptance Limits are outlined in Table 10.2.

10.6. The 10 NTU LFB standard (4.1.6.) is run at the beginning of each batch of 20

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samples as a laboratory fortified blank (LFB) (Spike code: T1). Acceptance limits for the recovery of the LFB are 90 – 110% (Table in 10.2.). A method blank (MBLK, 4.1.5) is also run at the beginning of each batch. It must be <0.1 NTU.

10.7. Sample Duplicates - Duplicates should be run for every batch of 20 samples or less. The RPD for the duplicates should be less than 20 % provided that the duplicate values are ≥1.0 NTU. Duplicates should be visually representative of the entire batch. If the RPD fails, stop and investigate the vials and instrument response. Document findings. Reanalyze the batch starting with the MBLK. . Contact your supervisor if possible before reanalysis. Any sample requiring reanalysis must be repoured from the sample bottle. The same aliquot vial cannot be analyzed more than once.

10.8. The 1.0 NTU standard and MBLK results are used for ongoing MDL and PQL verification.

10.9 For general method performance and acceptance criteria for QC measures and corrective action for out-of-control data and contingencies for handling out-of-control or unacceptable data refer to DEP SOPS NU-038 and NU-043.

11. MAINTENANCE

11.1. The turbidimeter is kept on or in sleep mode when running daily. Complete shutdown performed before the weekend by pressing and holding the on/off button until it turns off.

11.2. Prior to performing the quarterly calibration, clean the filter (located in the cell holder compartment) by wiping with a Kimwipe and spraying with compressed air.

11.3. Treat the outside of the cells with a thin coating of silicone oil using the cloth provided and spread the oil uniformly. Then, wipe off any excess so that only a thin coat of oil is left. Wipe excess oil off the cells with a Kimwipe. Test all vials with DI water to make sure they are <0.1 NTU. Do this at least every two weeks. Every other week, the vials are wiped with an oil cloth and a Kimwipe to maintain the optics of the vials.

11.4. If vials are particularly dirty or are >0.1 NTU in the DI water test, set the vials aside to deeply clean them. Add a small amount of 1:1 HCl solution in the vial along with some DI water. Use a cotton tipped applicator to gently wipe any obvious residue off. Cap the vial and vortex or vigorously shake. Rinse thoroughly with DI water and repeat the DI water test in the instrument.

11.5. To prevent storage from becoming full, reading logs and previous calibrations that have been electronically archived (see 9.5.) need to be periodically deleted from the turbidimeter.

11.6. Maintenance performed on days when quarterly calibration is performed should be documented on the calibration form NU-TURB-CAL that will also be used to document the quarterly calibration. The file is saved per steps 7.5.9. and 7.5.10.

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Note: Any maintenance or troubleshooting performed on non-calibration days should be documented in the instrument specific Maintenance Logbook.

12. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

12.1. Review the Laboratory Health and Safety Plan (available at https://floridadep.sharepoint.com/dear/SitePages/Lab%20Health%20and%20Safety.aspx).

12.2. Read the MSDS information for safe reagent and sampling handling.

12.3. Safety glasses, gloves and lab coats should always be worn whilst handling samples and reagents.

12.4. Dispose of excess samples by flushing them down the sink with plenty of water. The 10 NTU ALTQC (4.1.3.) polymer bead standards can be disposed of down the drain. Unusable sample cells should be rinsed clean prior to their disposal in the waste glass container.

12.5. The formazin standards (4.1.1, 4.1.2., and 4.1.6) should be disposed of as hazardous waste. The sealed standards can be lab packed, labelled as hazardous/formazin, and taken to the hazardous waste room. Pour the unsealed formazin standards from the sample cells into a labelled container. Fill the cell with water to rinse and dump the rinse in the container. Once the hazardous waste container is full, it should be lab packed, labelled, and transferred into the hazardous waste room for disposal.

13. REFERENCES

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use.

13.1. EPA Method 180.1, Rev. 2.0 (1993).

13.2. Turbidimeter Manual for the Hach Model TL2300, Edition 2, 2016.

13.3. DEP SOP LB-006, Standard Operating Procedure for Records Maintenance and Storage

13.4. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

13.5. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

13.6. DEP SOP NU-041, Reagent Preparation Procedure.

13.7. DEP SOP NU-042, Standard Preparation Procedure.

13.8. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data

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Authorization Guidelines.

13.9. Quality Manual, Bureau of Laboratories, Florida DEP

13.10. Laboratory Health and Safety Plan

Appendix of Significant Changes

12/14/2020 In Section 3, the instrumentation, equipment, and supplies were updated and inputted into table format. In Section 4, Reagents and Standards were updated and inputted into table format.

12/13/2021 Separated the 10 NTU-LFB_ALTQC into a standalone LFB (HACH) and ALTQC (Fisher) from two different sources. 10 NTU ALTQC is analyzed at the start of every run as part of the Initial QC. 10 NTU LFB is analyzed at the start of every batch along with an MBLK (DI Water) as part of the Batch QC.

In the Initial QC at the beginning of each run, the 40 NTU STD has replaced the 200 NTU STD. The term BLK has been replaced with MBLK at the beginning of each batch. The term BLK has been replaced with CCB as part of continuing QC throughout the run.

Updated Section 10 (Quality Control and Corrective Actions) to include and update all the new QCs analyzed during the Calibration Verification and daily calibration check.

Added 4 digital forms for calibration, calibration verification, sample analysis runs, and dilutions as part of proper documentation and storage of data to accommodate the operations of the new instruments.

Complete revision and update of all sections to integrate instructions of operating the new turbidimeter instruments (HachT-1, and HachT-2) and documentation of data on the new digital forms.

Data Archival section has been updated to include instructions on proper archiving of the new digital forms, and requirements for e-signatures on these forms.

02/16/2022 Clarified instructions on the following:

– Uploading of run files as CSV files

– Documentation of maintenance of instruments

– Documentation of errors and corrected files

– Documentation of file name on the instrument generated data files

Provided instructions on how to resolve “unstable” or “error” readings on the instrument

Updated ALTQC storage to allow saving closed vials if stored in the dark. Clarification on Field/Equipment Blank procedure. Updated cleaning of the instrument filter.

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07/18/2023	40 NTU standard removed from analysis. 200 NTU standard has replaced the 40 NTU standard for initial QC verification.
02/18/2024	Effective date 07/18/2023: Gelex Stray Light acceptability language changed in sections 7.6.6 and 10.4. Added instructions on saving the raw data reading log daily onto the Nutrients Drive. Edited data drive file path links.
07/17/2024	Instrument verification standard changed from <0.1 NTU to 20 NTU because of instrument software update.
09/24/2024	Clarification added to section 6. Instructions for deep cleaning vials added to section 11.
09/02/2025	Software version for both instruments updated in section 3.1.
08/31/2026	Added instructions for pouring samples to Sections 6.3 and 7.6.8.2.

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