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EPA 180.1 – Turbidity

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1 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters in the range of turbidity from 0 to 40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample. The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity.

1.2 Restricted Procedure
This procedure is restricted to use by an analyst experienced in the operation of a Micro 100+ Turbidity meter. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

2 Definitions

2.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

2.2 Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3 Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4 Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary source calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5 Continuing Calibration Check (CCC) or Continuing Calibration Verification (CCV) – A standard used to verify that the response of the instrument has not changed since initial calibration. The CCC is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.6 Calibration Blank (CB), Initial Calibration Verification Blank (ICB), Method

Blank (MBLK) or Continuing Calibration Blank (CCB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes.

2.7 LCS (Laboratory Control Sample) and LCSD (Laboratory Control Sample Duplicate) are prepared by spiking laboratory reagent water, Ottawa sand or air sampling device with the target analyte or compound. They are used to validate the analytical batch with respect to accuracy and precision. In this method, the CCC results are used for LCS/LCSD for Labworks purposes.

3 Interferences

3.1 The presence of floating debris and coarse sediments, which settle out rapidly, will give low readings. Finely divided air bubbles will affect the results in a positive manner.

3.2 The presence of true color, that is the color of water, which is due to, dissolved substances, which absorb light, will cause turbidities to be low, although this effect is generally not significant with finished waters.

4 Safety

4.1 Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Plan for Quality Control Definitions, online revision.

5 Apparatus and Equipment

5.1 Sample Container: half gallon plastic
5.2 Cuvette 28 x 70 mm 10/pk) P/N 50052
5.3 HF Scientific Micro100 Laboratory Turbidity meter
5.4 Lamp module (Infrared) HF Scientific P/N 22423S
5.5 Laboratory Tissues

6 Reagents

6.1 <u>Calibration Standards (Primary Source):</u>

6.1.1 The calibration standards are 0.02, 10.0, and 1000 NTU.

6.1.2 The standards are certified, commercially prepared solutions that are purchased.

6.1.3 The ProCal Primary Calibration Kit, Full Range, 0.02, 10.0, and 1000 NTU is purchased from Fisher, Catalog No. 15-393-101. The HF Scientific P/N is 39957.

6.2 <u>Continuing Calibration Check (CCC)/Laboratory Control Sample (LCS):</u>

6.2.1 ProCal Primary Calibration standard 10 NTU from ProCal Primary Calibration kit.

6.2.2 Same source as the calibration standard.

6.3 <u>Continuing Calibration Blank (CCB):</u>

6.3.1 Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest. (Deionized, HPLC, Milli-Q water or equivalent. Milli-Q water has a resistivity of 18.2 [MΩ.cm] @ 25°C and a TOC of 50 ug/L or less).

6.4 <u>Low Level Initial Calibration Verification Standard (ICV):</u>

6.4.1 ProCal 1.0 NTU Primary Turbidity Standard P/N 53080 or equivalent.

6.4.2 Note: Special order from Fisher Scientific

6.5 <u>Mid-Level Initial Calibration Verification Standard (ICV)</u>:

6.5.1 ProCal 10.0 NTU Primary Turbidity Standard P/N 53000 or equivalent

6.5.2 Note: Special order from Fisher Scientific

6.6 Reagent Water:

6.6.1 Purified water which does not contain any measurable quantities of target analytes or interfering compounds for each compound of interest (Deionized, HPLC, Milli-Q water, or equivalent. Milli-Q water has a resistivity of 18.2[MΩ.cm] @ 25oC and a TOC of 50 ug/L or less).

6.7 RBS Cleaning Solution:

6.7.1 20 ml of Thermo Scientific™ RBS™ 35 brought to volume in a 1000 ml volumetric flask with reagent water.

7 Sample Collection

7.1 Samples are collected in a half-gallon plastic container.

7.2 The samples are stored at 0 - 6° C (not frozen).

7.3 Sample holding time is 48 hours.

7.4 Samples are refrigerated at 0 - 6°C (not frozen) until analysis.

7.5 No chemical preservation is required.

8 Calibration

8.1 Calibration Standards

The calibration standards consist of standards at the following concentrations: 0.02 NTU, 10.0 NTU, and 1000 NTU.

8.2 Calibration Curve

Not applicable.

8.3 Calibration Verification

8.3.1 Initial calibrations are performed at a minimum of once per day, prior to sample analysis. Read back each standard and record results. Recoveries must be 90-110% for each standard.

8.3.2 A Low Level ICV at a concentration of 1.0 NTU must be analyzed once per day and prior to sample analysis. Recovery of the standard must be 90- 110%.

8.3.3 A Mid -Level ICV at a concentration of 10 NTU must be analyzed once per day and prior to sample analysis. Recovery of the standard must be 90- 110%.

8.3.4 A Continuing Calibration Check (CCC) and a Continuing Calibration Blank (CCB) must be analyzed every 10 samples and at the end of the sample run. For this method, there is no difference between a Continuing Calibration Check (CCC) standard and a Laboratory Control Sample (LCS).

8.3.4.1 The CCC must be within 10% of its true value

8.3.4.2 The CCB must be less than the method RL.

9 Quality Control

9.1 The default control limits for EPA 180.1 are 90 - 110% recovery for Turbidity LCS recoveries. The EPD Laboratory applies LCS recovery limits to LCSDs. Note, unless specified by method, the EPD Laboratory does not validate batch quality based on LCSD recoveries.

9.2 By default, the EPD Laboratory sets LCS/LCSD precision limits for this method to be 0 - 15% RPD.

9.3 LCS/LCSD recovery and precision limits are static (i.e. not adjusted by control chart results).

9.4 Batch samples in groups of 17.

9.5 Refer to Table 14.1 for Reporting Limits (RLs), Table 14.2 for Quality Control Acceptance Criteria and Table 14.3 for Quality Control Procedures associated with this method and Standard Operating Procedures for Control Charts and Control Limits.

9.6 The control limits are static and appear in Table 14.2. Control charts must still be performed even though limits are static.

10 Procedure

10.1 Turn on turbidity meter and let it warm up for at least 30 minutes.

10.2 If the screen is not lit up, tap on display and the screen will indicate "error lamp not detected" and be in red then it will change to a green "good".

10.3 <u>Index vials:</u>

10.3.1 To obtain the most accurate results all sample vials should be indexed before use.

10.3.2 The instrument will need to be switched to automatic reading. To change to automatic reading, select the settings ICON⚙ and scroll down to "Auto/manual readings," select auto then ✓ .

10.3.3 To index, pour the 10NTU standard into vial and place in compartment and slowly rotate in 5 degree increments until the lowest value is found.

10.3.4 Mark the front of the vial where the mark on sample compartment is located. The Procal calibration standards come with rubber index rings and are indexed in the same manner.

10.3.5 Once the lowest value is found on the calibration standard, align the arrow with mark on instrument sample compartment.

10.3.6 After indexing vials change back to manual mode.

10.4 <u>Calibration:</u>

10.4.1 Turn on turbidity meter and let it warm up for at least 30 minutes.

10.4.2 If the screen is not lit up, tap on display and the screen will indicate "error lamp not detected" and be in red then it will change to a green "good".

10.4.3 Once the meter has warmed up tap the calibration key ⟲.

10.4.4 Select "procal" and click ✓.

10.4.5 The screen will request the highest standard be selected. Select 1000 for highest calibration.

10.4.6 The boxes will turn green after selecting.

10.4.7 Insert Indexed vial containing 1000 NTU. Tap the box "Calibrate 1000," insert sample, then press "start here."

10.4.8 The timer will start a count down from 60 seconds. Once finished the display will request the 10 NTU be inserted.

10.4.9 Insert the 10NTU standard and tap the box "calibrate insert 10NTU sample then press start here" the timer will count down from 60 seconds.

10.4.10 Once the timer is completed the display will request the 0.02 NTU be inserted.

10.4.11 Insert the 0.02NTU standard and tap the box "calibrate insert 0.02NTU sample then press start here" the timer will count down from 60 seconds.

10.4.12 Once the timer is completed the display will change to, "Calibration Ok." Tap the box.

10.4.13 The display will switch to home mode where samples and standards can be analyzed. Follow the analyzing sample procedure below. Calibration must be verified by reanalyzing the 0.02, 10, and 1000NTU standards. In addition, a low-level 1.0 and mid-level 10 NTU ICV will be analyzed. The recovery for all standards must be within 90-110%. Calibration must be performed daily.

10.5 Sample Analysis:

10.5.1 Remove sample bottles, from cold storage and allow to equilibrate to room temperature prior to sample preparation and/or analysis.

10.5.2 Make sure instrument is plugged in.

10.5.3 Tap display. The message will indicate the lamp is not detected and then change to "good".

10.5.4 If "Sample ID" is not displayed select the home key 🏠.

10.5.5 Mix sample and pour into indexed sample vial. Wipe outside with Kimwipe and insert into sample compartment.

10.5.6 Select the capture Icon ⟳. Once the reading is stable the value will be displayed. Record value on bench sheet.

10.5.7 Calibration standards 0.02,10, 1000NTU should be analyzed per day. The 1.0 low-ICV and mid-level ICV10 are poured fresh daily and analyzed with every batch. The 10NTU calibration standard (CCC) should be analyzed every 10th samples along with a DI water blank (CCB). The recovery for the standards should be 90-110%. The blank must be less than 1 NTU.

10.5.8 When all the samples are completed, the power can be turned off by selecting the power icon ⏻.

10.6 Cleaning Vials:

10.6.1 The vials should be washed daily with DI water and allowed to dry. The vials should be soaked in diluted RBS cleaning solution (Section 6.7) once per week.

10.6.2 Once sample vials are cleaned, do not handle glass portion without gloves.

10.6.3 The calibration vials should be wiped with a lint free rag before use. The sides should never be touched. Handle by lids only.

10.6.4 Discard vials that become scratched.

10.7 Instrument Settings: ⚙

10.7.1 Once settings features are set then the analyst should not have to change.

10.7.2 If settings need to be changed, the default for this laboratory will be as follows: Screen brightness=80, Auto/manual=1, Auto power down= off, chart range & options =manual.

10.7.3 If touch screen calibration is needed or the display becomes less responsive, follow prompt.

10.7.4 Set date/time= (time format= 12, daylight savings= on), Delete samples= NA, data resolution =000.00, offset=off, Device ID= current number 0004, 0005 etc., calibration reminder=on and set to 1, calibration download= NA, Log Download = NA, Firmware update= as needed, reset defaults=NA, about device= as needed.

10.8 Sample Dilutions:

10.8.1 If sample is over 40.0 NTU's, dilute as needed with reagent water and read again. Multiply result by dilution factor.

10.8.2 When entering result in Labworks, multiply reporting limit by dilution factor as well.

10.8.3 Comment on sample. TURB-EPA 180.1 – Reporting limits elevated due to high levels of target analyte.

11 Calculations

11.1 Percent Recovery:

$$\mathbf{LCS\ (CCC), LCSD, ICV\ \%Recovery} = \frac{\mathbf{R_{spike}}}{\mathbf{Expected\ Result}} * \mathbf{100}$$

11.1.1 Where:

R_{spike} = Calculated spike concentration in NTU

11.2 Duplicate or LCS/LCSD Precision:

11.2.1 *Relative Percent Difference (%RPD)*:

$$\%RPD = \frac{\left|R_{sample} - R_{duplicate}\right|}{\left(\frac{R_{sample} + R_{duplicate}}{2}\right)} * 100$$

11.2.2 Where:

%RPD = Relative percent difference
R_{sample} = Result of the sample replicate
$R_{duplicate}$ = Result of the duplicate replicate

11.3 Dilution Factor:

$$\text{Dilution Factor} = \frac{Volume_{final}}{Volume_{initial}}$$

11.3.1 Where:

$Volume_{final}$ = Final volume after dilution
$Volume_{initial}$ = Initial volume of aliquot diluted

11.4 Sample Concentration Calculation:

11.4.1 Sample concentration is read directly from the turbidity meter and multiplied by a dilution factor, if necessary:

$$\text{Turbidity (NTU)} = \text{Turbidity}_{reading} * DF$$

11.4.2 Where:

$Turbidity_{reading}$ = Turbidity (NTU) read directly from the instrument
DF = Dilution Factor

12 Waste Management

12.1 See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures, SOP Reference 13.3.

13 References

13.1 Methods for Chemical Analysis of Water and Wastes, Turbidity, Method 180.1 (Nephelometric). U.S. EPA Office of Research and Development: Cincinnati,

OH, 1983, EPA 600/4-79-020, Revision 2.0 (1993).

13.2 GA EPD Laboratory SOP's – Initial Demonstration of Capability SOP 6-001, current revision, or Continuing Demonstration of Capability SOP 6-002, online revision.

13.3 GA EPD Laboratory SOP - EPD Laboratory Procedures for Control Charts and Control Limits SOP, SOP 6-025, online revision.

13.3 GA EPD Laboratory SOP - EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.4 GA EPD Laboratory – Laboratory Quality Assurance Plan, online revision.

13.5 GA EPD Laboratory SOP - Laboratory Safety/ Chemical Hygiene Plan & Fire Safety Plan, online revision.

13.6 Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005.

14 Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

Table 14.1 RLs for Method EPA 180.1

Parameter/Method	Analyte	Matrix (aqueous)	
		RL	Unit
EPA 180.1	Turbidity	1.0	NTU

Table 14.2 Acceptance Criteria for Method EPA 180.1

Method	Analyte	Accuracy Water (%R)	Precision Water (RPD)
EPA 180.1	Turbidity	90-110	15

Table 14.3 Summary of Calibration and QC Procedures for Method EPA 180.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance criteria	Corrective Action	Flagging Criteria
EPA 180.1	Turbidity	Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four analyses of a QC check sample, a method blank and a blind sample. In addition, the analyst must calibrate the instrument.	Once per analyst	QC Acceptance Criteria Table 14.2 and Initial Demonstration SOP (Reference 13.3)	Recalculate results: locate and correct problem with system and rerun demonstration for those analytes that did not meet criteria	
		Continuing Demonstration: Demonstrate ability to generate acceptable accuracy and precision using a variety of analysis options of a QC sample(s)	Every 6 months	QC Acceptance Criteria Table 14.2 and Continuing Demonstration of Capability SOP (Reference 13.3)		
		Initial Calibration Blank (ICB)	Once per batch	Values must be < RL	Correct problem and repeat initial calibration	
		Low level ICV (1.0 NTU)	Once per day prior to sample analysis	QC Acceptance Criteria Table 14.2	Correct problem and reanalyze	
		Mid- level ICV (10 NTU)	Once per day prior to sample analysis	QC Acceptance Criteria Table 14.2	Correct problem and reanalyze	
		Laboratory Control Sample (LCS/ LCSD/CCC)	LCS/LCSD immediately following calibration, after every 10 samples and at the end of the run	QC Acceptance Criteria Table 14.2	Correct problem and reanalyze the LCS/LCSD/CCC and all samples in the affected batch	If unable to reanalyze, flag with a “J”
		Continuing Calibration Blank (CCB)	After every 10 samples and at the end of the run.	Value must be < RL	Correct problem and reanalyze blank and all samples processed with the contaminated blank.	

Updates to Previous Version:

Updated for online revision.
Section 1
Section 10