

Designation: D 6698 - 01

Standard Test Method for On-Line Measurement of Turbidity Below 5 NTU in Water¹

This standard is issued under the fixed designation D 6698; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is applicable to the on-line measurement of turbidity under 5 nephelometric turbidity units (NTU) in water.

1.2 It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: ²

- D 1129 Terminology Relating to Water
- D 1193 Specification for Reagent Water
- D 1889 Test Method for Turbidity in Water
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water
- D 3370 Practices for Sampling Water from Closed Conduits
- D 3864 Guide for Continual On-line Monitoring for Water Analysis

2.2

- EPA 180.1 Methods for Chemical Analysis of Water and Wastes, Turbidity
- Standard Methods for the Examination of Water and Wastewater 2130B, 19th edition
- ISO 7027 (The International Organization for Standardization) for the Determination of Turbidity

Hach Method 8195

GLI Method 2

- US Patent 4,283,143 from Advanced Polymer Systems, Inc., 123 Saginaw Dr., Redwood City, CA 94063
- US Patent 4,291,980 from Advanced Polymer Systems, Inc., 123 Saginaw Dr., Redwood City, CA
- US Patent 5,777,011 from Hach Company, 5600 Lindbergh Drive, Loveland, CO 80537

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method refer to Terminology D 1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration turbidity standard*—a turbidity standard that is traceable and equivalent to the reference turbidity standard to within statistical errors, including commercially prepared 4000 NTU Formazin, stabilized formazin, and styrenedivinylbenzene (SDVB). These standards may be used to calibrate the instrument.

NOTE 1-Calibration standards may be instrument specific.

3.2.2 *calibration verification standards*—defined standards used to verify the accuracy of a calibration in the measurement range of interest. These standards may not be used to perform calibrations, only calibration verifications. Included standards are opto-mechanical light scatter devices, gel-like standards, or any other type of stable liquid standard.

NOTE 2-Calibration verification standards may be instrument specific.

3.2.3 *in-situ nephelometer*—a turbidimeter that determines the turbidity of a sample using a sensor that is placed directly in the sample. This turbidimeter does not require transport of the sample to or from the sensor.

3.2.4 *nephelometric turbidity measurement*—the measurement of light scatter from a sample in a direction that is at 90° with respect to the centerline of the incident light path. Units are NTU (Nephelometric Turbidity Units); when ISO 7027 technology is employed, units are FNU (Formazin Nephelometric Units).

3.2.5 *ratio turbidity measurement*—the measurement derived through the use of a nephelometric detector that serves as the primary detector and one or more other detectors used to

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

Current edition approved July 10, 2001. Published November 2001.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

compensate for variation in incident light fluctuation, stray light, instrument noise, or sample color.

3.2.6 *reference turbidity standard*—a standard that is synthesized reproducibly from traceable raw materials by the user. All other standards are traced back to this standard. The reference standard for turbidity is formazin.

3.2.7 *seasoning*—the process of conditioning labware with the standard that will be diluted to a lower value. The process reduces contamination and dilution errors. See Appendix X2 for suggested procedure.

3.2.8 *slip stream nephelometer*—an on-line turbidimeter that determines the turbidity of a sample as the sample flows through a sampling chamber. The sample is drawn from the source into the turbidimeter, analyzed and then transported to drain.

3.2.9 *stray light*—all light reaching the detector other than that contributed by the sample. For example: ambient light leakage and divergent light in optical systems.

3.2.10 *turbidimeter*—an instrument that measures light scatter using a nephelometric detector. Examples include photoelectric nephelometers and ratio photoelectric nephelometers.

3.2.11 *turbidity*—an expression of the optical properties of a sample that causes light rays to be scattered and absorbed rather than transmitted in straight lines through the sample. (Turbidity of water is caused by the presence of matter such as clay, silt, finely divided organic matter, plankton, other microscopic organisms, organic acids, and dyes.)

4. Significance and Use

4.1 Turbidity is undesirable in drinking water, plant effluent waters, water for food and beverage processing, and for a large number of other water-dependent manufacturing processes. Removal of suspended matter is accomplished by coagulation, settling, and filtration. Measurement of turbidity provides a rapid means of process control to determine when, how, and to what extent the water must be treated to meet specifications.

4.2 This test method is suitable for the on-line monitoring of turbidity such as that found in drinking water, process water, and high purity industrial waters.

4.3 The instrumentation used must allow for the continuous on-line monitoring of a sample stream.

Note 3—See 7.2 for discussion on signal spikes resulting from bubbles.

5. Safety

5.1 Wear appropriate personal protection equipment at all times.

5.2 Follow all relevant safety guidelines.

5.3 Refer to instrument manuals for safety guidelines when installing, calibrating, measuring or performing maintenance with any of the respective instrumentation.

5.4 Refer to all Material Safety Data Sheets (MSDSs) prior to preparing or using standards and before calibrating or performing instrument maintenance.

6. Interferences

6.1 Bubbles, color, and large suspended particles may result in interferences. Bubbles cause positive interference and color

causes negative interference. Dissolved material that imparts a color to the water may cause errors in pure photoelectric nephelometric readings (versus ratio photoelectric nephelometric readings) unless the instrument has special compensating features. Certain turbulent motions also create unstable reading conditions of nephelometers.

6.2 Scratches, finger marks, or dirt on any part of an optical component through which light must travel to reach the sample, or through which scattered light leaves the sample to a detector, may give erroneous readings. Keep these surfaces scrupulously clean and replace damaged (etched or scratched) components.

7. Apparatus

7.1 The sensor used for the on-line monitoring of turbidity is designed for continuous monitoring of the turbidity of the sample stream.

7.2 The instrument design should eliminate signal spikes resulting from bubbles present in samples through the use of either internal or external bubble rejection chambers (traps), sample pressurization, and/or electronic rejection methods.

7.3 The sensor must be designed to be calibrated. The calibration should be performed by following the manufacturer's recommended procedures. If a calibration algorithm for the instrument is used, it should be derived through the use of a reference or calibration turbidity standard.

7.4 The resolution of the instrument should permit detection of turbidity differences of 0.01 NTU or less in waters having turbidities of less than 1.00 NTU. The instrument should permit detection of turbidity differences of 0.10 NTU or less in waters with turbidity between 1.0 and 5.0 NTU.

7.5 *Instrument Types*—Two types of instruments are available for the nephelometric turbidity method, the nephelometer and ratio nephelometer.

7.5.1 The Photoelectric Nephelometer—(see Fig. 1). This instrument uses a light source for illuminating the sample and a single photo-detector with a readout device to indicate the intensity of light scattered at 90° to the centerline of the path of the incident light. The photoelectric nephelometer should be so designed that minimal stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period. The light source should be a Tungsten lamp operated at a color temperature between 2200 and 3000 K. Light Emitting Diodes (LEDs) and laser diodes in defined wavelengths ranging from 400-900 nm may also be used. If LEDs or laser diodes are used, then the LED or laser diode should be coupled with a monitor detection device to achieve a consistent output. The total distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. Angle of light acceptance to the detector: centered at 90° to the centerline of the incident light path and not to exceed $\pm 10^{\circ}$ from the 90° scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used.

7.5.1.1 Differences in physical design of photoelectric nephelometers will cause slight differences in measured values for turbidity even though the same suspension is used for calibrations. Comparability of measurements made using instruments differing in optical and physical design is not

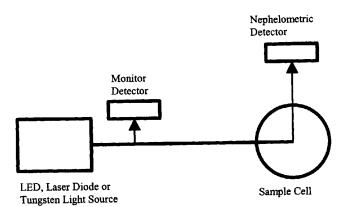


FIG. 1 Photoelectric Nephelometer

recommended. To minimize initial differences, observe the following design criteria:

7.5.2 Ratio Photoelectric Nephelometer-(see Fig. 2 for single beam design; see Fig. 3 for multiple beam design). This instrument uses the measurement derived through the use of a nephelometric detector that serves as the primary detector and one or more other detectors used to compensate for variation in incident light fluctuation, stray light, instrument noise, or sample color. As needed by the design, additional photodetectors may be used to sense the intensity of light scattered at other angles. The signals from these additional photodetectors may be used to compensate for variations in incident light fluctuation, instrument stray light, instrument noise and/or sample color. The ratio photoelectric nephelometer should be so designed that minimal stray light reaches the detector(s), and should be free from significant drift after a short warm-up period. The light source should be a tungsten lamp, operated at a color temperature between 2200 and 3000 K. LEDs and laser diodes in defined wavelengths ranging from 400 to 900 nm may also be used. If an LED or a laser diode is used in the single beam design, then the LED or laser diode should be coupled with a monitor detection device to achieve a consistent output. The distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. The angle of light acceptance to the nephelometric detector(s) should be centered at 90° to the centerline of the incident light path and should not exceed $\pm 10^{\circ}$ from the scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used. The instrument calibration (algorithm) must be designed such that the scaleable reading is from the nephelometric detector(s), and other detectors are used to compensate for instrument variation described in 3.2.5.

7.5.2.1 Differences in physical design of ratio photoelectric nephelometers will cause slight differences in measured values for turbidity even when the same suspension is used for cali-brations. Comparability of measurements made using instruments differing in optical and physical design is not recommended. Examples of ratio nephelometers are shown in Figs. 2 and 3.

8. Summary of Test Method

8.1 The optical property expressed as turbidity is measured by the scattering effect that suspended solids have on light; the higher the intensity of scattered light, the higher the turbidity. In samples containing particulate matter, the manner in which the particulate matter interacts with light transmittance is related to the size, shape and composition of the particles in the water, and also to the wavelength of the incident light.

8.2 The method is based upon a comparison of the intensity of light scattered by the sample with the intensity of light scattered by a reference suspension. Turbidity values are

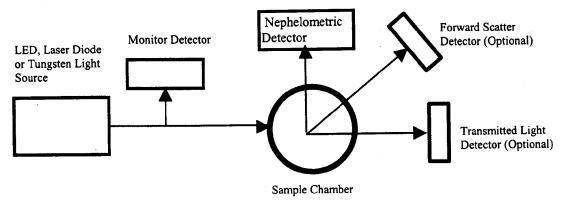


FIG. 2 Single Beam Design

🖽 D 6698 – 01

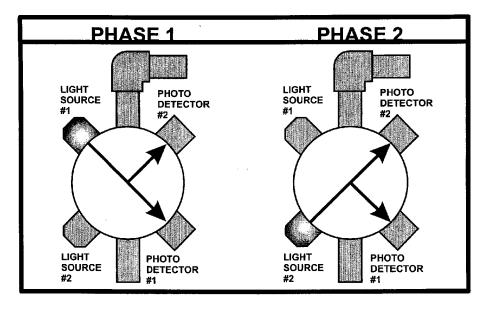


FIG. 3 Multiple Beam Design

determined by a nephelometer, which measures light scatter from a sample in a direction that is at 90 degrees with respect to the centerline of the incident light path.

9. Purity of Reagents

9.1 ACS grade chemicals of high purity (99+ %) shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used providing it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE 4—Refer to product MSDS for possible health exposure concerns.

9.2 Standard dilution, reagent and rinse waters shall be prepared by filtration of Type III water, or better, through a 0.22 microns or smaller membrane or other suitable filter within 1 hour of use to reduce background turbidity. Reverse osmosis (RO) water is acceptable and preferred in this method.

10. Reagents

10.1 Reagent, dilution and final rinsing water, see 9.2. 10.2 *Turbidity Standards*:

NOTE 5—A standard with a turbidity of 1.0 NTU is the lowest formazin turbidity standard that should be produced on the bench. Preparation of formazin standards shall be performed by skilled laboratory personnel with experience in quantitative analysis. Close adherence to the instructions within this section is required in order to accurately prepare low-level turbidity standards.

10.2.1 Equivalent, commercially-available, calibration standards may be used. These standards, such as stabilized formazin and SDVB, have a specified turbidity value and accuracy. Such standards must be referenced (traceable) to formazin. Follow specific manufacturer's calibration procedures. NOTE 6—All volumetric glassware must be scrupulously clean. The necessary level of cleanliness can be achieved by performing all of the following steps: washing glassware with laboratory detergent followed by 3 tap water rinses; then rinse with portions of 1:4 HCl followed by at least 3 tap water rinses; finally, rinse 3 times with rinse water as defined in 9.2. Reference Formazin Turbidity Standard (4000 NTU) is synthesized on the bench.

10.2.1.1 Dissolve 5.000 grams of ACS grade hydrazine sulfate (99.5 % + purity) (N_2H_4 · H_2SO_4 into approximately 400 mL of dilution water (see 9.2) contained in a 1-liter Class A volumetric flask.

10.2.1.2 Dissolve 50.000 grams of ACS grade hexamethylenetetramine (99 %+ purity) in approximately 400 mL of dilution water (see 9.2) contained in another flask. Filter this solution through a 0.2 mm filter.

10.2.1.3 Quantitatively pour the filtered hexamethylenetetramine solution into the flask containing the hydrazine sulfate. Dilute this mixture to 1 liter using dilution water (see 9.2). Stopper and mix for at least 5 minutes, and no more than 10 minutes.

10.2.1.4 Allow the solution to stand for 24 hours at 25 \pm 1°C. The 4000 NTU formazin suspension develops during this time.

10.2.1.5 This suspension, if stored at $20-25^{\circ}$ C in amber polyethylene bottles, is stable for 1 year; it is stable for 1 month if stored in glass at $20-25^{\circ}$ C.

10.2.2 Stabilized formazin turbidity standards are prepared stable suspensions of the formazin polymer. Preparation is limited to inverting the container to re-suspend the formazin polymer. These standards require no dilution and are used as received from the manufacturer.

10.2.3 Styrenedivinylbenzene (SDVB) polymer turbidity standards are prepared stable suspensions which are used as received from manufacturer or distributor. These standards exhibit calibration performance characteristics that are specific to instrument design.