

DETERMINATION OF TURBIDITY BY 360° NEPHELOMETRY

Hach Method 10258

**Revision 1.0
January, 2016**

**Hach Company
5600 Lindbergh Drive
Loveland, Colorado 80538**

DETERMINATION OF TURBIDITY BY 360° NEPHELOMETRY

Revision 1.0

January 2016

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, that has a turbidity of less than or equal to 40,000 *mNTU*.
- 1.2 The applicable turbidity measuring limit for this method is 0.5 *mNTU*. Note that 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 and the USEPA Long-term Treatment Rule, LT1/LT2.

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 *mNTU* are made in a 360° nephelometer designed according to specifications outlined in Section 6 of this method. A primary standard suspension of Formazin or stabilized Formazin 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 (MSDS) – 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 (LCR) - The turbidity range over which the instrument response is linear.
- 3.3 PRIMARY CALIBRATION STANDARD (PCAL) - A Formazin suspension prepared from the primary dilution stock standard suspension or a stabilized Formazin standard such as StablCal. The PCAL suspensions are used to calibrate the instrument response with respect to turbidity.
- 3.4 QUALITY CONTROL SAMPLE (QCS) – A Formazin suspension prepared from a source that is different from the primary standards. This may be from an alternate preparation of the stock standard suspension (SSS) or an alternate lot of a commercial preparation such as StablCal (PCAL).

3.5 SECONDARY CALIBRATION STANDARD (SCAL) – A manufacturer supplied standard for checking instrument performance between required calibration checks with a primary standard. An example would be optical glass rod that is calibrated by the manufacturer or user to the turbidity of a primary standard.

3.6 STOCK STANDARD SUSPENSION (SSS) - Concentrated Formazin solution (40,000 *mNTU*) that is used to prepare diluted primary calibration and calibration verification standards.

4.0 INTERFERENCES

4.1 Condensation around sample vials and light path materials may cause inaccurate readings.

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

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

6.1.1 The turbidimeter shall be a 360° nephelometer.

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

6.1.3 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.

- 6.1.4 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*.
- 6.1.5 The divergence from parallelism of the incident radiation shall be less than $\pm 1.5^\circ$ in the measurement volume.
- 6.1.6 The distance traversed by incident light shall be ≤ 10 *cm* in the measurement sample.
- 6.1.7 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.
- 6.1.8 A means of directing the light collected at the Light Collector to a photodetector responsive to the full spectrum of the incident light.
- 6.1.9 A readout device indicating the scattered light collected at 90° to the incident light.
- 6.1.10 The volume of the sample vial to be ≤ 20 *mL*.
- 6.2 Sample Vial and Optical Glass Rod - The sample vial and optical glass rod to be used with the turbidimeter must be of clear, colorless optical glass.
 - 6.2.1 Vials and rods are to be kept scrupulously clean, both inside and out, and discarded when they become scratched or etched.
 - 6.2.2 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.

7.0 REAGENTS AND STANDARDS

- 7.1 Reagent water for sample dilutions and facility prepared Formazin calibration standards, turbidity-free to less than 30 *mNTU*.
- 7.2 StablCal Primary Calibration Standard Suspensions or equivalent.
- 7.3 Hach Optical Glass Rod or equivalent.
- 7.4 User Prepared Stock Standard Suspension (Formazin only).
 - 7.4.1 Dissolve 1.00 g hydrazine sulfate, $(\text{NH}_2)_2\text{H}_2\text{SO}_4$, (CASRN 10034-93-2) in reagent water and dilute to 100 *mL* in a volumetric flask. **CAUTION** -- carcinogen.

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

7.4.3 Intermediate Suspension - In a 100 mL volumetric flask, add 5.0 mL of each stock solution in 10.5.1 and 10.5.2. Allow to stand 24 hours at $25 \pm 3^\circ\text{C}$, then dilute to the mark with reagent water.

7.4.3.1 Primary Calibration Formazin Standards - Mix and dilute 10.0 mL of the Intermediate Suspension (10.5.3) to 100 mL with reagent water. The turbidity of this suspension is defined as 40,000 mNTU. For other values, mix and dilute portions of this suspension as required.

7.4.3.2 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 4°C 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

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 (LCR)

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 10,000 mNTU to 800 mNTU. 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 (QCS)

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 standards and acceptable instrument performance with the preparation and analysis of a QCS as

defined in Section 3.4. If the determined concentrations are not within $\pm 10\%$ 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 (PCAL), calibrate the instrument following the manufacturer's operating instructions.

10.2 Calibration Verification

10.2.1 The calibration verification standard (PCAL or SCAL) should be in the range of 10,000 *mNTU* to 800 *mNTU*. Lower level calibration standards may be used if their accuracy can be independently established within 10% of its stated value.

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

10.2.3 If upon following step in 10.2.2 the calibration verification continues to fail, perform a new calibration as described in Section 10.1.1 and verify calibration as in Section 10.2.2.

11.0 TURBIDITY MEASUREMENT PROCEDURE

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

11.1.1 Turbidities exceeding 40,000 *mNTU*: Dilute the sample with one or more volumes of distilled water or membrane filtered water until the turbidity falls below 40,000 *mNTU*. 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 30,000 *mNTU*, then the turbidity of the original is calculated to be 180,000 *mNTU*. Samples requiring dilution shall be analyzed using static flow conditions.

11.2 Calibration Verification and Calibration Frequency

11.2.1 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.

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 *mNTU* turbidity result

$A =$ *mNTU* 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:

Measurement from diluted sample (A) – 35 *mNTU*

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) = 35 *mNTU* x 100 = 3,500 *mNTU*

13.0 DATA REPORTING

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

Turbidity Range (<i>mNTU</i>)	Record to the Nearest Value (<i>mNTU</i>)	Water Treatment Application
0.0 – 100	0. 1	Membrane Filtration
100 – 1000	1	Membrane Filtration
100 – 1000	3	Conventional Filtration
1000 – 10000	10	Conventional Filtration
10000 – 40000	20	Conventional Filtration

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 (mNTU)	Average % Recovery of Spike	% RSD of Recovery
Membrane Filtration	10	1.5	102	3.1
		111	103	1.2
		555	102	0.4
Conventional Filtration	10	1.4	102	12
		111	94.0	0.3
		431	97.5	0.1

- 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 (mNTU)	Average % Recovery of Spike	% RSD of Recovery
Membrane Filtration	1.5	93.3	11
	100	106	1.9
	500	104	0.7
Conventional Filtration	1.5 – 2.5	92.9	5.3
	100	104	1.5
	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.

17.0 BIBLIOGRAPHY

1. Annual Book of ASTM Standards, Volume 11.01 Water (1), Standard D1889-88A, p. 359, (1993).
2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, pp. 2-9, Method 2130B, (1992).
3. Standard Methods for the Certification of Laboratories Analyzing Drinking Water: Criteria and Procedures, Quality Assurance, EPA/570/9-90/008, April, 1990.
4. Hach Method 10133, Determination of Turbidity by Laser Nephelometry, Hach Company, 5600 Lindbergh Drive, Loveland, Colorado 80538.
5. Guidance Manual for Compliance with the Interim Enhanced Surface Water Treatment Rule: Turbidity Provisions, United States Environmental Protection Agency, Office of Water (4607), EPA 815-R-99-010, April 1999.
6. Final Long Term 1 Enhanced Surface Water Treatment Rule, United States Environmental Protection Agency, EPA 815-F-02-001, January 2002.
7. Long Term 2 Enhanced Surface Water Treatment Rule, United States Environmental Protection Agency, January 5, 2006.