DETERMINATION OF TURBIDITY IN WATER USING THE HACH 2100AN TURBIDIMETER

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. Samples are shipped and stored at $\leq 6\ ^{o}C$ (not frozen) to minimize the microbiological decomposition of solids. Samples must be analyzed within 48 hours of sampling.

2.4. 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 2100AN (Turbidimeter 1)

3.2. Hach Glass Turbidity Cells

3.3. Assorted Graduated Cylinders (Class A)

3.4. Assorted Pipets

3.5. 100 mL Volumetric Flask (Class A)

3.6. Silicone oil

3.7. Oiling cloth

3.8 Timer

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.

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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 Turbidity folder within the REAGENT_PREP folder on the Nutrients drive. All containers holding standards must be labeled with their unique ID and expiration date.

4.1. Set of Hach StablCal Primary Standards (TUR- CAL SET; cat. No. 26595-05). The expiration date is provided by the manufacturer. The unique ID generated in the Standard Preparation Tracker module applies to the entire set of these standards.

4.2. Turbidity Second Source Calibration Check Standard (TUR-ALTQC-STD) purchased from AccuStandard Inc, Ricca Chemical Company, or other vendors. The expiration date is provided by the manufacturer.

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

5.1. Quarterly Calibration of Turbidimeter (manufacturers' instructions):

5.1.1. Before using the StablCal Turbidity standards (<0.1 NTU, 20 NTU, 200 NTU, 1000 NTU, 4000 NTU, 7500 NTU) invert the case containing the standards 20 times. Always remove the < 0.1 NTU standard from the case before inverting the rest of the set due to the interference caused by finely divided air bubbles.

5.1.2. Make sure the Turbidimeter has been on for at least one hour. Press the **Cal/Zero** key. Pour deionized (DI) water into a clean sample cell. Wipe the cell clean and apply a thin film of silicone oil to its surface using the oiling cloth. Place it in the cell holder, close the cover and press the **enter** key. The instrument will count down from 60 to 0, and then take a measurement. The instrument will automatically increment to the next standard and the display will read 20 NTU.

5.1.3. Repeat section 5.1.2. using the 20 NTU standard and each subsequent standard.

5.1.4. After the last standard (7500 NTU) has been measured, press the **Cal/Zero** key. The instrument will revert to measurement mode.

5.1.5. To print the calibration details, press the **Cal/Zero** key and then the **Print** key.

5.1.6. Prior to analysis, prepare the 35 NTU Second Source Calibration Check Standard (TUR-ALTQC 35 NTU). This can be prepared making an appropriate dilution of the Turbidity Second Source Calibration Check Standard (4.2). The hold time for this standard is 1 day.

5.1.7. Analyze the 35 NTU Second Source Calibration Check standard and print the result with the calibration data. Acceptable limits for the recovery of

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the check standard are 90 – 110 %. If the recovery is outside this range, the instrument should be recalibrated.

5.1.8. Analyze the DI water and all six calibration standards. Print the results with the calibration data. Acceptable limits for the recoveries of the standards are 90 - 110 % except for the DI water and the < 0 .1 NTU standard which should be < 0.1 NTU. If any recovery is outside this range, the instrument should be recalibrated. If the recovery is acceptable, press the **print** key.

5.1.9. Initial the printout and attach to a page in the turbidimeter maintenance logbook. The printout contains the calibration readings and the results for the calibration standards and ALTQC.

5.1.10. Attach a label to the turbidimeter containing the date of calibration and date the next calibration is due (3 months from the date of previous calibration).

5.2. Calibration Verification of Turbidimeter:

Note: Prior to each analysis, the calibration of the turbidimeter is verified using three of the StablCal Primary Standards (4.1.). The meter is recalibrated at least every three months (see section 5.1.) or if any of the calibration verification checks below consistently fail. All standards should be carefully wiped with a kimwipe or the provided oiling cloth before use.

5.2.1. Once the turbidimeter is turned on, allow the instrument to warm up for at least 20 minutes. During this time, the calibration data should be printed. Press the **Cal/Zero** key, and then press the **Print** key to print all the calibration data in effect. Press the **Units/Exit** key to return to the operating mode.

5.2.2. The calibration check should begin with a blank. Pour DI water into a clean sample cell and place it in the meter. Close the cover and allow the reading on the instrument to stabilize for one minute. Press the **Print** key and record the result on the turbidity worksheet. Verify that the reading is less than 0.10 NTU. Perform the same step with the < 0.1 NTU standard.

5.2.3. Perform step 5.2.2. for the standard with a value of 20 NTU. Verify that the result is within 5 % of the certified value.

5.2.4. Perform step 5.2.2. for the standard with a value of 200 NTU. Verify that the result is within 5 % of the certified value.

5.2.5. If any of these initial results are outside 5 % of the certified values, make sure the standards are clean and are not scratched. If the sample cell is in good condition, the meter may need to be recalibrated (see section 5.1.).

5.3. Sample Analysis:

5.3.1. Record the instrument and its unique ID, analyst (primary and if applicable, secondary), date, start time/end time, file name, calibration set serial number, calibration date, and expiration date, and the second source serial number, expiration date, and % recovery (see 5.1.7.) on the turbidity cover sheet NU-TURBCOVER (see Appendix A) located at

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\\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\Wet_chem\templates\. Also print a blank copy of the Excel worksheet NU-TURBIDITY (see Appendix B) to record sample analysis results.

5.3.2. Allow samples to reach room temperature before analysis. Condensation on the cells will affect the results.

5.3.3. Shake the sample well to thoroughly disperse any solids. Wait until any air bubbles disappear, rinse the cell with sample, discard the rinse solution, and refill the cell with sample. The sample level must be above the line on the cell. Cap the sample cell, and wipe to remove water spots and fingerprints. Scratches or other defects can cause false readings; see section 8.2 for cleaning and oiling instructions if necessary. Place the sample cell in the instrument cell compartment, aligning the arrow marks. Close the sample compartment lid. Set a timer to one minute. Adjust the sample number on the turbidimeter using the up and down arrow keys. Note the sample number in the # field on the turbidity worksheet. Observe the readings on the meter. They should stabilize. After the minute has elapsed and the readings are stable press the **Print** key. Read the turbidity on the printout and record the value on the worksheet (5.3.1.). Note any sample comments on the worksheet as well (light yellow color, particulate matter present, field duplicates visibly different, etc.).

5.3.4. For samples with turbidities exceeding 40 NTU, carefully and quantitatively dilute the sample with DI water. Use a clean graduated cylinder and do the minimal convenient dilution needed to bring the sample turbidity below 40 NTU. Record the dilution factor on the turbidity worksheet.

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

5.3.5. Note any equipment blanks or field duplicates on the backlog. The backlog fields Matrix, Field ID, and Sampling Location are useful for identifying these sample types.

5.3.5.1. Equipment 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.

5.3.5.2. 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. 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 any visible differences between the field duplicates.

5.3.6. Transfer the turbidity results to the Excel Turbidity upload template, located at \\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\Wet_chem\templates\.

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Save the Turbidity upload template as an Excel file and as a text only file (.txt) in the current years' subfolder under the \Wet_Chem\ folder on the Nutrients drive (see section 6.4., below).

Then use the GenChem: Turbidity driver for the correct turbidimeter to convert the text file into QC Manager. Check that the number of significant figures has been set correctly.

5.4. Calculations:

5.4.1. The instrument will give the analysis results for Turbidity in NTU.

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

5.4.3. Report results following the requirements of EPA method 180.1. See table below:

NTU	Record to Nearest
0.10 - 1.0	0.05
1 – 10	0.1
10 – 40	1
40 – 100	5
100 - 400	10
400 - 1000	50
>1000 - 9999	100

5.4.4. The QC Manager calculates RPDs for duplicate samples. For general calculations including RPDs, see the FDEP Quality Manual (Section 5.9).

6. DATA ARCHIVAL

6.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test (Turbidity), filename/s (YYMMDDT# (Turbidimeter 2), or YYMMDDU# (Turbidimeter A), where the # indicates the 1st, 2nd, etc., run of the day), and initials of the analyst. The quarterly calibration data is recorded and archived in the instrument's maintenance manual.

6.2. Each report folder should contain the following items – instrument printout with calibration data and sample raw results, the Turbidity worksheet, a sample backlog, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

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6.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

6.4. Turbidity instrument data excel transfer files for upload to QC manager are stored in electronic format in the current year's subfolder located in: \\FLDEP1\DEAR\LABS\Lab3\Chem_Data\Inorganic\Nutrients\Wet_chem\.

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

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

7.1. Sample Duplicates - Duplicates should be run for every batch of 10 samples or less. The RPD for the duplicates should be less than 20 %, provided the duplicate values are at least 1.0 NTU. If the duplicates are below 1.0 NTU, all reported results in the batch must be accompanied by an appropriate comment for lack of a precision value.

7.2. Blank - DI water and the < 0.1 NTU standard should be run at the beginning of each run. DI water is also run at the beginning and end of each batch of 10 samples. The blank results should be **LESS THAN** 0.10 NTU.

7.3. Calibration Check - The 20 NTU standard should be run every 10 samples as well as at the end of the run to verify that the turbidimeter is operating properly. The value measured should agree within ± 5 % of the accepted value.

7.4. If either the blank or the calibration check fails, rerun the affected samples or consult a supervisor.

7.5. 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 SOP NU-038 and DEP SOP NU-043.

7.6. Annual MDL and PQL verification is required, (see NU-043, 2.3.7). Method blank (MBLK) data is collected and utilized for the ongoing MDL and PQL verification. Acceptance limits for the MBLKs are < 0.1 μmhos/cm. Perform annual verifications using statistics tracker.

8. MAINTENANCE

8.1. All maintenance is recorded in the instrument maintenance manual.

8.2. If the sample cells are scratched, false readings can be observed. Check cells before use for any visible blemishes. Discard any that appear scratched and give readings above 0.1 NTU when filled with DI water. Take care in cleaning cells, rinse with DI water and wipe down with kimwipes. If QC blanks (laboratory

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blanks, equipment blanks, etc.) give readings above 0.1 NTU, re-pour and reanalyze in a different cell as this could be due to a scratched cell. 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 the excess so that only a thin coat of oil is left. Do this at least every two weeks.

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

9. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

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

9.2. Dispose of excess samples by flushing them down the sink with plenty of water. The formazin standards (See 4.1.) should be labeled as Hazardous and placed in the waste room for disposal once expired.

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

10. 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. Method validation data is on file with the supervisor.

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

10.2. Turbidimeter Manual for the Hach Model 2100AN.

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

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

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

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

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

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

10.9. Quality Manual, Bureau of Laboratories, Florida DEP.

10.10. Laboratory Health and Safety Plan.

Appendix of Significant Changes

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03/24/2008	Reporting limit changed from 0.05 NTU to 0.10 NTU
03/10/2009	Added 100 NTU Intermediate Standard and changed concentration of Second Source Standard to 35 NTU.
03/12/2015	File naming convention modified to specify the instrumentation used. Data archival information clarified in section 6. Example worksheet added as Appendix B. More detailed description of sample analysis procedure in section 5.
03/09/2016	Turbidity template information and file paths in sections 5.3.1. and 5.3.6 were updated. Appendix A and B were updated. The document was checked for 508-compliance.
12/10/2019	Certificate of analysis folder file path added to Section 4. In Section 5.3, change in when to inform color analysts of high turbidity samples. Method Validation file path added in Section 6. Ongoing MDL/PQL verification information added to Section 7.

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Appendix A

Example Turbidity cover sheet, NU-TURBCOVER

Effective Date: 10/01/2015
Revision Date: 09/30/2015
Revision Author(s): A. Lomasney

Turbidity Cover Sheet

NU-TURBCOVER-1.0

Turbidity (EPA Method 180.1, rev. 2.0 (1993))
DEP SOP NU-072

Instrument: Hach Turbidimeter, Model 2100AN

Unique ID:

Primary Analyst:

Secondary Analyst:

Date:

File Name:

Start Time:

End Time:

Turbidity Cal. Set S/N:

AltQC S/N:

Exp. Date:

Exp. Date:

Cal. Date:

% Recovery:

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NU-TURBCOVER-1.0

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Appendix B

Example Turbidity worksheet, NU-TURBIDITY

Effective Date: 10/01/2015
Revision Date: 09/30/2015
Revision Author(s): A. Lomasney

TURBIDITY

NU-TURBIDITY-1.0

Analyst: _____ Date: _____ Time: _____

Sample ID	Dilution	NTU	#	Comment
Blank				
<0.1 NTU STD				
_____ NTU STD				
_____ NTU STD				
_____ NTU STD				
***** Samples over 40 NTU must be diluted below 40 NTU *****				
Blank				
20 NTU Std				
Blank				
20 NTU std				

Reporting

NTU Range	1 - 10	10 - 40	40 - 100	100-400	400-1000	>1000
Report to nearest	0.10	1	5	10	50	100

Page _____ of _____

NU-TURBIDITY-1.0

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