DETERMINATION OF TURBIDITY IN WATER USING THE HACH TL2300 TURBIDIMETER

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1. SCOPE AND APPLICATION

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.

MDL	0.1 NTU
PQL	1.0 NTU STD
LFB/ALTQC	10 NTU STD

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 (Refer to Section 10).

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 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
HACH T-1	Turbidimeter	Hach	TL-2300
HACH T-2	Turbidimeter	Hach	TL-2300

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
Isopropyl Alcohol	Fisher	A459-500
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 for purchased standards are maintained in the REAGENT_PREP folder for Turbidity analysis on the Nutrients drive at: \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\. All containers holding standards, i.e. each standard, must be labeled with their unique ID and expiration date.

	Chemical	Alias	Manufacturer	Catalog Number	Purity	Expiration	Storage	Notes
4.1.1.	StablCal Turbidity Standard Kit, 0-4000 NTU	TUR-CAL SET	Hach	2662105	See COA	See COA	Room Temp	Unique IDs generated for each separately in the Standard Preparation Tracker.
4.1.2.	StablCal Turbidity Standard, 1 NTU	TURBIDITY-STD-1.0 NTU	Hach	2659853	See COA	See COA. Rec. to be used within 30 days after opening.	Room Temp	Poured fresh each use or if kept sealed and stored in the dark, as needed. Follow instructions on bottle.
4.1.3.	AmcoClear 10 NTU Turbidity Standard	10 NTU-ALTQC	Fisher (GFS Chemical)	NC1876559 (87277)	See COA	See COA	Room Temp	Poured fresh each use. Follow instructions on bottle.
4.1.4.	Gelex Stray Light Turbidity Standard	GELEX STRAY LIGHT	Hach	2589100	See COA	See COA	Room Temp	Serves to measure the drift of the instrument for maintenance purposes. Stored in the dark.
4.1.5.	DI Water	H_2O	In house	N/A	N/A	Daily	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	See COA	Room Temp	Sealed vial.

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	Chemical	Alias	Manufacturer	Catalog Number	Purity	Expiration	Storage	Notes
4.1.7	StablCal Turbidity Standard, 40 NTU	TURBIDITY-STD-40 NTU	Hach	2746353	See COA	See COA	Room Temp	Poured fresh each use or if kept sealed and stored in the dark, as needed.
Or Equivalent								

5. Forms

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 New Forms folder on: \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T\Templates\.

Note: Always ensure that most recent version of the digital forms are used to document and store data. The latest version of the forms can be found in the following folder:

\\FLDEP1\DEAR\Labs\SOPs\CHEMSOPS\INORG\FORMS\

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. Condensation on the cells will affect results.

6.3. Invert the sample bottle well to thoroughly disperse any solids. Wait until any air bubbles have disappeared then rinse the cell with sample. Discard the rinse solution. Fill the cell with sample to a level above the line on the cell.

6.4. Cap the cell and wipe to remove water spots and fingerprints. Visually inspect for scratches or other defects which can cause false readings. Oil if necessary.

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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.

- 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.) then the Turbidimeter Quarterly Calibration Verification (7.4. & 7.5.) if calibration has expired.

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 isopropyl alcohol before calibration (10.2.).

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.) on a laboratory PC. Choose the appropriate instrument (Hach-1 or Hach-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 standards' S/N, 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.

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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 inthe software screen.

7.3.6. Appropriately oil and clean the outside of the standard cell with a Kimwipe. 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 10.2.) in the "Calibration Curve" table of **NU-TURB-CAL** form. All STD 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. Report repeated failure to the supervisor.

7.4. Turbidimeter Quarterly Calibration Verification:

7.4.1. **The quarterly calibration verification 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 <1.0 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.

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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, 40 NTU, and Gelex Stray Light. Record NTU value in the NU-TURB-CAL "Calibration Verification" table Raw(NTU) column.

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

7.4.6. Verify Quarterly Calibration Verification meets acceptance criteria (refer to 10.3., 10.4., 10.6., and 10.8.) in the "Calibration Verification" table of form NU-TURB-CAL by ensuring all STD 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'sinitials.

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 thedate 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_Calibration logger"**. Then rename the calibration verification file to "**YYMMDDXX_Verification logger"**.

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 \\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T.

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 logger files in the newly created folder (7.5.5). Delete the files from the USB drive after saving. Add 'Analyst' and 'Reviewer' to separate cells for e-signatures on the pdf versions of these files.

7.5.7. Confirm the STD Raw (NTU) data entered in the **NU-TURB-CAL** form during the calibration/verification process matches the values in Calibration and Verification loggers, and all values are "Acceptable".

7.5.8. Record any maintenance performed during or prior to calibration, including filter

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cleaning in the appropriate maintenance logbook (HachT-1 or HachT-2).

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

7.5.10. Re-save all files with the same names as PDF format and follow instructions in 9.3 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, and appropriate year's "Analysis Runs" folder in:
\\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T

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

7.6.3. Delete the previous sequence in the turbidimeter by selecting Sample ID → Options → Delete All Sample IDs → OK.

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

7.6.4.1. Generate the backlog in the Excel (.xls) format using LIMS by right clicking the backlog → Save → Excel table (XML) → OK. Save the backlog in the newly created folder (step 7.6.2.). Add 'Analyst:' and 'Reviewer:' fields in two separate cells for the e-signatures on the pdf versions of this file.

7.6.4.2. Insert the USB flash drive into a laboratory computer and open the **SampleID Import** file. Copy and Paste the Sample IDs from the saved backlog (7.6.4.1.) into the #SampleID column starting after the initial Run and Batch QCs (CCB through 10 NTU LFB).

7.6.4.3. 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 → OK.

7.6.5. Open Form **NU-TURBSHEET** (refer to section 5.) on a laboratory 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**).

7.6.6. 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 (7.6.4.1.). Ensure each run batch (total of 20 samples) has the proper batch QC including sample duplicates

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(10.7) – Follow the example batch sequence below:

- One MBLK (4.1.5.)
- One laboratory fortified blank (LFB, 4.1.6)
- A sample to be analyzed in duplicate to calculate precision
- A CCB and 20 NTU STD CCV after each 10th sample analyzed

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.

An example run sequence includes:

Initial QC

Weekly Gelex Stray Light Check
CCB (4.1.5.)
<0.1 NTU STD (4.1.1.)
20 NTU STD (CCV, 4.1.1.)
40 NTU STD (4.1.7.)
1.0 NTU STD (4.1.2.)
10 NTU STD (**ALTQC**, 4.1.3.)

Run Batch

MBLK (4.1.5)
10 NTU STD (LFB, 4.1.6)
First 10 batched samples (of the 1st batch) with a **sample duplicate** (7.6.6.)
CCB
20 NTU STD CCV
Next 10 batched samples (of the 1st batch)
CCB
20 NTU STD CCV

Note: If an error message is observed during analysis, then the error must be documented on the **NU-TURBSHEET**, and analysis can be repeated.

7.6.7. Turbidimeter calibration must be verified prior to each run. Initiate the verification by pressing SampleID on the turbidimeter digital screen. Select Weekly Gelex Stray Light – if Gelex Stray Light has been performed for the week (see **Note** below) skip to the step 7.6.8. Otherwise insert Gelex Stray Light cell into the meter ensuring 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.

Note: The Gelex Stray Light standard is verified weekly (e.g. the first day of analysis for that week) and must be ± 5.0% percent of the previous calibration verification value. If already checked/verified, document date/time of verification and NTU value (see 9.8.) on **NU-TURBSHEET** and skip to step 7.6.9.

7.6.8. Complete the verification of calibration by running the remaining Initial QCs as listed on the **NU-TURBSHEET** form. Press SampleID on

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turbidimeter digital screen, select the appropriate QC, inset 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.

- Blank/CCB (4.1.5.)
- <0.1 NTU standard (4.1.1.)
- 20 NTU standard (4.1.1.)
- 40 NTUstandard (4.1.7.)
- 1.0 NTU standard (4.1.2.)
- 10 NTU ALTQC (4.1.3.)

The acceptance limits for the recovery of the 20 NTU and 40 NTU standards 95 - 105%. The acceptance limits for the recovery of the 1.0 NTU and 10 NTU standards are 90 – 110% (see 9.4).

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

7.6.9. Once the Initial QC is completed, follow the run batch sequence on the **NU-TURBSHEET** to initiate sample analysis. Press SampleID on turbidimeter digital screen, select the appropriate sample #, 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. When it is time to run batch QC, scroll up to beginning of list and select the appropriate QC.

7.6.10. Note any unsual observations of the sample or errors during the analysis on the digital bench sheet. 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.

7.6.11. 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.

7.6.12. The precision value (relative percent difference or RPD) for the field duplicates must be less than 20 %, provided the sample value is at least 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 duplicates.

7.6.13. Samples with turbidities exceeding 40 NTU need to be diluted and re-analyzed. Open the form **NU-PIPETTE-Turbidity** located on:

\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T\Templates

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

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sample # of the diluted sample manually (see step 7.6.14) as a new sample in the SampleID menu and add the dilution factor. Save the form **YYMMDDXX#_Dilution" (YY-Year, MM-Month, DD-Day, XX(HTor TT)-instrument's name, #-Run number**). The file will be saved with other forms in the run's folder.

7.6.14. Imported sample IDs cannot be altered. Additional samples 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 inthe sequence.

7.6.15. Complete the run by repeating steps 7.6.9 through 7.6.14. for each sample and batch QC.

Note: Samples with results 15 NTU or higher should be immediately reported to the analyst performing Color Analysis.

7.6.16. 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 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.

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

8.1.3. In the USB logger folder, open the correct daily run file. Format the sample numbers in ascending order by pasting them from the digital bench sheet. Edit sample IDs as necessary to denote duplicates and any re-runs. Add the 'Analyst' and 'Reviewer' to separate cells for e-signatures on the pdf versions of this file. Rename the correct daily run file as "**YYMMDDXX#_Reading Log**" and copy the file to the Hach-T folder in the appropriate instrument and year.

\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T

Delete the file from the USB after saving.

8.1.4. In the open digital bench sheet (**NU-TURBSHEET**) on the computer, copy and paste the raw data into the 'NTU' column. Make sure all sample IDs and QC match in both files. Copy and paste the 'Date/Time' column as well. Save this file, ensure the file is properly named as step 7.6.5.

8.1.5. Log into LIMS, choose the appropriate instrument (GenChem: HachT-1 or HachT-2). Convert the excel file from step 8.1.4. into QC Manager. Check that the number of significant figures has been set correctly.

8.2. Calculations:

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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. The raw analysis data, and logger files are archived in the appropriate instrument and year folder located at:
\\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\.Hach-T

An electronic report folder (7.6.2) is created for each analytical run and saved at:

\\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\Hach-T\HACHT-#\Year.

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-TURBCOVER)
- Digital Bench Sheet – NU-TURBSHEET (7.6.5.)
- Reading Log (8.1.3.)
- A sample Backlog (7.6.4.)
- QC Assessment uploaded from QC Manager to LIMS at the time of test authorization
- NU-PIPETTE-Turbidity form, if needed (7.6.13.)

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. Then use Digitally Sign to sign the PDF document in the corresponding spot at the bottom of all pages or in appropriate 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 or a comment must be added afterwards, corrections must be made in the Excel file and then saved as a PDF with the file name format: **YYMMDDXX#_<Form Name>_Corrected.**

9.3. Method validation data is on file with the supervisor. Digital copy is also stored onthe network, under an appropriate sub-folder in

\\fldep1\dear\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

9.4. Once a quarter, the entire reading and calibration log is exported to the Hach-Tfolder in the Nutrients drive as a backup. After saving, the data stored in the turbidimeter can be deleted. Do not delete the most current calibration log.

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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 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%
40 NTU	40	38 ≤ x ≤ 42	95% – 105%
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.), 10 NTU LFB (4.1.6.), 40 NTU (4.1.7.) are analyzed to verify the calibration values and ensure

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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.), 40 NTU standard (4.1.7.), and 1.0 NTU standard (4.1.2.). The Acceptance Limits for all the standards are outlined in the 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 ± five percent NTU of the last calibration verification value, the instrument must be recalibrated.

10.5. The continuing calibration verification (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.

10.6. The 10 NTU LFB standard (4.1.6.) is run at the beginning of each batch of 20 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 greater than 1.0 NTU. Duplicates should be visually representative of the entire batch.

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

11. MAINTENANCE

11.1. The turbidimeter is kept on or in sleep mode when running daily. Completeshutdown is performed before the weekend.

11.2. Prior to performing the quarterly calibration, clean the filter (located in the cellholder compartment) by applying isopropyl alcohol with a cotton-tipped swab.

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 thincoat 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.

11.4. To prevent storage from becoming full, reading logs and previous calibrations that have been electronically transferred will need to be periodically deleted from the

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turbidimeter.

11.5. Maintenance performed prior to the calibration is recorded in the **YYMMDDXX#_Calibration** (7.3.6.). These files are saved as in PDF format at:

\\fldep1\DEAR\Labs\Lab5\Chemistry\Inorganic\Nutrients\Turbidity Final Report in the corresponding Calibration Maintenance folder. **If any maintenance is performed without a new calibration, the same file template is used but thecalibration and verification tables are left empty.**

12. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

12.1. Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/content/dear/bureau-laboratories-health-and-safety-plan).

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 wasteglass container.

12.5. The formazin standards (4.1.1, 4.1.2., 4.1.6, and 4.1.7) should be disposed of as hazardous waste. The sealed standards can be lab packed, labelled as hazardous/formazin, andtaken 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.

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

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

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

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

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

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

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

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12.8. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines.

12.9. Quality Manual, Bureau of Laboratories, Florida DEP, http://www.dep.state.fl.us/labs/library/progplan.htm.

12.10. Laboratory Health and Safety Plan, http://depnet/burlabs/safety.htm.

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 updatedand 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 the 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.

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