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.

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.

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 give 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. Hach Turbidimeter, Model TL2300:

- S/N: 2018010C0108, (Unique ID: HACHT-1)
- S/N: 2018010C0041, (Unique ID: HACHT-2)

3.2. Hach Glass Turbidity Cells (P/N 2084900)

3.3. Assorted Graduated Cylinders (Class A)

3.4. Assorted Pipets (Class A)

3.5. 100 mL Volumetric Flask (Class A)

3.6. Silicone oil

3.7. Oiling cloth

3.8. Isopropyl Alcohol

3.9. USB Flash Drive

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

Note: Before preparing standards or running samples, please 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

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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.

4.1. DI-water – Used for all blanks (MDL, BLK, MBLK, CCB). Poured fresh each use.

4.2. Set of Hach StablCal Primary Standards (TUR- CAL SET; cat. No. 2662105) – Contains <0.1 NTU, 20 NTU, 200 NTU, 1000 NTU, 4000 NTU. Each standard in the Hach StablCal primary standards set is entered separately and a unique ID generated for it in the Standard Preparation Tracker module. The expiration dates are provided by the manufacturer.

4.3. 1.0 NTU Standard – Purchased from Hach Company. Poured fresh each use or if kept sealed and stored in the dark, as needed. The expiration date is provided by the manufacturer, but is recommended to be use within 30 days after opening.

4.4. 10 NTU Standard – Serves as the second source calibration check standard (ALTQC) and as the laboratory fortified blank (LFB). Purchased from GFS Chemicals, FisherSci, or other vendors other than Hach. Poured fresh each use. The expiration date is provided by the manufacturer.

4.5. Gelex Stray Light Secondary Turbidity Standard – Purchased from Hach. Serves to measure the drift of the instrument for maintenance purposes. Stored in the dark.

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

5.1. 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.**

5.2. 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.

5.3. 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.

5.4. 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.

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

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

These settings should be verified prior to each quarterly calibration and analysis runs 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

6.2. If the current quarterly calibration is still valid (less than 3 months old), proceed to Sample Analysis (6.5.). If not, proceed to the Quarterly Calibration of Turbidimeter (6.3.) then the Turbidimeter Quarterly Calibration Verification (6.4.).

Note: Watch every measurement reading to catch error messages, if any. If an error message is observed during calibration, then the calibration needs to be restarted. If an error message is observed during analysis, then the error must be documented, and analysis can be repeated.

6.3. Quarterly Calibration of Turbidimeter:

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

6.3.1. Clean the USEPA filter assembly before calibration (10.2), or at least every 3 months.

6.3.2. Make sure the Turbidimeter has been on for at least one hour before calibrating.

6.3.3. Remove the <0.1 NTU standard from the case containing the set of Hach StablCal Primary standards (4.2). 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.

6.3.4. 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. Appropriately oil and clean the outside of the standard with a Kimwipe. Place the standard in the turbidimeter aligning the arrow marks. Press Read and the machine will automatically take a stable reading.

6.3.5. Repeat for the remaining StablCal standards.

6.3.6. Once the calibration has finished, click View Graph and document the correlation coefficient (R^2).

6.3.7. If the calibration passes (see 9.2.), press Store to save the calibration then proceed immediately to the quarterly calibration verification (6.4.)

6.4. Turbidimeter Quarterly Calibration Verification:

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6.4.1. **The quarterly calibration verification must be performed immediately after a new calibration and before samples are analyzed.**

6.4.2. 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.

6.4.3. Click on the first line that reads BLK then press Select. Pour DI water (4.1.) into a sample cell and cap it. Insert the cell, close the cover, and press Read.

6.4.4. Next measure the 10 NTU ALTQC. Select Sample ID → TURBALTQC → Select. Pour the 10 NTU Standard (4.4.) into a sample cell and cap it. Insert the cell, close the cover, and press Read.

6.4.5. Repeat for the remainder of the calibration verification sequence.

6.4.6. If the quarterly calibration verification also passes (see 9.3., 9.4., and 9.8.), attach a label to the turbidimeter containing the instrument ID, calibration date, calibration expiration (3 months from calibration date), and analyst's initials.

6.4.7. Proceed to the electronic transfer of the quarterly calibration and verification (7.1).

6.5. Daily Analysis:

6.5.1. Prior to each analysis, the calibration of the turbidimeter is verified at the beginning of the run. (see 9.4.)

6.5.2. Make sure that the turbidimeter is turned on and allowed to warm up for at least one hour before analysis is started.

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

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

6.5.4.1. Generate and the backlog in the Excel (.xls) format using LIMS by right clicking the backlog → Save → Excel table (XML) → OK. Save it in the corresponding folder located in the Hach-T folder at: \\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\ using the format **YYMMDDXX#_BackLog**, where YY is the year, MM is the month, DD is the day, HT (for HACHT-1) or TT (for HACHT-2) is the turbidimeter, and # is a number that denotes the 1st, 2nd, etc., runs of the day.

6.5.4.2. In the saved file, batch the samples by highlighting each batch with a distinct color then save the file.

6.5.4.3. Note any equipment blanks or field duplicates on the backlog.

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6.5.4.4. For each batch, prepare:

- One MBLK
- One laboratory fortified blank (LFB)
- A sample to be analyzed in duplicate to calculate precision

6.5.4.5. Insert the USB flash drive into a computer and open the **Sample ID Import** file. Create a run sequence by updating the #SampleID column. The #Reading column must be in ascending order without any repeated or skipped numbers.

Note: **Samples or QC with the same name, date, or time will be considered as duplicates by the turbidimeter and not transferred. Thus, repeating ID's must be accompanied by a number that denotes their count in the run.**

An example run sequence includes:

BLK1 (First batch's **MBLK**, 4.1.)
<0.1 NTU (4.2.)
20 NTU1 (4.2.)
200 NTU (4.2.)
1.0NTU (4.3.)
10 NTU1 (**LFB**, 4.4.)
First 10 batched samples (of the 1st batch) with a **sample duplicate** (6.5.4.4.)
BLK2
20 NTU2
Next 10 batched samples (of the 1st batch)
BLK3
20 NTU3

6.5.5. Save the updated **Sample ID Import** file and insert it into the turbidimeter. To import the sequence, press Sample ID → Options → Import Sample ID List → OK.

6.5.6. To analyze, press Sample ID. Click on the first standard in the sequence then press Select. Insert the cell into the meter while making sure to align the arrows. Close the cover then press Read. Wait for the meter to automatically read and store the data.

6.5.7. If necessary, add a comment to the backlog file for any sample observations or errors. This can also be done manually on the main screen of the turbidimeter.

6.5.8. Repeat for the rest of the sequence.

6.5.9. Since imported sample ID's cannot be altered, samples or additional QC can be manually added to the turbidimeter. 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.

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6.5.10. For samples with turbidities exceeding 40 NTU, dilute the sample with DI water. Add the sample ID of the diluted sample manually as a new sample in the SampleID menu and add the dilution factor. (6.5.9.)

Note: For samples with turbidities exceeding 15 NTU inform the analyst performing color analysis immediately.

6.5.11. Once all analysis is done, proceed to the Electronic Transfer of Analysis Raw Data (7.2).

7. POST RUN PROCEDURE

7.1. Electronic Transfer of Quarterly Calibration and Verification:

7.1.1. 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.

7.1.2. 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 → OK (x3). Then hit Options → Send Data → Filtered Data → OK.

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

7.1.4. In the USB logger folder, rename the calibration file to **YYMMDDXX# Calibration logger**. Then rename the calibration verification file to **YYMMDDXX# Verification logger**.

7.1.5. Save these two files to the Hach-T folder in the appropriate year at: \\fldep1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\. Delete the files from the USB drive after saving.

7.1.6. Then go to the Hach-T folder and open the calibration log sheet template file (**NU-TURB-CAL-1.1)**. Open the two logger files and drag them both into the template file as separate sheets.

7.1.7. Manually update the corresponding cells with the calibration data from **YYMMDDXX# Calibration logger**. Use the documented correlation coefficient to update the R^2 cell. (6.3.6.).

7.1.8. Rename the **YYMMDDXX# Verification logger** sheet as "Sheet 2." This will auto-populate the corresponding cells in Sheet 1.

7.1.9. Record any maintenance performed.

7.1.10. Save the updated calibration log sheet in the Hach-T folder in the appropriate year using the format **YYMMDDXX#_Calibration.**

7.2. Electronic Transfer of Daily Analysis Raw Data:

7.2.1. To transfer the run to the USB flash drive, select Data Log → Reading Log → Filter → On → Time Interval→ Set the date range → OK (x3). Then hit Options → Send Data → Filtered Data → OK.

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

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

7.2.3. In the USB logger folder, rename the correct daily run file to "**YYMMDDXX#_Reading Log**" and copy the file to the Hach-T folder in the appropriate year. **This is the raw data file to be uploaded to LIMS QC Manager.** Delete the file from the USB after saving.

7.3. Final Report Procedure:

7.3.1. In the Hach-T folder, open the Turbidity Final Report template (**NU-HACHTURB-1.1**) and the saved Reading Log (7.2.3.).

7.3.2. Transfer the entire Reading Log into the Turbidity Final Report template. Rename the Reading Log that was transferred into the Turbidity Final Report template into "Sheet 2." This will auto-populate the corresponding cells in Sheet 1.

7.3.3. Fill out the remaining cells on Sheet 1 and check that all the auto-populated cells were transferred correctly.

7.3.4. Use Save As to save the updated final report into the Hach-T folder in the appropriate month using the format **YYMMDDXX#_Turbidity Final Report**.

7.3.5. Dilutions, if used, will have to be manually entered in the **Turbidity Final Report** which will then automatically calculate the reported value with rounding. 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

8. DATA ARCHIVAL

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

8.2. An electronic report folder is created for each analytical run and saved at: \\fldep1\DEAR\Labs\Lab5\Chemistry\Inorganic\Nutrients\Turbidity Final Report. The folder is labelled using **YYMMDDXX#** of the analysis run. Each folder should contain the following **in both Excel and PDF format**:

- Calibration (7.1.10.)
- Turbidity Final Report (7.3.4.)
- Reading Log (7.2.3.)
- Backlog with batching and comments for all samples analyzed (6.5.4.1.)
- QC Assessment uploaded from QC Manager to LIMS at the time of test authorization (PDF only)

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8.3. Once all data in the Excel file formats are updated and verified for correctness, use Save As to save a pdf copy of the report. Select Tools → Certificates. Then use Digitally Sign to sign the PDF document in the corresponding spot at the bottom of all pages that contain data.

Note: When first signing the PDF, the changes will be saved by overwriting the existing Final Report 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 of the Turbidity Final Report and then saved as a PDF with the file name format: **YYMMDDXX#_Turbidity Final Report_Corrected.**

8.4. Method validation data is on file with the supervisor. Digitcal copy is also stored on the network, under an appropriate sub-folder in \\fldep1\dear\Labs\Lab5\Chemistry\Inorganic\Nutrient\Validations.

8.5. Once a quarter, the entire reading and calibration log is exported to the Hach-T folder 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.

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

9.1. Blank- DI water (4.1) and the <0.1 NTU standard (4.2) are analyzed on every run. The results should be **LESS THAN** 0.10 NTU.

9.2. Quarterly calibration – The correlation coefficient of the calibration range must be equal to or larger than 0.995. Acceptance limits for the recovery of the calibration standards 20-4000 NTU are 90 - 110%.

9.3. One certified standard reference material, ALTQC (4.4), is analyzed with every quarterly calibration for verification. Acceptance limits for the recovery of the ALTQC are 90 – 110%.

9.4. Prior to each analysis, the calibration of the turbidimeter is verified at the beginning of the run with a blank, <0.1 NTU standard, 20 NTU standard, 200 NTU standard, and 1.0 NTU standard (4.3). The acceptance limits for the recovery of the 20 NTU and 200 NTU standard are 95 - 105%. The acceptance limits for the recovery of the 1.0 NTU standard is 90 – 110%.

9.5. The continuing calibration verification, BLK and 20 NTU, is analyzed at the beginning, after every 10 samples, and at the end of each batch.

9.6. The 10 NTU standard (4.4.) 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%.

9.7. 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.

9.8. The measured value for the Gelex Stray Light standard should be within ± 0.02 NTU of the initially recorded value. Initially recorded values are in the

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maintenance book. 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.

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

10. MAINTENANCE

10.1. The turbidimeter is kept on or in sleep mode when running daily. Complete shutdown is performed before the weekend.

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

10.3. 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. Do this at least every two weeks.

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

10.5. Maintenance performed prior to the calibration is recorded in the **YYMMDDXX#_Calibration** (7.1.9.). 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.

10.6. **If any maintenance is performed without a new calibration, the same file template is used but the calibration and verification tables are left empty.**

11. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

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

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

11.3. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples and reagents.

11.4. Dispose of excess samples by flushing them down the sink with plenty of water. The 10 NTU (4.2.) 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.

11.5. The formazin standards (4.2) 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 standard (4.3.) 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.

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12. 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 and Quality Control.

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

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

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.

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