DETERMINATION OF TURBIDITY BY 360º NEPHELOMETRY

Hach Method 10258

Revision 2.0
March 2018

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1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of turbidity from colorless, membrane filtered drinking water, conventional filtered drinking water, ground water, surface water, and treated wastewater that has a turbidity of less than or equal to 40 NTU.

1.2 The applicable turbidity measuring limit for this method is 0.0005 NTU. Note that the regulatory minimum reporting limit for turbidity may be higher.

1.3 This method may be used for compliance monitoring under the Safe Drinking Water Act (SDWA) 40 CFR 141, the USEPA Long-term Treatment Rule, LT1/LT2, and California Code of Regulations, Title 22.

2.0 SUMMARY OF METHOD

2.1 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 the scattered light, the higher the turbidity. Measurements in NTU or mNTU are made with a nephelometer designed according to specifications outlined in Section 6 of this method. A primary standard suspension of Formazin or stabilized Formazin (StablCal™) is used to verify the analytical system's on-going accuracy and stability of measurement (Section 9).

3.0 DEFINITIONS

3.1 MATERIAL SAFETY DATA SHEET – Written information provided by vendors concerning a chemical/s toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.2 LINEAR CALIBRATION RANGE - The turbidity range over which the instrument response is linear.

3.3 PRIMARY CALIBRATION STANDARD - A Formazin suspension prepared from a primary dilution stock standard suspension or StablCal, either bulk solution or StablCal prefilled all-glass flame-sealed vials. The suspension solutions are used to calibrate the instrument response with respect to turbidity.

3.4 QUALITY CONTROL SAMPLE – A Formazin suspension prepared from a source that is different from the primary prepared standards. This may be from an alternate preparation of the

stock standard suspension or an alternate lot of a commercial preparation primary standard such as StablCal bulk solution or StablCal prefilled all-glass flame-sealed vials

3.5 SECONDARY CALIBRATION STANDARD– A manufacturer supplied standard for checking instrument performance between required calibrations. An example would be an optical glass rod that is calibrated by the manufacturer, or user, with reference to the turbidity of a primary standard. A secondary standard may only be used for calibration verification and not for initial or ongoing calibration.

3.6 STOCK STANDARD SUSPENSION - Concentrated Formazin solution (40 NTU) that is used to prepare diluted primary calibration and calibration verification standards.

4.0 INTERFERENCES

4.1 Condensation around sample vials or sealed calibration vials, scratches in vials, and light path materials may cause inaccurate readings.

4.2 The presence of true color (e.g., humic substances) will cause turbidity readings to be low, although this effect is generally not significant in finished treated drinking water and treated wastewater final effluent.

4.3 Light-absorbing materials such as activated carbon in significant concentrations may interfere with turbidity readings.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in executing this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

5.2 Each field and laboratory facility is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of MSDS documents should be made available to all personnel involved in the chemical analysis. A formal safety plan should also be made available.

5.3 Hydrazine sulfate is highly toxic and may be fatal if inhaled, swallowed or absorbed through the skin. Formazin can contain residual hydrazine sulfate. Proper protection should be employed.

6.0 EQUIPMENT

6.1 Turbidimeter – Hach TU5

6.1.1 Hach Automatic Cleaning Module (Recommended for water matrices containing high levels of iron or manganese, or in wastewater applications.

6.1.2 The turbidimeter shall be a 360^{o} nephelometer.

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6.1.3 The scattered light produced from the sample shall be collected at an angle of 90° to the incident light and 360º around the sample vial.

6.1.4 The energy and stability of the light source and detector shall be sufficient to meet the performance criteria of the measuring limit in Section 1.2, sensitivity/resolution of data reporting in Section 13.1, and percent relative standard deviation of spike recovery in Section 14.1 of this method.

6.1.5 The spectral bandwidth (full width at half maximum) of the incident radiation from a non-incandescent light source shall be contained in the range of 630 to 690 nm.

6.1.6 The divergence from parallelism of the incident radiation shall be less than ± 1.5° in the measurement volume.

6.1.7 The distance traversed by incident light shall be ≤ 10 cm in the measurement sample.

6.1.8 A light collector shall be centered at 90° to the incident light path not to exceed 30° from its center. The scattered light shall be collected around 360° to the axis of the incident light.

6.1.9 A means of directing the light collected at the light collector to a photodetector responsive to the full spectrum of the incident light.

6.1.10 A readout device indicating the scattered light collected at 90° to the incident light.

6.1.11 The volume of the sample vial to be ≤ 20 mL.

6.2 Vials and Optical Glass Rod - Sample vials, calibration sealed vials, and secondary calibration optical glass rods used with the turbidimeter must be of clear, colorless optical glass.

6.2.1 To avoid scratching of the sample vial and sealed calibration vials during use and storage, the vial's bottom shall be recessed.

6.2.2 Vials and rods are to be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched.

6.2.3 The vials and rods must not be handled at all where the light strikes them, but should be provided with sufficient extra length, or with a protective case, so that they may be handled.

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7.0 REAGENTS AND STANDARDS

7.1 Reagent water for sample dilutions and facility prepared Formazin calibration standards, turbidity-free to less than 0.03 NTU.

7.2 StablCal bulk or StablCal filled flamed-sealed glass-vial.

7.2.1 Commercially manufactured StablCal bulk solution and StablCal filled flamed-sealed glass-vial standards are valid for regulatory compliance use up to their manufacturer's stated expiration date. The expiration dates are printed on the StablCal bulk solution bottle and sealed vial and vary according to their manufacturing date.

7.3 User Prepared Stock Standard Suspension (Formazin only).

7.3.1 Dissolve 1.00 g hydrazine sulfate, $(NH_2)_2.H_2SO_4$, (CAS 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask. **CAUTION** -- carcinogen.

7.3.2 Stock Standard Suspension - Dissolve 10.00 g hexamethylenetetramine (CAS 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask.

7.3.3 Intermediate Suspension - In a 100-mL volumetric flask, add 5.0 mL of each stock solution in 7.3.1 and 7.3.2. Allow to stand 24 hours at 25 ± 3°C, then dilute to the mark with reagent water.

7.3.3.1 Primary Calibration Formazin Standards - Mix and dilute 10.0 mL of the Intermediate Suspension (7.3.3) to 100 mL with reagent water. The turbidity of this suspension is defined as 40 NTU. For other values, mix and dilute portions of this suspension as required.

7.3.3.2 A new intermediate suspension should be prepared each month. Primary calibration standards should be prepared daily by dilution of the stock standard suspension.

8.0 GRAB SAMPLES FOR DILUTION AND ANALYSIS

8.1 Collect each sample in a soft or hard plastic or glass container. Immediately refrigerate or ice the sample to 4ºC and analyze within same day of collection.

8.2 Prolonged time to analysis of grab samples may yield different turbidity results than in-line samples, as turbidity in water is unstable.

9.0 QUALITY CONTROL

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9.1 Each facility using this method for regulated environmental monitoring is required to operate a formal quality assurance/control program. The minimum initial requirements of this program consist of the demonstration of the facility's capability with this method by analyzing an LCR and QCS.

9.1.1 Linear Calibration Range

9.1.1.1 Verify the analytical system's linearity (light source, sample vial, and detector) using either StablCal or Formazin Calibration Standard as described in Section 7. The calibration verification linearity test solution should be in the range of 10 NTU to 0.8 NTU. Lower level calibration standard solutions may be used if the accuracy can be established within 10% of its stated value.

9.1.2 Quality Control Sample

9.1.2.1 When beginning the use of this method, on a quarterly basis or as required complying with data-quality needs, verify the calibration and acceptable instrument performance with the preparation and analysis of a quality control sample as defined in Section 3.4. If the measured turbidities are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

10.0 CALIBRATION AND CALIBRATION VERIFICATION STANDARDIZATION

10.1 Calibration

10.1.1 Using StablCal or Formazin, calibrate the instrument with bulk Formazin suspension, StablCal, or StablCal prefilled all-glass flame-sealed vials following the manufacturer's operating instructions.

10.1.2 Calibration frequency follows the requirements in the USEPA Turbidity Guidance Document and is required at initial instrument startup, on a quarterly basis, and whenever the calibration verification fails.

10.2 Calibration Verification

10.2.1 Primary or secondary standards may be used for calibration verification.

10.2.2 The calibration verification standard should be in the range of 10 NTU to 0.8 NTU. Lower level calibration standards may be used if their accuracy can be independently established within 10% of its stated value.

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10.2.3 The measured value of the standard shall be within 10% of its stated value. If the measured value greater than 10%, repeat the test with a new standard and clean test vial, or a new sealed calibration vial.

10.2.4 If upon following steps in 10.2.2 and 10.23 the calibration verification continues to fail, perform a new calibration as described in Section 10.1.1 and verify calibration as in Section 10.2.1.

10.2.5 Calibration verification test frequency is dependent on the conditions of the sample vial (biofilm, scratches in the wall surface) and the less likely change in light source and detector stability. Calibration verification should be performed at a minimum of once per week.

11.0 TURBIDITY MEASUREMENT PROCEDURE

11.1 Follow the manufacturer's instructions for the proper set up and operation of the instrument.

11.1.1 Turbidities exceeding 40 NTU: Dilute the sample with one of more volumes of distilled water or membrane filtered water until the turbidity falls below 40 NTU. The turbidity of the original sample is then computed from the turbidity of the diluted sample and the dilution factor. For example, if 5 volumes of 0.2 µm filtered distilled water were added to 1 volume of sample, and the diluted sample showed a turbidity of 30 NTU, then the turbidity of the original is calculated to be 180 NTU. Samples requiring dilution shall be analyzed using static flow conditions.

12.0 CALCULATIONS

12.1 No analyst calculations are required for undiluted sample analysis as the processor has a built-in sample calculator.

12.2 Matrix Sample Dilution Calculation – An additional calculation is required for diluted samples.

12.2.1 Dilution Formula:

$T = Ax$ where: T = Final calculated NTU turbidity result

A = NTU result from diluted sample
x = (B + C) / C = Dilution factor

B = volume of dilution water, in mL

C = sample volume taken for dilution, in mL

12.3 Example of Dilution Calculation:

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Measurement from diluted sample (A) – 0.035 NTU

Sample volume to be diluted (C) – 10 mL
Volume of dilution water (B) – 990 mL

Dilution factor (x) = (990 + 10) / 10 = 100

Final Turbidity (T) = 0.035 NT*U* x 100 = 3.500 NTU

13.0 DATA REPORTING

13.1 The recommended rounding of data for regulatory reporting purposes is presented in the following table:

Turbidity Range (NTU)	Record to the Nearest Value (NTU)	Water Treatment Application
0.0 – 0.100	0.0001	Membrane Filtration
0.100 – 1	0.001	Membrane Filtration
0.100 – 1	0.003	Conventional Filtration
1 – 10	0.01	Conventional Filtration
10 – 40	0.02	Conventional Filtration and Treated Wastewater

14.0 METHOD PERFORMANCE

14.1 In a single laboratory using RO membrane filtered and conventional filtered water, the following average percent recovery of turbidity spike and relative standard deviation was determined with Hach Method 10258.

Single Facility Matrix Spike Recovery

Water Type	Number of Measurements at Each Spike Level	Spike Value (NTU)	Average % Recovery of Spike	% RSD of Recovery
Membrane Filtration	10	0.0015	102	3.1
		0.111	103	1.2
		0.555	102	0.4
Conventional Filtration	10	0.0014	102	12
		0.111	94.0	0.3
		0.431	97.5	0.1

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14.2 In the multiple-facility validation study using membrane filtered and conventional filtered finished water, the following average percent recovery and relative standard deviation of turbidity spike was determined with Hach Method 10258.

Multiple-facility Matrix Spike Recovery

Water Type	Spike Value (NTU)	Average % Recovery of Spike	% RSD of Recovery
Membrane Filtration	0.0015	93.3	11
	0.100	106	1.9
	0.500	104	0.7
Conventional Filtration	0.0015 – 0.0025	92.9	5.3
	0.100	104	1.5
	0.500	106	0.6

15.0 POLLUTION CONTROL

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

15.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

16.0 WASTE MANAGEMENT

16.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 15.3.

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17.0 BIBLIOGRAPHY

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