

# Standard Test Method for In-situ Determination of Turbidity Above 1 Turbidity Unit (TU) in Surface Water<sup>1</sup>

This standard is issued under the fixed designation D7937; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 This test method covers the in-situ field measurements of turbidity in surface water. The measurement range is greater than 1 TU and the lesser of 10 000 TU or the maximum measurable TU value specified by the turbidimeter manufacturer.
- 1.1.1 Precision data was conducted on both real world and surrogate turbidity samples up to about 1000 TU. Many of the technologies listed in this test method are capable of measuring above that provided in the precision section (see Section 16).
- 1.2 "In-situ measurement" refers in this test method to applications where the turbidimeter sensor is placed directly in the surface water in the field and does not require transport of a sample to or from the sensor. Surface water refers to springs, lakes, reservoirs, settling ponds, streams and rivers, estuaries, and the ocean.
- 1.3 Many of the turbidity units and instrument designs covered in this test method are numerically equivalent in calibration when a common calibration standard is applied across those designs listed in Table 1. Measurement of a common calibration standard of a defined value will also produce equivalent results across these technologies. This test method prescribes the assignment of a determined turbidity values to the technology used to determine those values. Numerical equivalence to turbidity standards is observed between different technologies but is not expected across a common sample. Improved traceability beyond the scope of this test method may be practiced and would include the listing of the make and model number of the instrument used to determine the turbidity values.

- 1.4 In this test method, calibration standards are often defined in NTU values, but the other assigned turbidity units, such as those in Table 1 are equivalent. For example, a 1 NTU formazin standard is also a 1 FNU, a 1 FAU, a 1 BU, and so forth.
- 1.5 This test method was tested on different natural waters and with standards that served as surrogates for samples. It is recommended to validate the method response for waters of untested matrices.
- 1.6 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:<sup>2</sup>

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3864 Guide for On-Line Monitoring Systems for Water Analysis

D4411 Guide for Sampling Fluvial Sediment in Motion

D7315 Test Method for Determination of Turbidity Above 1 Turbidity Unit (TU) in Static Mode

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

 $<sup>^{\</sup>rm 1}$  This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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<sup>&</sup>lt;sup>2</sup> 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.



# TABLE 1 Summary of Known in-situ Instrument Designs, Applications, Ranges, and Reporting Units

Design and Reporting Unit	Prominent Application	Key Design Features	Typical Instrument Range	Suggested Application Ranges
Nephelometric Non-Ratio (NTU)		Detector centered at 90° relative to the incident light beam. Uses a white light spectral source.	0.0–40	0.0–40 Regulatory
Ratio White Light Turbidimeters (NTRU)	Complies with U.S. EPA regulations and EPA 2130B. Can be used for both low and high level measurement.	source. Primary detector cen-	0–10 000	0–40 Regulatory 0–10 000 other
Nephelometric, Near-IR Turbidimeters, Non- Ratiometric (FNU)	Complies with ISO 7027. The wavelength is less susceptible to color interferences. Applicable for samples with color and good for low level monitoring.	Detector centered at 90° relative to the incident light beam. Uses a near-IR (780-900 nm) monochromatic light source.	0-1 000	0-40 Regulatory (non-US) 0-1 000 other
Nephelometric Near-IR Turbidimeters, Ratio Metric (FNRU)	Complies with ISO 7027. Applicable for samples with high levels of color and for monitoring to high turbidity levels.	Uses a near-IR monochromatic light source (780–900 nm). Primary detector centered at 90°. Other detectors located at other angles. An instrument algorithm uses a combination of detector readings to generate the turbidity reading.	0–10 000	0–40 Regulatory 0–10 000 other
Formazin Back Scatter (FBU)	Not applicable for regulatory purposes. Best applied to high turbidity samples. Backscatter is common probe technology and is best applied in higher turbidity samples.	Uses a near-IR monochromatic light source in the 780–900 nm range. Detector geometry is 30 ± 15° relative to the incident light beam.	100–10 000+	100–10 000
Backscatter Unit (BU)	Not applicable for regulatory purposes. Best applied for samples with high level turbidid- ity.	Uses a white light spectral source ( $400$ – $680$ nm range). Detector geometry is $30 \pm 15^\circ$ relative to the incident light beam.	10–10 000+	100-10 000+
Formazin Attenuation Unit (FAU)	May be applicable for some regulatory purposes. This is commonly applied with spectrophotometers. Best applied for samples with high level turbididity.	is 780–900 nm.	20–1 000	20-1 000 Regulatory
Light Attenuation Unit (AU)	Not applicable for some regulatory purposes. This is commonly applied with spectrophotometers.	Detector is geometrically centered at 180° relative to incident beam (attenuation). Wavelength is 400–680 nm.	20–1 000	20–1 000
Nephelometric Turbidity Multi- beam Unit (FNMU)	Is applicable to EPA regulatory method GLI Method 2. Appli- cable to drinking water and wastewater monitoring applica- tions.	Detectors are geometrically centered at 90° and 180°. An instrument algorithm uses a combination of detector readings, which may differ for turbidities varying magnitude.	0.02–4000	0–40 Regulatory 0–4 000 other
Forward Scatter Ratio Unit (FSRU)	The technology encompasses a single, light source and two detectors. Light sources can vary from single wavelength to polychromatic sources. The detection angle for the forward scatter detector is between 0 and 90° relative to the centerline of the incident light beam.	The technology is sensitive to turbidities as low as 1 TU. The ratio technology helps to compensate for color interference and fouling.	1-800 FSRU The measurement of ambient waters such as streams, lakes, rivers.	Forward Scatter Ratio Unit (FSRU)
Forward Scatter Unit (FSU)	The technology encompasses a single, light source and one detector between 0 and 90° relative to the centerline of the incident light beam.		1-1000 FSU The measurement of ambient waters such as streams, lakes, rivers and process waters.	Forward Scatter Unit (FSU)

2.2 Other Referenced Standards:

EPA 180.1 Determination of Turbidity by Nephelometry<sup>3</sup>
EPA 2130B Analytical Method For Turbidity Measurement<sup>3</sup>
ISO 7027 (International Organization for Standardization)
Water Quality for the Determination of Turbidity<sup>4</sup>
GLI Method 2 Turbidity<sup>3</sup>

## 3. Terminology

- 3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.
- 3.2 Definitions of Terms Specific to This Standard—Unless otherwise noted, the term 'light' means visible light or near-infrared (NIR) radiation or both.
- 3.2.1 *ambient light, n*—light or optical path or both that does not originate from the light source of a turbidimeter.
- 3.2.2 attenuation, n—the amount of incident light that is scattered and absorbed before reaching a detector, which is geometrically centered at 180° relative to the centerline of the incident light beam.
- 3.2.2.1 *Discussion*—Attenuation is inversely proportional to transmitted signal.

Attenuated Turbidity = Absorbed Light + Scattered Light

- 3.2.2.2 *Discussion*—The application of attenuation in this test method is as a distinct means of measuring turbidity. When measured in the FAU or AU mode, the turbidity value is a combination of scattered (attenuated) light plus absorbed light. The scattered light is affected by particle size and is a positive response. The absorption due to color is a negative response. The sum of these two responses results in the turbidity value in the appropriate unit.
- 3.2.3 automatic power control (APC), n—the regulation of light power from a source such that illumination of the sample remains constant with time and temperature.
- 3.2.4 broadband, white-light source, n—a visible-light source that has a full bandwidth at half of the source's maximum intensity (FWHM) located at wavelengths greater than 200 nm.
- 3.2.4.1 *Discussion*—Tungsten-filament lamps (TFLs) and white LEDs are examples of broadband sources.
- 3.2.5 calibration turbidity standard, n—a turbidity standard that is traceable and equivalent to the reference turbidity standard to within defined accuracy; commercially prepared 4000 NTU Formazin, stabilized formazin, and styrenedivinylbenzene (SDVB) are calibration turbidity standards.
- 3.2.5.1 *Discussion*—These standards may be used to calibrate the instrument. All meters should read equivalent values for formazin standards. SDVB-standard readings are instrument specific and should not be used on meters that do not have defined values specified for that instrument. Calibration standards that exceed 10 000 turbidity units are commercially available.
- <sup>3</sup> Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20004, http://www.epa.gov.
- $^4$  Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

- 3.2.6 *calibration-verification standards*, *n*—defined standards used to verify the instrument performance in the measurement range of interest.
- 3.2.6.1 *Discussion*—Calibration-verification standards may not be used to adjust instrument calibration, but only to check that the instrument measurements are in the expected range. Examples of calibration-verification standards are optomechanical light-scatter devices, gel-like standards, or any other type of stable liquid standard. Calibration-verification standards may be instrument-design specific.
- 3.2.7 *color*, *n*—the hue (red, yellow, blue, etc.) of a water sample produced by the combination of: the selective absorption of visible light, the spectral reflectivity, and the degree of darkness or blackness of suspended matter.
- 3.2.7.1 *Discussion*—The combination above is defined by the Munsell  $(1)^5$  color-classification scheme.
- 3.2.8 *detector*, *n*—a solid-state device that converts light into electrical current or voltage.
- 3.2.9 *detector angle, n*—the angle between the axis of the detector acceptance cone and the axis of the source light or NIR beam.
- 3.2.9.1 *Discussion*—The detector angle equals  $180^{\circ} \theta$  ( $\theta$  is the scattering angle).
- 3.2.10 *narrow-band source*, *n*—a light source with a full bandwidth (at half of the source's maximum intensity) (FWHM) located at wavelengths less than 5 nm.
- 3.2.11 *operating spectrum*, *n*—the wavelength-by-wavelength products of source intensity, filter transmittance, and detector sensitivity.
- 3.2.11.1 *Discussion*—The operating spectrum determines the relative contributions of wavelengths in the light-to-current conversions made by a turbidimeter.
- 3.2.12 ratio turbidity measurement, n—the measurement derived through the use of a primary detector and one or more other detectors to compensate for variation in incident-light intensity, stray light, sample color, window transmittance, and dissolved NIR-absorbing matter.
- 3.2.13 *reference turbidity standard*, *n*—a standard that is synthesized reproducibly from traceable raw materials by a skilled analyst.
- 3.2.13.1 *Discussion*—All other standards are traced back to this standard. The reference standard for turbidity is formazin.
- 3.2.14 *sample volume*, *n*—the water-sample volume wherein light from a turbidimeter source interacts with suspended particles and is subsequently detected.
- 3.2.15 scattering (also referred to as scatter), n—light interaction that alters the direction of light transport through a sample without changing the wavelength.
- 3.2.15.1 *Discussion*—The light interaction can be with suspended particles, water molecules, and variations in the sample's refractive index.
- 3.2.16 *scattering angle* ( $\theta$ ), n—the angle between a source light or NIR beam, and the scattered beam.

<sup>&</sup>lt;sup>5</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.



- 3.2.17 forward-scattered radiation, n—the scattered incident light that is detected at an angle between 0-degrees and less than 90-degrees, relative to the direction of the projected incident-light beam.
- 3.2.17.1 *Discussion*—Most designs will have an angle between 0-degrees and 45-degrees.
- 3.2.18 *stray light*, *n*—all light reaching the detector(s) other than light that is scattered by the sample.
- 3.2.18.1 *Discussion*—Stray light could be ambient-light leakage, internal reflections, and divergent light in optical systems. For this test method, stray light is likely to be negligible. The instrument design is intended to reduce or eliminate stray light.
- 3.2.19 *transmittance*, *n*—the ratio of light power transmitted through a sample to the light power incident upon the sample.
- 3.2.20 turbidimeter design, n—an arrangement of optical (lenses, windows, filters, apertures, etc.) and optoelectronic (light sources and detectors, etc.) components, mechanical components, and electrical circuits for determining the turbidity of water.
- 3.2.21 *turbidity*, *n*—an expression of a sample's optical properties that cause light rays to be scattered and absorbed rather than transmitted in straight lines through the sample.
- 3.2.21.1 *Discussion*—Turbidity of water is caused by the presence of suspended and dissolved matter such as clay, silt, finely divided organic matter, plankton, other microscopic organisms, organic acids, and dyes.
  - 3.3 Symbols:
  - A = amperes
  - b =scattering coefficient
  - $\theta$  = scattering angle
  - W = Watts
  - 3.4 Acronyms:
  - 3.4.1 APC, n—automatic power control
  - 3.4.2 AU, n—attenuation unit
  - 3.4.3 BU, n—backscatter unit
  - 3.4.4 FAU, n—formazin attenuation unit
  - 3.4.5 FBU, n—formazin backscatter unit
  - 3.4.6 FNMU, n—nephelometric turbidity multi-beam unit
  - 3.4.7 FNRU, n—formazin nephelometric ratio unit
  - 3.4.8 FNU, n—formazin nephelometric unit
  - 3.4.9 FSU, n—forward scatter unit
  - 3.4.10 FSRU, n—forward scatter ratio unit
- 3.4.11 *FWHM*, *n*—full bandwidth at half of the source's maximum intensity
  - 3.4.12 *IRED*, *n*—infrared-emitting diode
  - 3.4.13 *LED*, *n*—light-emitting diode
  - 3.4.14 NIR, adj—near infrared
  - 3.4.15 NTRU, n—nephelometric
  - 3.4.16 NTU, n—nephelometric turbidity unit
  - 3.4.17 SDVB, n—styrenedivinylbenzene
  - 3.4.18 SSC, n—suspended sediment concentration

- 3.4.19 *TFL*, *n*—tungsten-filament lamp
- 3.4.20 TU, n—turbidity unit

Note 1—See Table 1 for description of all acronyms related to turbidity reporting units.

#### 4. Summary of Test Method

- 4.1 Turbidity is a numerical expression, in relative units, of the optical properties that cause light to be scattered and absorbed rather than transmitted straight through a water sample. It is measured with a turbidimeter, which in simplest form has a light source to illuminate the water sample and light detectors to measure the relative intensity of light scattered from the sample (2). In some meter designs, a second detector is positioned to respond to transmitted light and to give a relative measure of attenuation resulting from light absorption in the beam and the scattering of light from the beam.
- 4.2 The area of illuminated particles, particle-volume concentration and the sample turbidity are directly proportional to one another in the linear range of a turbidimeter. Depending on meter design, the range can be as little as 40 TU or as large as 10 000 TU. As the concentration of light-scattering particles increases, the relative intensity of scattered light will increase linearly whereas the intensity of transmitted light will decrease exponentially. Beyond the linear range, the indicated turbidity value will be a nonlinear function of concentration. The linear range is larger for turbidimeters with closely spaced sources and detectors than for meters with wider source-detector spacing.
- 4.3 The method is based upon a comparison of the intensity of light scattered from and transmitted by a surface water sample with the intensity of light scattered from and transmitted by a reference light-scattering suspension (turbidity calibration or reference turbidity standard) using an in-situ turbidimeter. Unlike static measurements for which sample vials are placed in a bench-top or portable turbidimeter, in-situ measurements allow the meter to be placed in the water. A recent ASTM precision and bias study (see Test Method D7315) and independent research have demonstrated that different turbidimeters indicate different TU values for the same water sample even when calibrated with the same turbidity calibration standards. For some suspended matter, the indicated values can differ by a factor of ten. These differences are caused by a number of factors including the instrument design, light source, detector orientation, sediment color and grain size characteristics. It is therefore recommended that metadata, in the form of reporting units and appended model numbers in certain cases, be used when reporting TU values. In this way, data can be traced to the type of meter used, data compatibility will be enhanced, and long-term surface-water quality trends may be more apparent.

## 5. Significance and Use

5.1 Turbidity is monitored to help control processes, monitor the health and biology of aquatic environments and to determine the impact of environmental events such as storms, floods, runoff, etc. Turbidity is undesirable in drinking water, plant-effluent waters, water for food and beverage production,

and for a large number of other water-dependent manufacturing processes. Turbidity is often reduced by coagulation, sedimentation and water filtration. The measurement of turbidity may indicate the presence of particle-bound contaminants and is vital for monitoring the completion of a particle-waste settling process. Significant uses of turbidity measurements include:

- 5.1.1 Compliance with permits, water-quality guidelines, and regulations;
- 5.1.2 Determination of transport and fate of particles and associated contaminants in aquatic systems;
- 5.1.3 Conservation, protection and restoration of surface waters;
- 5.1.4 Measure performance of water and land-use management;
- 5.1.5 Monitor waterside construction, mining, and dredging operations:
- 5.1.6 Characterization of wastewater and energy-production effluents:
- 5.1.7 Tracking water-well completion including development and use; and
- 5.1.8 As a surrogate for other constituents in water including sediment and sediment-associated constituents.
- 5.2 The calibration range of a turbidimeter shall exceed the expected range of TU values for an application but shall not exceed the measurement range specified by the manufacturer.
- 5.3 Designs described in this standard detect and respond to a combination of relative absorption, intensity of light scattering, and transmittance. However, they do not measure these absolute physical units as defined in 3.2.15 and 3.2.19.
- 5.4 Several different turbidimeter designs may be used for this test method and one design may be better suited for a specific type of sample or monitoring application than another. The selection flowchart in Annex A1 provides guidance for the selection of an appropriate turbidimeter design for a specific application.
- 5.5 Report turbidity in units that reflect the design of the turbidimeter used as recommended in 4.3. See Table 1 and Section 7 for a discussion of the design criteria and derivation of reporting units.
- 5.6 Table 1 and Section 7 lists the turbidimeter designs currently used for in-situ measurements. Future revisions of the method may include additional designs.

#### 6. Interferences

- 6.1 Bubbles may interfere with turbidity determined by this test method. Bubbles cause turbidity values to be higher than they would be in bubble-free water and result in a positive interference.
- 6.2 Depending on the application color may or may not be considered as an interference. Color is characterized by absorption of specific wavelengths of light. If the wavelengths of incident light are significantly absorbed, a lower turbidity reading will result unless the instrument has special compensating features.

6.2.1 Color has less effect on a turbidimeter with an NIR operating spectrum, however, particle and water color may indicate the presence of NIR-absorbing matter as well as NIR reflectivity that can cause interferences. Particle reflectivity is considered an intrinsic turbidity factor. Those designs where color effects can be reduced or eliminated include nephelometric-based designs with incident light sources in the 780-900 nm range. Those designs that have additional detectors, such as ratioing instruments also help to reduce the effects of color regardless of the light source. Single detector systems with light sources below 780 nm will be more impacted by the effects of color in the sample, that is color visible to the naked eye. Color can have a significant impact on attenuation-based instruments if it has absorption spectrum that overlaps the spectral output of the incident light source. In some applications, the spectral reflectivity or color of suspended matter and light-absorbing dissolved matter are considered to be part of a turbidity measurement and not an interference.

Note 2—The user should not automatically assume that sample color will interfere with turbidity measurements. The only way to reliably determine whether or not it can is to filter the samples with 0.2-micron membrane filters and measure the absorbance spectra in the operating band of the turbidimeter with a spectrophotometer. If the integral of absorbance differs by more than  $10\,\%$  from the integral of absorbance for turbidity-free water in the same band, then measurable negative interferences from dissolved color can be expected.

Note 3—Particle color becomes an interference when it changes in an application while other factors remain constant, that is, particle size, shape, and composition. This can occur, for instance, during dredging operations and re-suspension events in settling ponds when light-colored oxidized sediment overlies dark-colored anoxic material of similar size and composition. In this situation, a turbidity spike occurs while light-colored sediment is re-suspended followed by a turbidity sag while anoxic material is re-suspended. The spike-sag sequence will occur even when the sediment concentration remains unchanged.

- 6.3 The particle-size distribution and operating spectrum will affect the relative sensitivity of turbidimeters. The intensity of light scattered from a water sample depends, among other factors, on the ratio of particle diameter to light wavelength. Since the operating wavelength of a turbidimeter is fixed, particle size is the controlling variable. Particle size can be a positive or negative interference when a user is unaware of decreases or increases in size while monitoring turbidity.
- 6.4 In-situ turbidimeters are intrusive devices that alter water flow and turbulence intensity near the turbidimeter. Flow disruption can change the location of light-scattering particles in the sampled water and the intensity of scattered light. The disturbed flow extends about three to five probe diameters away from the meter. Flow around a turbidimeter might cause particles to separate from the water in the sample volume and decrease the indicated TU value, or conversely, flow stagnation can concentrate particles and cause the indicated TU values to increase.
- 6.5 A large temperature difference between a turbidimeter and the surrounding water can result in measurement errors. In such situations, temperature can be an interference. Rapid surveys of thermal plumes or profiling in thermally stratified water can produce temperature interferences. The user should establish the magnitude of temperature interference by alternately testing water samples having the same turbidity but

substantially different temperatures, ~20°C. Fixed monitoring sites in river and lakes are less susceptible to temperature fluctuations because they are gradual, typically less <1°C per hour and the probe has sufficient time to come to temperature equilibrium with the stream temperature.

Note 4—Ambient light is a positive interference with some sensor designs. Locate the sensor to minimize ambient light or surface reflections, or both.

# 7. Apparatus

7.1 The turbidimeters discussed herein can be submerged in water for extended periods (weeks to years). Many of them are stand-alone instruments containing batteries, a microcontroller, and solid-state memory for data logging, whereas others are components of multiparameter instruments or must be connected to a host device such as a data logger or current meter for power and data recording.

Note 5—Meters with processing capabilities may perform real-time digital filtering, signal averaging, or smoothing that could obscure real transients in surface-water turbidity of interest to a user. For example, a meter with signal averaging installed a stream to monitor waterside construction could fail to record brief turbidity spikes caused by equipment operation. See manufacturer's specifications and instruction on signal averaging or smoothing before selecting a meter for real-time monitoring.

7.1.1 There are several technologies that are capable of measuring turbidity that exceed 1.0 turbidity unit. A summary of these technologies is provided in the Table 1. Within this table, suggested reporting units, which are representative to the technology, are included.

7.1.2 Clean optics are important in applications where biofouling, chemical precipitation, or sedimentation can render a turbidimeter dysfunctional between service calls. Fouling is the biggest challenge facing users and manufacturers. Several approaches have been devised to cope with it, including: wipers, shutters, water and compressed-air jets, ultrasonic shakers, and anti-foulant coatings. In this standard, they are collectively referred to as automatic-cleaning/anti-fouling (AC/AF) features. Tests in surface waters have shown that no combination of AC/AF features performs satisfactorily in all environments for more than a few months. They can, however, prolong the time between service visits and field recalibrations from weeks to a few months, which makes them key meterselection criteria for users who establish unattended monitoring stations. The tradeoff between increased power consumption for automatic cleaners and extended service requirements needs to be factored into the selection process.

7.1.3 Because of the variety of turbidimeter designs and manufacturers, selection of a design for a particular application is important. See 7.2 and 7.6 for a discussion of each of the designs. Annex A1 provides guidance to assist a user in the selection of a turbidimeter appropriate for a particular application. Appendix X1 provides detailed apparatus design considerations for in-situ turbidimeters. It is highly recommended that the user read these sections carefully before selecting a turbidimeter and using this test method.

## 7.2 *The Nephelometer:*

7.2.1 This instrument uses a light source for illuminating the sample and a single photodetector with a readout device to

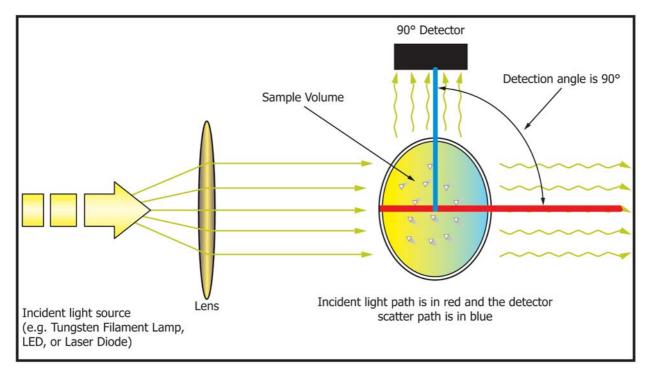
indicate the intensity of light scattered at right angle(s) (90°) to the centerline of the path of the incident light. The photoelectric nephelometer should be designed so that minimal stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period. The light source shall be a Tungsten lamp operated at a color temperature between 2200 and 3000 K (EPA 180.1). Light Emitting Diodes (LEDs) or laser diodes in defined wavelengths ranging from 400-680 nm and 780-900 nm may also be used if accurately characterized to be equivalent in performance to tungsten using the same type of calibration and calibration verification standards. It is important to note that new technologies may not be covered by this test method. If LEDs or laser diodes are used, then the LED or Laser diode should be coupled with a monitor detection device to achieve a constant output. LEDs and laser diodes should be characterized by a wavelength of between 400 and 900 nm with a bandwidth of less than 60 nm. The total distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. The angle of light acceptance to the detector shall be centered at 90° to the centerline of the incident light path and shall not exceed  $\pm 10^{\circ}$  from the 90° scatter path centerline. The detector must have a spectral response that is sensitive to the spectral output of the incident light used.

7.2.2 Differences in physical design of nephelometers may cause 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 designs is not recommended. To minimize initial differences, the design criteria discussed herein should be observed (see Fig. 1).

7.2.3 Report in units of NTU if a white light source was used), or in units of FNU if a 780–900 nm light source was used.

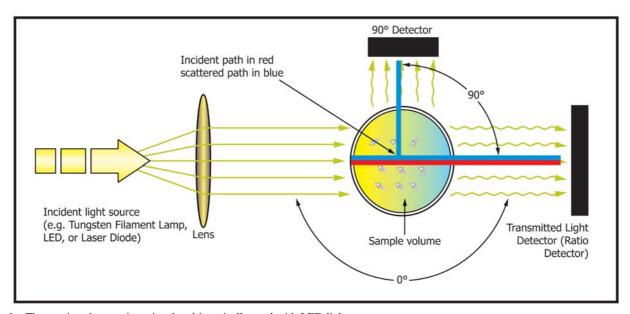
## 7.3 Ratio Nephelometer:

7.3.1 Ratio Nephelometer (see Fig. 2 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 detect 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, and 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 (EPA 180.1). 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



Note 1-Monitor detector is optional and its use is typically with LED light sources.

FIG. 1 The Photoelectric Nephelometer



Note 1—The monitor detector is optional and it typically used with LED light sources.

FIG. 2 The Ratio Photoelectric Turbidimeter

at  $90^{\circ}$  to the centerline of the incident light path and should not exceed  $\pm 10^{\circ}$  from the scatter path centerline. 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 7.2.1.

7.3.2 Differences in physical design of ratio photoelectric nephelometers may cause differences in measured values for turbidity even when the same suspension is used for calibrations. Comparability of measurements made using instruments differing in optical and physical design is not recommended. To minimize initial differences, the design criteria described in

- 7.3.1 should be observed (see Fig. 2 for a single beam design and Fig. 3 for a multiple beam design).
- 7.3.3 Report in the appropriate units using Table 1 as guidance.
- 7.3.3.1 FNRU, and FNMU signify the use of an incident light wavelength between 780–900 nm. NTRU and NTMU signify the use of an incident light in the wavelength range of 400–680 nm for a ratio technology.
  - 7.4 Backscatter Turbidimeters:
- 7.4.1 The instrumentation contains a light source that meets or exceeds the criteria specified in 7.2.1 for illumination of the sample.
- 7.4.2 The response curve of the detector should be such that it overlaps the output of the light source.
- 7.4.3 The detection angle for backscatter is to be set between 90° and 180° relative to the centerline of the incident light beam. See Fig. 4.
- 7.4.4 When reporting turbidity, report in units that best fit the light source and detector in Table 1. Report in BU (white light source) or FBU (if a 780–900 nm light source was used).
  - 7.5 Attenuation-Based Turbidimeters:
- 7.5.1 The instrument contains a light source that meets or exceeds the criteria specified in 7.4.1 for illumination of the sample. Examples include monochromatic light such as those generated in spectrophotometers.
- 7.5.2 The detector response curve should overlap the incident light source.
- 7.5.3 The detection angle for attenuation is to be set at 180 Degrees relative to the centerline of the incident light beam. See Fig. 5.
- 7.5.4 When reporting turbidity, report in units that best fit the light source and detector in Table 1. Report in AU (white light source) or FAU (if a 780–900 nm light source was used).
  - 7.6 Forward Scatter Turbidimeters:
- 7.6.1 Forward Scatter Technologies—This technology encompasses a single, solid-state light source and either a single detector or multiple detectors (ratio).

- 7.6.2 The detection angle for the forward scatter detector is greater than  $0^{\circ}$  but less than  $90^{\circ}$  relative to the centerline of the incident light beam.
- 7.6.3 A second ratioing detector may be incorporated into some designs. See Fig. 6.

#### 8. Purity of Reagents and Materials

- 8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>6</sup>
- 8.1.1 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.

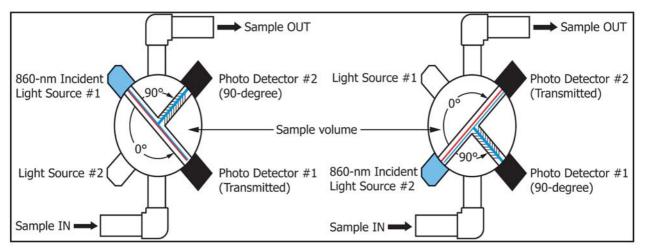
Note 6—Refer to product SDS for possible health exposure concerns.

8.2 Reverse osmosis (RO) water is acceptable and preferred in this test method. Standard dilution waters and rinse waters should be prepared by filtration through a 0.22 µm or smaller membrane filter or any other suitable filter within 1 hour of use to reduce background turbidity. Type III water is also acceptable (see Specification D1193). These types of water should be used in preparation of turbidity standards for calibration or verification.

### 9. Reagents

- 9.1 Dilution and final rinsing water, see 8.2.
- 9.2 Turbidity Standards:

<sup>&</sup>lt;sup>6</sup> Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



Note 1—The incident light path is in red and the scattered light paths are in blue.

FIG. 3 Multiple Beam Design Utilizes Two Detectors and Two Light Sources

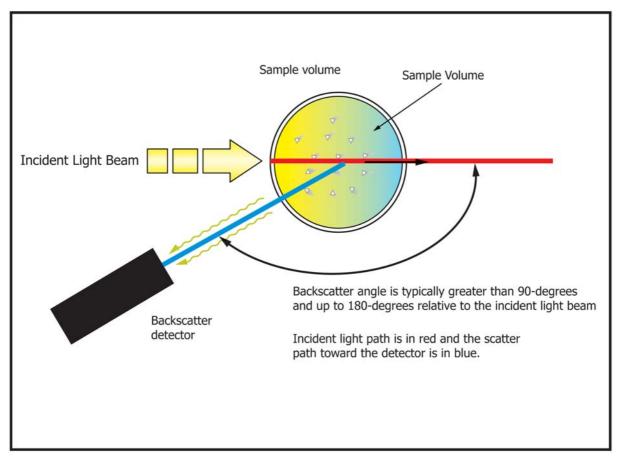


FIG. 4 Backscatter Measurement Design

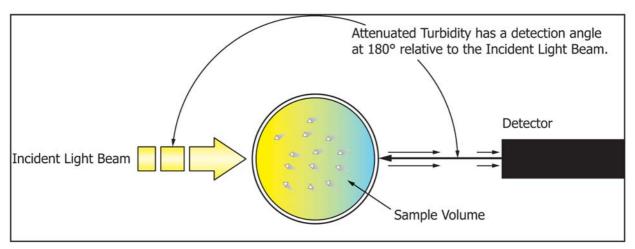


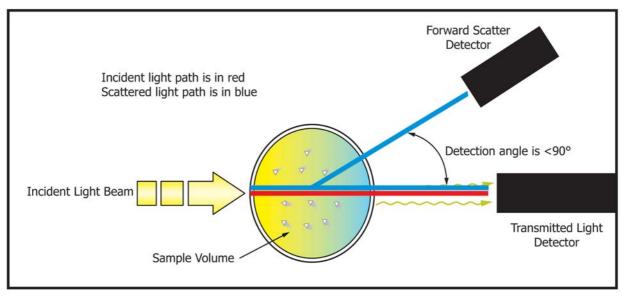
FIG. 5 Geometric Design of Attenuation for Turbidity Measurement

Note 7—A standard with a turbidity of 1.0 NTU is the lowest formazin turbidity standard that should be produced on the bench. Skilled laboratory personnel with experience in quantative analysis shall perform preparation of formazin standards. Close adherence to the instructions within this section is required in order to accurately prepare low-level turbidity standards.

Note 8—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 bench-synthesized formazin (see 9.2.2). Follow specific manufacturer's calibration procedures.

9.2.1 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 with rinse water as defined in 8.2.

9.2.2 Reference Formazin Reference Turbidity Standard, 4000 NTU—This standard is synthesized in the lab and is the primary standard against which other standards are traced.



Note 1—The FSU design only includes the forward scatter detector. The FSRU design incorporates both the forward scatter and transmitted detectors.

FIG. 6 Forward Scatter (FSU) and Forward Scatter Ratio (FSRU) Measurement Designs

9.2.2.1 Quantitatively transfer 5.000 grams of reagent grade hydrazine sulfate (99.5 % + purity) ( $N_2H_4\cdot H_2SO_4$ ) into approximately 400 mL of dilution water (see 8.2) contained in a 1-litre Class A volumetric flask; stopper and completely dissolve by swirling.

Note 9—To quantitatively transfer this powdered reagent, transfer the hydrazine sulfate into the flask containing the dilution water. Rinse the weighing bowl with dilution water, adding the rinsings to the flask. Repeat the rinsing again adding the second rinsings to the flask.

9.2.2.2 Quantitatively transfer 50.000 grams of reagent grade hexamethylenetetramine (99+ % purity) in approximately 400 mL of dilution water (see 8.2) contained in a clean flask; stopper and completely dissolve by swirling. Filter this solution through a 0.2 µm filter into a clean flask.

9.2.2.3 Quantitatively transfer the filtered hexamethylenete-tramine into the flask containing the hydrazine sulfate. Dilute this mixture to 1 L using dilution water (see 8.2). Stopper and mix for at least 5 minutes, and no more than 10 minutes.

Note 10—To quantitatively transfer this liquid mixture, transfer the hexamethylenetetramine into the flask containing the hydrazine sulfate. Rinse this flask two times using 50 mL aliquots of dilution water, adding each rinsing to the flask containing the hydrazine sulfate.

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

Note 11—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.

9.2.3 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.

9.2.4 SDVB standards are prepared stable suspensions of copolymer microspheres which are used as received from the

manufacturer or distributor. These standards exhibit calibration performance characteristics that are specific to instrument design.

9.2.5 Formazin Turbidity Suspension, Standard (40 NTU)— This is an example on how to prepare a calibration standard of a specific turbidity value. All labware shall be seasoned (see Appendix A.4). Invert 4000 NTU stock suspension 25 times to mix (1 second inversion cycle); immediately pipette, using a Class A pipette, 10.00 mL of mixed 4000 NTU stock into a 1000-mL Class A volumetric flask and dilute with water to mark. The turbidity of this suspension is defined as 40 NTU. This 40-NTU suspension must be prepared weekly.

9.2.6 Other Formazin Calibration Standards—Using a similar procedure as in 9.2.5, prepare the appropriate standards as required to calibrate the instrument as instructed by the instrument calibration protocol.

9.2.7 Dilute Formazin Turbidity Suspension Standard (1.0 NTU)—Prepare this standard daily by inverting the 40 NTU (9.2.5) stock suspension 25 times to mix (1 second inversion cycle) and immediately pipet a volume of 40 NTU standard. All glassware shall be seasoned (see Appendix A.4).

Note 12—The instructions below result in the preparation of 200 mL of a 1-NTU formazin standard. Users of this test method will need different volumes of the standard to meet their instrument's individual needs; glassware and reagent volumes shall be adjusted accordingly.

9.2.7.1 Within one day of use, rinse both a glass Class A 5.00 mL pipette and a glass Class A 200 mL volumetric flask with laboratory glassware detergent or 1:1 hydrochloric acid solution. Follow with at least ten rinses with rinse water. Cap and store in a clean environment until use.

9.2.7.2 Using the cleaned glassware, pipet 5.00 mL of well-mixed 40.0 NTU formazin suspension (9.2.5) into the 200 mL flask and dilute to volume with the dilution rinse water. Stopper and invert (1 second inversion cycle) 25 times to mix. The turbidity of this standard is 1.0 NTU.

- 9.2.8 Miscellaneous Dilute Formazin Turbidity Suspension Standard—Prepare all turbidity standards with values below 40 NTU daily. Standards ≥40 NTU have a useful life of one week. All labware shall be seasoned (See Appendix C). Use Class A glassware that has been cleaned in accordance with the instructions in 9.2.1 and prepare each dilution by pipetting the volume of 40 NTU (9.2.5) into a 100-mL volumetric flask and diluting to mark with dilution water (8.2). For example, prepare the solution so that 50.0 mL of 40 NTU diluted to 100 mL is 20.0 NTU and 10.0 mL of 40 NTU diluted to 100 mL is 4.00 NTU.
- 9.2.8.1 Prepare standards at the turbidity concentrations that are required to meet the specific calibration requirements for the instrument that is to undergo calibration.
- 9.2.9 Stable turbidity standards are commercially available. These standards, such as stabilized formazin and styrenedivinylbenzene, have a specific turbidity value and accuracy. Such standards must be traceable to the reference turbidity standard.

#### 10. Hazards

- 10.1 Wear appropriate personal protection equipment at all times.
  - 10.2 Follow all relevant safety guidelines.
- 10.3 Refer to instrument manuals for safety guidelines when installing, calibrating, measuring or performing maintenance with any of the respective instrumentation.
- 10.4 Refer to all Safety Data Sheets (SDS) prior to preparing or using standards, before calibrating or performing instrument maintenance, and for proper disposal information.

## 11. Sampling

11.1 The method requires in-situ measurements for which a turbidimeter is placed in the surface water. The water is not sampled and then placed in a meter as in the static methods.

#### 12. Preparation of Apparatus

12.1 The equipment and supplies commonly used for field measurement of in-situ turbidity are listed in Table 2. These include supplies generally needed for the maintenance, storage, and cleaning of the selected instrument. Routine maintenance of turbidity instrumentation is critical, particularly for continuously deployed, dynamic applications. See Guide D3864 for additional information regarding on-line monitoring.

## 13. Calibration and Calibration Verification

- 13.1 Calibrate the instrument using calibration turbidity solutions before leaving for the field. While in the field, check instrument performance periodically using a calibration or verification calibrant and turbidity-free water. Consult the manufacturer's specification for the expected accuracy of the measurement.
- 13.2 The optical surface of the sensor must be clean before beginning the calibration procedure. In deployed, continuous monitoring situations, pipes or other structures that house the sensor also may require periodic cleaning.

Note 13-To calibrate a submersible turbidity sensor modify the

#### TABLE 2 Equipment and Supplies Used for Measuring Turbidity

Turbidimeter, spectrophotometer, or submersible-sensor instrument (such as a multi-parameter instrument with a turbidity sensor).

Calibration turbidity stock solutions and standards

- -Formazin stock suspension, commercially obtained or prepared from scratch with hydrazine sulfate and hexamethylenetetramine chemicals. or
- -Instrument-specific polymer solutions containing styrene divinylbenzene beads.

Sample cells (cuvettes), clear colorless glass (supplied from instrument manufacturer).

Sample bottle (preferably an amber bottle that does not adsorb suspended material).

Silicon oil, optical grade (with same index of refraction as sample cells; supplied by instrument manufacturer).

Lint free tissues or cloths.

Turbidity-free water, deiononized water filtered through a  $\leq$ 0.2 mm filter membrane with precision-sized pores.

Bottle to hold turbidity-free water, cleaned and rinsed three times with filtered water

Volumetric flask, Class A, 100 mL or 500 mL.

Volumetric pipet, Class A, 5.0 mL and pipet filler.

general instructions that follow as necessary so that they are compatible with the manufacturer's instructions.

- 13.3 Prepare a sufficient volume of the selected calibration solution or verification calibrant. The volume of calibrant required can vary significantly, depending on the sensor being calibrated. The container that is to contain the calibrants should be scrupulously cleaned prior to use. Consult the manufacturer for the required volume of calibration material, type of calibrant needed, and recommended cleaning steps.
- 13.3.1 Select Procedure A or Procedure B. The same procedure, once tested and selected, also should be applied to instruments used in future studies against which the data could be compared:
- 13.3.1.1 *Procedure A*—Immersion of the entire sensor (bundle of field-measurement sensors, including the turbidity sensor) requires larger volumes of calibrant; calibrant is vulnerable to contamination and dilution. The sensor guard may need to be removed.
- 13.3.1.2 *Procedure B*—Immersion of turbidity sensor only—depending on sensor configuration, isolation of the turbidity sensor and achieving a bubble-free optical surface could be difficult. This technique minimizes the volume of calibrant required for calibration.

Note 14—If the instrument has a mechanical wiper, it is usually a good idea to clean and run the wiper. This will help to insure clean optics before performing calibrations.

- 13.3.2 Determine the number of calibration points to be used (a minimum of two, but three is preferred) and configure the instrument for this number of points, if applicable.
  - 13.3.3 For a turbidity-free water calibrant:
- 13.3.3.1 Rinse sensor with deionized water, followed by a portion of the turbidity calibrant.

- 13.3.3.2 Immerse sensor in calibrant, or add enough calibrant to cover the sensor in the calibration chamber.
- 13.3.3.3 Agitate the sensor repeatedly to remove bubbles from the optical surface (activate mechanical wiper, if present).
- 13.3.3.4 Set sensor vertically on a flat surface or use a ring stand to hold it.
- 13.3.3.5 Monitor turbidity readings for 1 to 2 minutes or longer to ensure that readings are stable (consult manufacturer's recommendations and signal-processing information). Record the pre-calibration value in the instrument logbook or on the field sheet.
- 13.3.3.6 Confirm the calibration value or adjust the instrument calibration using the manufacturer's instructions.
- 13.3.3.7 Remove the sensor and dry thoroughly to minimize dilution or contamination of the next calibrant or rinse thoroughly with DI water to avoid possible damage to the sensor.
- 13.3.3.8 Discard the calibrant into a labeled waste container and hold for proper disposal.
- 13.3.3.9 If measurement of color-derived turbidity is not desired, filter (using a 0.2- $\mu$ m pore-size filter) an aliquot of the sample water and use the filtered water in place of turbidity-free water.
- 13.3.4 Using calibrants necessary to calibrate over the expected working range, repeat steps in 13.3.3.1 through 13.3.3.9. Calibrations are typically performed from lowest to highest values. Follow manufacturer's instructions for calibration.
- Note 15—If an "out of range" error is displayed, verify the intended calibrant value and start again with the first (zero) calibrant solution. Repeat the calibration procedure if the measurement is not within the specification. Record all calibration and verification measurements in the instrument logbook.
- 13.3.5 On a one-time basis, determine the maximum value that can be reported by the instrument by holding a lint-free cloth over the optical sensor and recording the turbidity. Use this value as an indicator that turbidity might have been greater than the range of the instrument during measurements in a water body.

Note 16—Calibration procedures vary widely for turbidimeters. Manufacturers typically provide the calibration procedure that will provide the best performance of their respective equipment. Many manufactures will also make procedures locked so users cannot calibrate in error. Because of this, the above procedures should be used as a guide to calibrating the instrument.

# 14. Procedure

- 14.1 Before making an in-situ turbidity determination, ensure that the instrument to be used has been cleaned, verified, and or calibrated properly, and that the verification/calibration process has been accurately documented.
- 14.1.1 Make sure all instrument settings are selected. These include signal averaging, measurement rate, data log rate, and bubble removal algorithms.
- 14.2 Multi-parameter instruments with internal batteries and memory can be used in surface-water studies that require long-term deployment. Guidelines for long-term instrument deployment fall under the topic of continuous monitors, and are beyond the scope of this standard—refer to the manufac-

turer's instructions and recommendations, and to guidance documents such as Wagner and others (3).

14.3 Perform a calibration verification on the instrument in the laboratory or office using a calibration solution before leaving for the field and if verification does not confirm the calibration is still within 10 percent, then re-calibrate as outline in Section 13.

Note 17—In this test method the pass/fail criteria are used as an example, the value may differ depending on the pass/fail criteria for the specific application in which this test method is referred. At the time this test method was drafted, a commonly accepted criterion was at 10~%.

- 14.4 At the field site, verify that the instrument has retained its calibration within 10 percent. If it fails verification, then the instrument must be re-calibrated.
- 14.4.1 Immerse the turbidity sensor in the water body in a portion of flow that is well mixed and represents the average condition of the cross section being measured.
- 14.4.2 Activate the instrument to display turbidity values in real time.
- 14.4.3 Agitate the turbidity sensor to remove bubbles from the optical surface: move the sensor up and down or in a circular pattern and (or) activate the wiper mechanism, if available.
- 14.4.4 Allow at least 60 seconds (or follow the manufacturer's guidelines) for sensors to equilibrate with sample water. Take instrument readings until the stabilization criteria of  $\pm 10$ % is met. Record the median of the final three or more readings as the value to be reported for that measurement point. (Some instruments may require as much as 10-20 minutes warm-up time.) Stability is reached if values for three or more sequential readings, spaced at regular time increments, are within 10 percent.

Note 18—Most current designs have rapid data logging rates. If these rates are available, it is recommended that several measurements be made on a sample and then averaged to generate a final result. The ASTM round robin generated data in this format, collecting 29 measurements and averaging them to generate the turbidity value on each sample.

- 14.4.5 Record turbidity readings on the field form and in field notes, including the instrument manufacturer and model. Use reporting units appropriate for the instrument, as described in Table 1.
- 14.4.6 For surface water sites that may not be well mixed or during rapidly changing conditions such as during storm events, multiple points of measurement are needed to determine a representative field-measurement value. It may be necessary to repeat steps 14.4.3 14.4.5 at several verticals across the stream in order to determine a turbidity value that best represents the mean turbidity of the surface water at that site. To determine the number and location of verticals in the cross section being measured, refer to USGS National Field Manual sections 6.0.2.A and 4.1 (4), Guide D4411, and Edwards and Glysson (5).
- 14.4.7 Before leaving the field, clean the sensor with a thorough rinse of deionized water and place it in the storage vessel. Follow the manufacturer's recommendations for storage of sensors.
- 14.4.8 If turbidities are higher than the instrument range, dilutions will be necessary. Turbidity will need to be measured

with static methods (Test Method D7315). Take a representative sample and dilute it with one or more equal volumes of turbidity-free water, recording the volume of water used for dilution. Also consider using a different technology that may have a higher instrument range.

Note 19—Test Method D7315, Appendix A, section A2.5, states "Dilutions: although the average of all technologies resulted in a reasonable error range, individual technologies, with the exception of FAU and FBU, experienced greater than 10 % differences when compared to the original sample readings."

## 15. Report

15.1 Report results using the units recommended in Table 1 and the numerical tolerances listed in Table 3. Report results using the units that represent the technology used (see Table 1).

### 16. Precision and Bias

16.1 The precision of this test method is based on an inter-laboratory study. Each of ten laboratories tested seven different materials. Every "test result" represents an individual determination, and all participants reported duplicate test results. Practice E691 was followed for the design and analysis of the data; the details are given in the published research report.<sup>7</sup>

Note 20—Even though it could not meet requirements of Practice D2777, a collaborative study with a statistically significant sample of the major designs and a group of representative water samples would produce valuable information about the efficacy of the recommended design-based data-reporting scheme. The success of such a study will critically depend on getting a sufficient number of meters of each major design type to characterize significant differences among designs should they exist. Another major challenge will be the selection of a manageably small group of water samples that represent the huge variability of suspended matter that is encountered in surface waters. The effects of size and SSC have been adequately represented and the collaborative study should focus on properties such as: shape, color and NIR reflectivity, refractive index, flocculation/aggregation, and particle absorbance that are not so well characterized.

16.2 Repeatability Limit (r)—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

16.2.1 The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of

TABLE 3 Reporting of Results for In-situ Turbidity Measurements

Measured Value – In appropriate Units	Report to Nearest		
1.0 < 9.9	0.1		
10 < 99	1		
100–999	10		
1000<	50		

time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

- 16.2.2 Repeatability limits are listed in Table 4.
- 16.3 Reproducibility Limit (R)—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.
- 16.3.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.
  - 16.3.2 Reproducibility limits are listed in Table 4.
- 16.3.2.1 The reproducibility limits are across all participating laboratories and technologies. This represents the broadest range. Thus, all values in Table 4 are in generic turbidity units (TU).
- 16.3.2.2 If the same technologies (with the same reporting units from Table 1) are used precision will improve. However, this study did contain the minimum number of participants with the same type of technology to generate a separate precision statement for each type of measurement technology. Please refer to Test Method D7315 precision section which was able to report precision for a given type of measurement technology.
- 16.4 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.
- 16.5 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method utilized, therefore no statement on bias is being made.

Note 21—Determination of the bias method is not possible because of the inherent instability of turbidity in surface water and the potential for misrepresenting meter performance in a lab simulation of turbid surfacewater flow. There are many possible causes for error in field measurements that are unrelated to the meters themselves. Few of them, if any, have been characterized for the designs presented here even in a relative sense.

16.6 The precision statement was determined through statistical examination of 143 results, from ten laboratories, on seven materials.

- 16.6.1 Round Robin Study Description:
- 16.6.1.1 This turbidity measurement round robin only determines precision. Bias determination was not possible because there is no acceptable primary turbidity standard material that occurs naturally and is capable of consistently delivering defined turbidity value.
- 16.6.1.2 A total of ten laboratories participated in this round robin.
- 16.6.1.3 Each laboratory performed duplicate measurements of each of seven different samples. Four of the samples were real world samples that were collected in close proximity to the round robin venue. Three surrogate samples were transported to the round robin venue for measurement.
- 16.6.1.4 All laboratories and samples were transported to a common venue where the round robin could be completed in a

<sup>&</sup>lt;sup>7</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1192. Contact ASTM Customer Service at service@astm.org.

TABLE 4 Turbidity (TU) - Precision for In-Situ Turbidity Measurement Technologies

Material	Average <sup>A</sup> x̄	Repeatability Standard Deviation s <sub>r</sub>	Reproducibility Standard Deviation s <sub>R</sub>	Repeatability Limit r	Reproducibility Limit R
Surrogate Unknown A: Expected Value 1-20 TU	11.902	0.028	2.355	0.077	6.593
Surrogate Unknown B: Expected Value 50-500 TU	114.730	0.222	21.582	0.622	60.429
Surrogate Unknown C: Expected Value 500-2000 TU	737.802	1.747	336.960	4.892	943.488
Real World Sample 4 in the field	4.223	0.118	1.173	0.331	3.284
Real World Sample 5 in the field	171.658	1.866	45.055	5.225	126.155
Real World Sample 6 in the field	29.831	0.634	7.596	1.776	21.268
Real World Sample 7 in the field	7.159	0.147	1.448	0.412	4.054

<sup>&</sup>lt;sup>A</sup> The average of the laboratories' calculated averages.

short period of time, thereby minimizing the possibility of samples changing in turbidity over the duration of measurement.

# 16.6.1.5 For a given sample:

- (1) All samples were allowed to acclimate to room temperature before measurement. Each sample was placed into a common black-walled container where it could be measured by each of the round robin sensors. The sample was continuously mixed before and during the measurement to insure homogeneity throughout the measurement. The sample container was black, with a capacity of 5 gallons and the lid covered the container between measurements.
- (2) Each in-situ sensor then measured the sample, one at a time, but collecting duplicate sets of measurements.
- (3) Between measurements, the sensor was rinsed clean prior to immersion into a sample.
- (4) When measuring the surrogates, they were measured from lowest to highest turbidity.
- (5) At the beginning and end of the in-situ sensor measurement cycle, aliquots of the sample were collected and measured on benchtop turbidimeters (references). These reference instruments were used to validate the sample stability over the duration of its in-situ analysis by all the participants.

## 16.6.2 Laboratories:

16.6.2.1 Several different laboratories participated in this round robin. These technologies report in distinct units that represent the respective technology (see Table 1). These included FNU (five laboratories), FNRU (two laboratories), FBU (one laboratory), FSRU (one laboratory), NTRU (one laboratory).

16.6.2.2 The round robin precision data (see Table 3) represents all the laboratories at the same time.

#### 17. Quality Control (QC)

- 17.1 In order to be certain that the analytical values obtained using this test method are valid and accurate, the following QC procedures must be followed when running the test.
- 17.2 Initial Instrument Calibration and Calibration Verification:
- Note 22—Instrument calibration and calibration verification is also covered in greater detail in Sections 13 and 14 of this test method.
- 17.2.1 The optical surfaces of the sensor and calibration containers must be clean before beginning the calibration procedure (13.2).

- 17.2.2 Calibrate the instrument using calibration standard before leaving for the field. Use calibration standards and procedures that are recommended or required by the specific instrument manufacturer (13.1).
  - 17.2.3 Initial Instrument Calibration Verification:
- 17.2.3.1 Immediately verify the instrument calibration by analyzing at least two turbidity standards that are not the original calibration solutions. Use standards in the anticipated range of use.
- 17.2.3.2 The response in measurement for the turbidity verification standard(s) shall fall within 0.5 turbidity units (TU) or 10 %, of the theoretical value of the turbidity verification standards, whichever is greater.
- 17.2.3.3 If calibration cannot be verified, recalibrate the instrument (Section 13).
- 17.3 Subsequent Instrument Verification (Frequency is determined by application):
- 17.3.1 Perform any required maintenance and cleaning of the sensor.
- 17.3.2 Verify the instrument calibration (frequency) by analyzing at least one turbidity standard.
- 17.3.3 The response in turbidity measurement for the turbidity verification standard shall fall within 0.5 TU or 10% of the theoretical value of the turbidity verification standard, whichever is greater.
- 17.3.4 If calibration cannot be verified, recalibrate the instrument (Section 13).
- 17.4 *Method Blank*—A method blank is not applicable to this test method. this test method is designed to measure waters greater than 1 TU.
- 17.5 Matrix Spike—A matrix spike is not feasible with this test method because in-situ meters measure waters directly without constraint of a sample cell or chamber of defined volume. If matrix spikes are attempted using a closed container, the container itself may cause interferences that would not be observed under real measurement conditions.

## 17.6 Duplicates:

17.6.1 In-situ measurements are continuous monitoring instruments. Duplicates of consecutive measurements, are not an adequate determination of operator or laboratory performance. Instead, a defined number of at least 30 consecutive measurements should be collected and averaged to produce duplicate number 1. This is then repeated to produce duplicate number 2.



17.6.2 Duplicates 1 and 2 can be used to assess the precision of the operator and laboratory, and compared to the precision contained in Section 16.

# 18. Keywords

18.1 90° scatter; backscatter; forward scatter; in-situ turbidity; multi-beam meter; turbidimeter; turbidity; turbidity interferences; turbidity standards

#### **ANNEX**

(Mandatory Information)

# A1. SELECTION OF IN-SITU TURBIDITY METERS

A1.1 Selection Criteria Flowchart for High-Level Turbidity

Measurement—This criteria was developed as a cooperative effort between the ASTM sub-committee on High-Level turbidity measurement and the USGS. See Fig. A1.1.

Note A1.1—The technologies listed in this flowchart are those that are best applied for in-situ turbidity measurements. Other technologies may be available but may not be suitable for in-situ turbidity measurement applications.

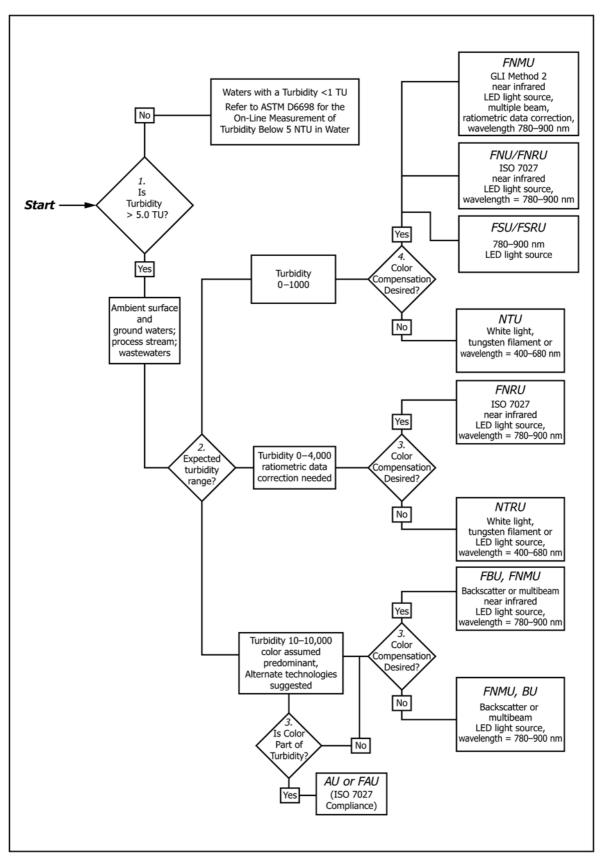


FIG. A1.1 Selection Criteria for In-Situ Measurement of Turbidity in Waters Above 1.0 NTU

#### **APPENDIXES**

(Nonmandatory Information)

#### X1. DETAILED APPARATUS DESIGN CONSIDERATIONS

#### **X1.1** General Design Considerations

X1.1.1 The intrinsic uncertainty of light-scattering measurements is governed by the statistics of the binomial distribution, which predicts a relative error proportional to  $N^{-1/2}$ , where N is the number of scattered photons that are detected. It is therefore advantageous to maximize N by selecting turbidimeters with: (1) bright, power-efficient sources, that is, sources that produce a lot of light per unit of electrical power, (2) large sample volumes, and (3) large-area detectors and big detector windows and field stops (see Figs. 1 and 2).

X1.1.2 Several features that enhance meter performance significantly and are useful criteria for turbidimeter selection include: (1) type of light source (that is, visible and NIR LEDs and laser diodes), and qualities such as source bandwidth, peak wavelength and spectral distribution; (2) source beam shape, such as collimated beams, comprising parallel light rays, while others shapes are conical and contain divergent, or convergent rays (7.1.3); (3) spectral sensitivity and temperature coefficient of detector(s); (4) the combined mean path length of source and scattered light beams in a sample should be appropriate for the application, for example shorter path lengths result in wider linear ranges and lower meter sensitivity, while long path lengths give high sensitivity; and (5) use of visible light rejection filters with NIR light sources or NIR rejection filters with visible light sources can extend the measurable range for scattered light intensity and improve meter resolution.

X1.1.3 In addition to optical characteristics, electronic features play an important functional role and should be considered when selecting a turbidimeter. These can include automatic power control of the source, phase-sensitive detection to reduce daylight interference, and computational algorithms that extend the range of a meter beyond the intrinsic linear operating range.

#### X1.2 Source Characteristics

X1.2.1 Solid-state sources used in in-situ turbidimeters produce light spectra that are not significantly blue-shifted after transmission through water in the way that tungsten-filament light is shifted. Light from the source is usually coupled directly to the source window by a collimating lens or by a lens focused on a fiber optic that conveys the light to the sample.

X1.2.2 A light baffle, for the light source, is an essential feature in backscatter turbidimeters because it prevents loss of measurement range resulting from in-phase coupling of light to the detector.

X1.2.3 The ISO 7027 standard states that the source beam can have no divergence and less than  $1.5^{\circ}$  of convergence. This is a very stringent specification, and to meet it a fiber-optic,

light-delivery systems must have a collimating optic at its distal end and a flat window or prism between the distal optic and the sample.

Note X1.1—It is the user's responsibility to verify ISO 7027 compliance by independent testing.

X1.2.4 In non-ISO designs, the maximum source-beam divergence angle should be less than 15° so that the maximum acceptance angle can be about 30°. These angles allow some flexibility in the meter optics.

X1.2.5 While it would seem advantageous to use short wavelengths to get the greater sensitivity, daylight interference must also be considered. In the sensitive band of a typical silicon photodiode, solar radiation contains roughly  $^{2}/_{3}$  rd visible-light energy and  $^{1}/_{3}$  rd NIR energy. The NIR is strongly absorbed, which limits its penetration into a water body as well as the propagation distance from a source. Visible light does not have these qualities and consequently is more likely to interfere with turbidity it measures is not true in general, that light-scattering intensity is proportional to  $\lambda^{-1}$ .

#### X1.3 Detector Characteristics

X1.3.1 Daylight-rejection filters and phase-sensitive detection circuits are important ways to compensate for daylight interference.

X1.3.2 It is important to account for refraction at the window-sample interface for in-situ turbidimeters. To ensure that the source beam and acceptance cone intersect at 90o in water, the detector angle in some turbidimeters will differ from 90° in order to compensate for refraction at water-window interfaces.

X1.3.3 The detector spectral sensitivity and source spectra should match one another as closely as possible. Blue-enhanced (NIR-repressed) photodiodes for instance should be used with white and visible monochromatic LEDs and NIR-enhanced photodiodes should be used with IRED's and NIR laser diodes. When used in combination with NIR-absorbing or daylight-rejection filters, as appropriate, these photodiodes reduce interferences caused by visible solar radiation.

X1.3.4 For the silicon photodiode detectors, not only should the spectral sensitivity approximately match the source spectrum but the temperature coefficients should be in the  $\pm 100$  ppm range. The detector-aperture diameter (field stops in Fig. 1) should equal the maximum cross section of the detector active area. In order to compensate for source-beam spreading at high turbidity levels, the acceptance-cone angle should exceed the source-divergence angle by a factor of about two. Coupling the scattered light from the acceptance cone to the detector may require a window and an optic (lens or prism) or multiple apertures. These components should be configured so as to prevent the incidence of stray light on the detector.



Daylight contains sufficient visible and NIR energy to saturate the photodiode electronics of an in-situ turbidimeter, even when it is under a meter of water. Saturation will cause a meter to display or record erroneous TU values. A daylight rejection filter placed over the photodiode, combined with phasesensitive detection to reject daylight that gets through the filter, can remedy this problem.

X1.3.5 In Formazin Back Scatter and Backscatter turbidimeters, a backscatter detector angle should be set to

measure light scattering at angles in the range from  $110^{\circ}$  to  $150^{\circ}$  because the sensitivity of the detectors remains nearly constant at these angles. So the design requires less optical components and is simpler to implement than other designs.

X1.3.6 In Formazin Back Scatter and Backscatter turbidimeters, the detector-aperture area should be as large as a sensor package permits to collect the maximum amount of scattered light and the detector active area should be about half of aperture area.

## X2. DISCUSSION OF OPTICAL TERMS USED IN THIS TEST METHOD

X2.1 Introduction—Appendix X2 provides additional discussion of several optical-engineering terms that may be related to technologies that are provided in this test method. This discussion is included to help clarify practical aspects of turbidity measurement, but is not part of the standard-specific definitions found in the Terminology section of this test method (Section 3); thus, Appendix X2's format does not follow the strict ASTM rules required for terminology.

## **X2.2** Optical Engineering Terms

X2.2.1 *absorption*, *n*—the conversion of light energy to heat as the light passes through water and suspended matter. Turbidity meters do not directly measure absorption; however, absorption affects TU value, independent of light scattering.

X2.2.2 automatic power control (APC), n—automatic power control is achieved by means of: (1) the output from a light-monitoring detector in the meter or incorporated in the solid-state light source and (2) an electronic-feedback circuit.

X2.2.3 *color*; *n*—color can indicate the presence of dissolved matter that absorbs light in the operating spectrum (3.2.11) and causes a turbidimeter to indicate a lower TU value than would be read in an uncolored sample. Suspended matter can also cause sample color that can raise or lower turbidity, depending on the reflectivity of suspended particles. For example, a meter that reads 10 TU in a sample containing 100 mg/L of 10-micron coal dust would read about 100 TU in a sample with 100 mg/L of 10-micron quartz silt.

X2.2.4 *detector*, *n*—the ratio of electrical current (in amperes, A) to the light power incident on a detector (in watts, W) gives the detector's sensitivity in A W<sup>-1</sup>. Sensitivity is a spectral quantity that varies with wavelength. Nearly all turbidimeters use silicon photodiodes because they are stable, compact, and inexpensive, in addition to having nearly constant sensitivity over five decades of light power.

X2.2.5 detector angle, n—in a bench-top meter with cylindrical, thin-walled sample vials, the detector angle equals 180° minus the scattering angle (3.2.16). As a result of refraction at window-sample interfaces, this identity gives erroneous results for Type I and Type II meters with parallel source and detector windows. For example, a turbidimeter with a source beam and an acceptance cone that intersect at 90° in air would be classified a backscatter meter, with beams intersecting at 66° when immersed in water.

X2.2.6 *intermediate-band source*, *n*—"white-light" sources with band-pass filters, LEDs, and infrared-emitting diodes (IREDs) can be used to create intermediate-band sources.

X2.2.7 narrow band source, n—visible and NIR-laser diodes are narrow-band sources.

X2.2.8 operating spectrum, n—the light-source and operating spectra are generally not the same because of optical filters used in the meter, absorption of light by a sample, and spectral sensitivity of a detector. Distortion of the source spectrum by these factors is insignificant for meters with LED, IRED, and laser-diode sources; however, the distortion can be substantial when unfiltered tungsten-filament lamps are used.

X2.2.9 *sample volume, n*—typical in-situ turbidimeters have sample volumes ranging from 25 to  $12 \times 10^4$  mm<sup>3</sup> (that is, BBs to tennis balls).

X2.2.10 scattering (also referred to as scatter), n—the scattering coefficient, b (in m<sup>-1</sup>), is the absolute measure of this property. Turbidimeters do not measure 'b' directly; however, they respond to scattering-coefficient variations that can be directly proportional to TU value.

X2.2.11 scattering angle  $(\theta)$ , n—in a bench-top meter, the scattering and detector angles are related by a simple identity. However, this identity will give erroneous results for Type I and Type II meters that have parallel source and detector windows.

X2.2.12 *transmittance*, *n*—scattering and absorption (both of which can be caused by water and suspended matter) remove light from a beam as it passes through a sample. These factors cause sample transmittance to decline exponentially in proportion to turbidity and to the concentration of dissolved light-absorbing matter in the sample. Transmittance can interfere with turbidity, but cannot be measured directly by turbidimeters.

X2.2.13 turbidimeter sensitivity, n—variation in sensitivity from one design to another is the primary reason that two meters do not read the same TU value in a water sample. Tests have shown that ISO 7027 and backscatter turbidimeters have sensitivities ranging from about 0.05 to 1.7 TU ppm<sup>-1</sup> for silt and clay suspensions, and that two meters can indicate TU values that differ by as much as a factor of 4 for a given sample.

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