



Designation: D 7315 – 07

## Standard Test Method for Determination of Turbidity Above 1 Turbidity Unit (TU) in Static Mode<sup>1</sup>

This standard is issued under the fixed designation D 7315; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the static determination of turbidity in water. Static refers to a sample that is removed from its source and tested in an isolated instrument. (See Section 4.)

1.2 This test method is applicable to the measurement of turbidities greater than 1.0 turbidity unit (TU). The upper end of the measurement range was left undefined because different technologies described in this test method can cover very different ranges. The round robin study covered the range of 0–4000 turbidity units because instrument verification in this range can typically be covered by standards that can be consistently reproduced.

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.

1.3.1 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.

1.4 This test method does not purport to cover all available technologies for high-level turbidity measurement.

1.5 This test method was tested on different natural waters and wastewater, and with standards that will serve as surrogates to samples. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.6 Depending on the constituents within a high-level sample, the proposed sample preparation and measurement methods may or may not be applicable. Those samples with the highest particle densities typically prove to be the most difficult to measure. In these cases, an alternative measurement method such as the process monitoring method can be considered.

<sup>1</sup> 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.

Current edition approved April 15, 2007. Published May 2007.

1.7 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. Refer to the MSDSs for all chemicals used in this procedure.

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- D 1129 Terminology Relating to Water
- D 1193 Specification for Reagent Water
- D 1889 Test Method for Turbidity of Water
- D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
- D 4411 Guide for Sampling Fluvial Sediment in Motion
- D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
- D 6855 Test Method for Determination of Turbidity Below 5 NTU in Static Mode
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

#### 2.2 Other Referenced Standards:

- USEPA Method 180.1 Methods for Chemical Analysis of Water and Wastes, Turbidity<sup>3</sup>
- ISO 7027 Water Quality—Determination of Turbidity<sup>4</sup>
- United States Geological Survey (USGS) National Field Manual for the Collection of Water Quality Data<sup>5</sup>

### 3. Terminology

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

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from United States Environmental Protection Association (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

<sup>4</sup> Available from International Organization for Standardization (ISO), 1 rue de Varembe, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

<sup>5</sup> Available from United States Geological Survey (USGS), 12201 Sunrise Valley Drive, Reston, VA 20192, <http://www.usgs.gov>.

**TABLE 1 Summary of Known 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)	White light turbidimeters. Comply with <b>USEPA Method 180.1</b> for low level turbidity monitoring.	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 ISWTR regulations and Standard Method 2130B. Can be used for both low and high level measurement.	Used a white light spectral source. 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
Nephelometric, near-IR turbidimeters, non-ratiometric (FNU)	Complies with <b>ISO 7027</b> . 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.	0–1000	0–40 Regulatory (non-US) 0–1000 other
Nephelometric near-IR turbidimeters, ratio metric (FNRU)	Complies with <b>ISO 7027</b> . 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
Surface Scatter Turbidimeters (NTU)	Turbidity is determined through light scatter from or near the surface of a sample.	Detector centered at 90° relative to the incident light beam. Uses a white light spectral source.	10–10 000	10–10 000
Formazin Back Scatter (FBU)	Not applicable for regulatory purposes. Best applied to high turbidity samples. Backscatter is common with but not all only 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 turbidity.	Uses a white light spectral source (400–680 nm range). Detector geometry is 30 ± 15° 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 turbidity.	Detector is geometrically centered at 180° relative to incident beam (attenuation). Wavelength is 780–900 nm.	20–1000	20–1000 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.	20–1000	20–1000
Nephelometric Turbidity Multibeam Unit (NTMU)	Is applicable to EPA regulatory method GLI Method 2. Applicable to drinking water and wastewater monitoring applications.	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 0–4000 other

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *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 suspended and dissolved matter such as clay, silt, finely divided organic matter, plankton, other microscopic organisms, organic acids, and dyes.

3.2.2 *turbidimeter*—an instrument that measures light scatter, attenuation, or both in a sample and quantitatively converts the light scatter, the attenuation, or both to a displayed value. The location and type and number of detectors used will dictate the relative sensitivity for a typical technology. See **Table 1** for examples of designs.

3.2.3 *reference turbidity standard*—a standard that is synthesized reproducibly from traceable raw materials by a skilled analyst. All other standards are traced back to this standard. The reference standard for turbidity is formazin (see **9.2.2**).

3.2.4 *calibration turbidity standard*—a turbidity standard that is traceable and equivalent to the reference turbidity standard to within defined accuracy, including commercially prepared 4000 NTU Formazin, stabilized formazin (see **9.2.3**), and styrenedivinylbenzene (SDVB) (see **9.2.4**). These standards may be used to calibrate the instrument.

NOTE 1—Calibration standards may be instrument design specific.

NOTE 2—Calibration standards that exceed 10 000 turbidity units are commercially available.

3.2.5 *calibration verification standards*—defined standards used to verify the instrument performance in the measurement range of interest. Calibration verification standards may not be used to adjust instrument calibration, but only to check that the instrument measurements are in the expected range. Included standards are opto-mechanical light scatter devices, gel-like standards, or any other type of stable liquid standard.

NOTE 3—Calibration verification standards may be instrument design specific.

3.2.6 *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 in FNU (Formazin Nephelometric Units).

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

3.2.8 *stray light*—all light reaching the detector other than that which is scattered by the sample. For example: 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.

3.2.9 *seasoning*—the process of conditioning laboratory glassware with the standard to be diluted to a lower value. The process reduces contamination and dilution errors.

3.2.10 *attenuation*—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. Attenuation is inversely proportional to transmitted signal.

$$\text{Attenuated Turbidity} = \text{Absorbed Light} + \text{Scattered Light}$$

NOTE 4—The application of attenuation in this test method is as a distinct means of measuring turbidity. When measuring in the FAU or AU mode, the turbidity value is a combination of scattered (attenuated) plus absorbed light. The scattered light is affected by particle size and is a positive response. The absorption due to color is a negative. The sum of these two entities results in the turbidity value in the respective units.

3.2.11 *surface scatter turbidimeter*—an instrument that determines the turbidity through incident light scatter that occurs at or slightly below the surface of a water sample. The detection angle is still at 90° relative to the incident light beam, but interferences are not as substantial as nephelometric non-ratio measurements.

## 4. Summary of Test Method

4.1 The optical property expressed as turbidity is measured by the scattering effect that constituents within a sample have on light; the higher the quantity of scattered or attenuated incident light, the higher the turbidity. In samples containing particulate material, light scatter and attenuation will vary (1) due to size, shape and composition of the particles in the water, and (2) the wavelength of the incident light.

4.2 This test method is based upon a comparison of the amount of light scattered or attenuated by the sample with the

amount of light scattered or attenuated by a reference suspension. Lower turbidity values are typically determined by a nephelometer, which measures light scatter from a sample in a direction that is at 90° with respect to the centerline of the incident light path. High-level turbidity determination can be performed using many different technologies. It is critical when reporting the measurement, traceability to the type of technology be used. Turbidity measurements are not often consistent among differing technologies.

## 5. Significance and Use

5.1 Turbidity at the levels defined in the scope of this test method are often monitored to help control processes, monitor the health and biology of water environments and determine the impact of changes in response to environmental events (weather events, floods, etc.). Turbidity is often 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 is often accomplished by coagulation, sedimentation, and various levels of filtration. Measurement of turbidity provides an indicator of contamination, and is a vital measurement for monitoring the characteristics and or quality within the sample's source or process.

5.2 This test method does overlap Test Method D 6855 for the range of 1–5 TU. If the predominant measurement falls below 1.0 TU with occasional spikes above this value, Test Method D 6855 may be more applicable. For measurements that are consistently above 1 TU, this test method is applicable.

5.3 This test method is suitable to turbidity such as that found in all waters that measure above 1 NTU. Examples include environmental waters (streams, rivers, lakes, reservoirs, estuaries), processes associated with water pollution control plants (wastewater treatment plants), and various industrial processes involving water with noticeable turbidity. For measurement of cleaner waters, refer to Test Method D 6855.

5.4 The appropriate measurement range for a specific technology or instrument type that should be utilized is at or below 80 % of full-scale capability for the respective instrument or technology. Measurements above this level may not be dependable.

5.4.1 Dilutions of waters are not recommended, especially in the case of samples with rapidly settling particles (that is, sediments). It is recommended that an appropriate instrument design that covers the expected range be selected to avoid the need to perform dilutions.

5.5 Technologies described in this standard may not measure all aspects (absorption and scatter) of a sample. Some of the properties of the water, the suspended material, or both may interfere with the certain measured property of the sample, such as the scattering of light that the particular instrument is measuring.

5.6 Several different technologies are available for use in the measurement of high-level turbidity. Some technologies may be better suited for specific types of samples, depending on the application and measurement criteria. Please refer to Table 1 and Appendix X1 which is a flow chart to help assist in selecting the best technology for the specific application.

5.6.1 When measuring high levels of turbidity the samples will often contain significant interferences such as that from absorbing particles, absorbance in the matrix, and rapidly settling particles. These may have a significant impact on how one measurement technology responds to changes in turbidity. Often times it will be prudent to run a series of linear dilutions to determine if the measured response was expected relative to the dilution. In cases where the response to dilution ratio is linear, the technology may be adequately accounting for the interferences. If the response is not expected, another technology should be considered to determine if a more accurate measurement could be obtained.

5.7 When reporting the measured result, appropriate units should also be attached. The units are reflective of the technology used to generate the measurements. The intention is to provide traceability for the technology used to generate the measured result, and if necessary, provide more adequate comparison to historical data. Section 7 describes technology that each type of traceable reporting units is based.

5.7.1 **Table 1** contains the list of technologies and respective reporting units that will be traceable to that technology.

5.7.1.1 The methods in **Table 1** can be broken down into two distinct groups of designs which are based on the type of incident light source used. These are broad-band white light source or light sources that provide a spectral output in the 400–680 nm range. These include polychromatic light sources, such as those that are necessary to comply with regulatory method **USEPA Method 180.1**, but also can include monochromatic light sources if the respective wavelength falls within the specified range. The second group of instruments uses a near IR monochromatic light source that is in the range of 780 to 900 nm. These designs are distinguishable in the reporting units and will always begin with the letter F.

5.7.1.2 For a specific design that falls outside of these reporting ranges, the turbidity should be reported in turbidity units (TU) with a subscripted wavelength value to characterize the light source that was used. See **7.4.3**.

5.7.1.3 Those designs listed in **Table 1** cover those that were currently identified by the ASTM subcommittee. Future designs that are not covered in this document may be incorporated into a future revision after review by the method subcommittee.

5.7.1.4 See Section 7 for more details regarding instrument designs.

5.7.1.5 Section 16 contains precision and bias data that incorporates the different classifications of technologies. The precision and bias section includes the overall data set of all laboratories and smaller segments of this data set to provide comparisons across distinguishing technological features that are exhibited by those technologies that are represented in this test method.

5.8 This test method covers the measurement of samples collected from waters and analyzed using typical laboratory based or portable-based instruments.

## 6. Interferences

6.1 Bubbles, although they cause turbidity, may result in interferences in measured turbidity as determined by this test method. Bubbles cause a positive interference and color

typically causes a negative interference. Dissolved material that imparts a color to the water may cause errors in pure nephelometric readings, unless the instrument has special compensating features to reduce these interferences. Certain turbulent motions also create unstable reading conditions of nephelometers.

6.2 Color is characterized by absorption of specific wavelengths of light. If the wavelengths of incident light are significantly absorbed, a negative interference will result unless the instrument has special compensating features. Depending on the application color may or may not be considered as an interference. Some instrument designs are intended to remove the effect that color imparts on a turbidity measurement. Other designs do not remove the effects of color.

6.2.1 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.

6.3 Scratches, finger marks, or dirt on the walls of the sample cell may give erroneous readings, especially at lower turbidity levels. Sample cells should be kept scrupulously clean both inside and outside and discarded when they become etched or scratched. The sample cells must not be handled where the light strikes them when positioned in the instrument well.

6.4 Sample cell caps and liners must also be scrupulously clean to prevent contamination of the sample. Seasoning of the sample cells should be performed each time a new sample is measured.

6.5 The optical quality and geometry of the sample cells can also impact results. At all turbidity levels, sample cells that are not optically consistent can result in error. Errors greater than 10 % relative to the turbidity value can be reduced through indexing or replacement of the cells. See Section 16 for additional information.

6.5.1 Sample cells should be optically matched or a single cell should be used to perform calibrations and measurements.

6.6 Particle size and distribution will also impact turbidity and is sensitive to the different types of technologies used. Typically, small particles will more effectively scatter light in the nephelometric direction (at 90° relative to the incident light beam) than larger particles. Overall, however, it is the net aggregate scatter and attenuation of the available incident light by all particles that in the sample that contribute to the measurement.

6.7 The path-length of the sample cell or equivalent will impact the sensitivity of measurements. A shorter path length will extend the range and reduce the interference proportionally. However, use of a shorter path-length will reduce the sensitivity of the measurement.

6.8 Ideally, the same indexed sample cell should be used first for standardization followed by unknown (sample) determination. If this is not possible, then sample cells must be matched. Refer to the instrument manual or the standard's manufacturer for instructions regarding the matching of sample cells.

NOTE 5—Indexing of the sample cell to the instrument well is accomplished by placing a mark on the top of the sample cell and a similar mark on the upper surface of the well so that the sample cell can be placed in the well in an exact position each time.

6.9 Condensation on optical elements or sample cells can lead to severe errors in measurement.

6.10 Rapidly settling particles are also an interference. Particles such as sand can settle rapidly and cause false high or false low turbidity readings. The user of this test method must use care to ensure particles are suspended in solution the instant that the measurement is taken.

**7. Apparatus**

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

7.2 Several technologies for measuring high-level turbidity are discussed in this test method. They include nephelometer-based instruments (see Figs. 1-3), backscatter based instruments (see Fig. 4), and attenuation-based instruments (see Fig. 5). These are all discussed in more detail.

7.2.1 Nephelometers include the Photoelectric Nephelometer, Ratio Photoelectric Nephelometer with single beam design, and ratio photoelectric nephelometer in the dual beam design. The correlation between detector response and increasing turbidity levels is positive.

7.2.2 Backscatter turbidimeters typically employ similar light sources used in the photoelectric photometer but utilize a detection angle that is capable of detecting backscattered light from a sample. The correlation between detector response and increasing turbidity levels is positive.

7.2.3 Attenuation-based turbidimeters employ a detection angle that is 180° relative to the incident light beam.

7.3 The resolution of the instruments should permit detection of differences of at least 1 % of the range in which it is used. See Section 14 for rounding the reporting values of turbidity.

7.3.1 Consult the manufacturer to determine that your instrument meets any of the designs that are discussed in this test method.

*7.4 The Nephelometer:*

7.4.1 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 (USEPA Method 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

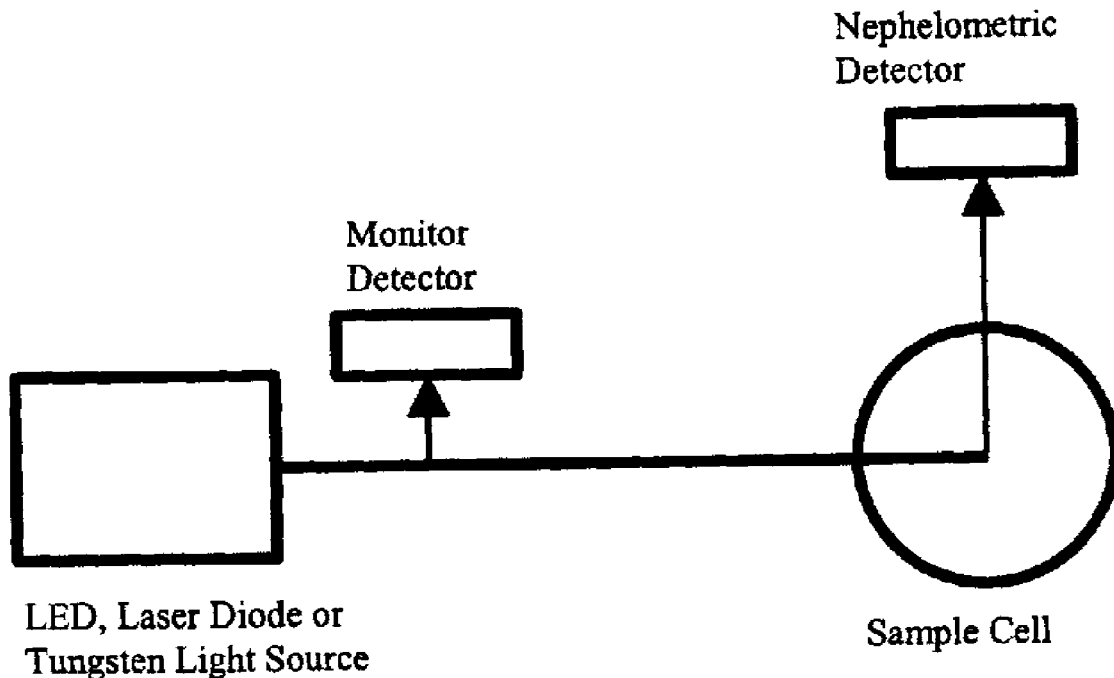


FIG. 1 Typical Nephelometer

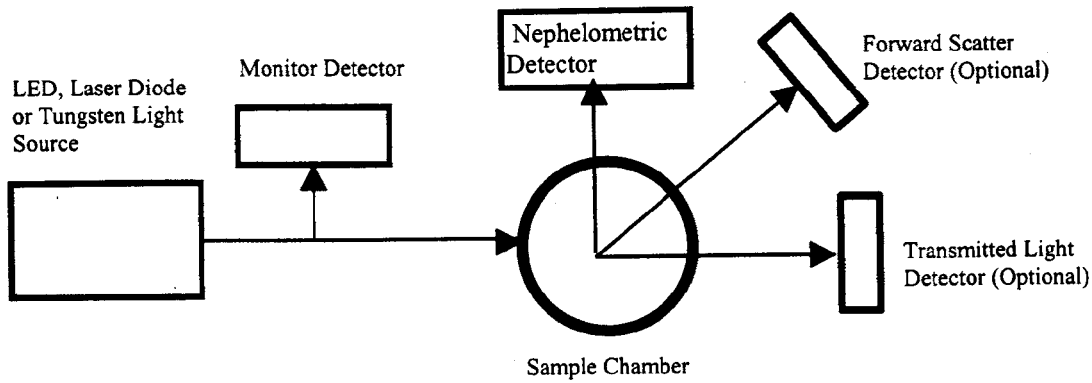


FIG. 2 Ratio Nephelometer (Single Beam Design)

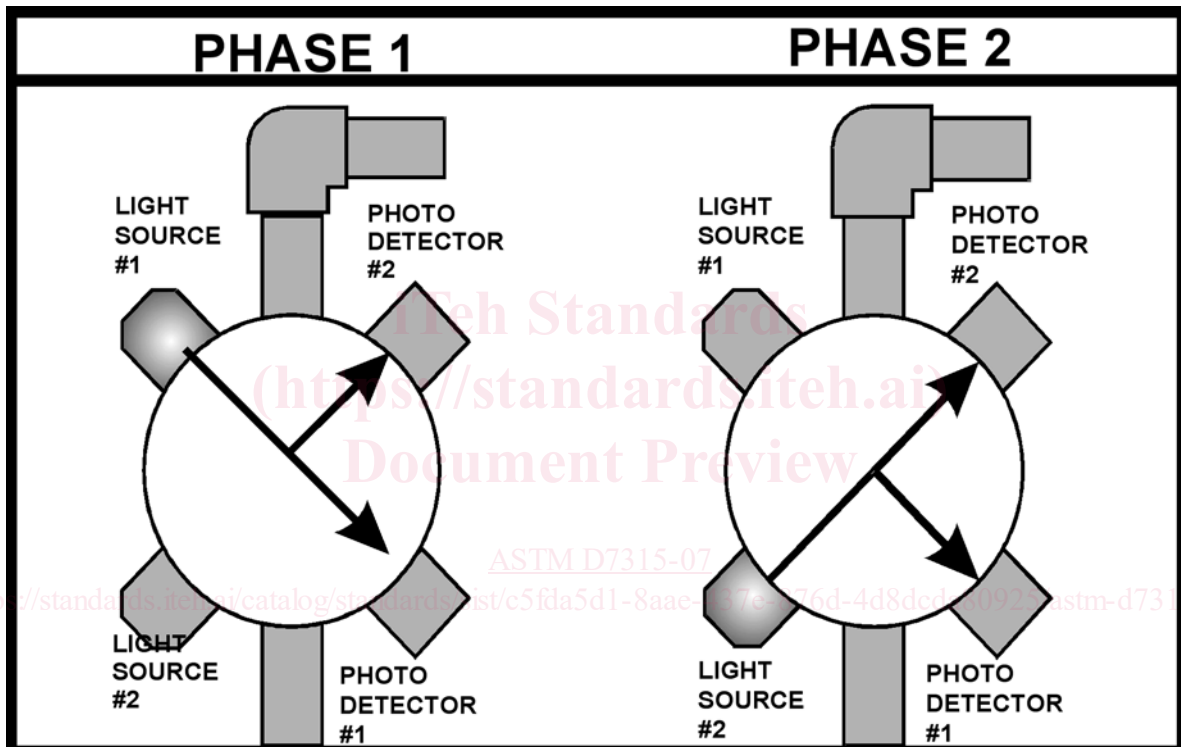


FIG. 3 Ratio Nephelometer (Multiple Beam Design)

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 ±10° 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.

7.4.2 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 following design criteria should be observed (see Fig. 1).

7.4.3 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.

7.5 Ratio Nephelometer:

7.5.1 Ratio 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 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, instrument noise, sample color, or combinations thereof. 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 (USEPA Method 180.1). LEDs and laser diodes in

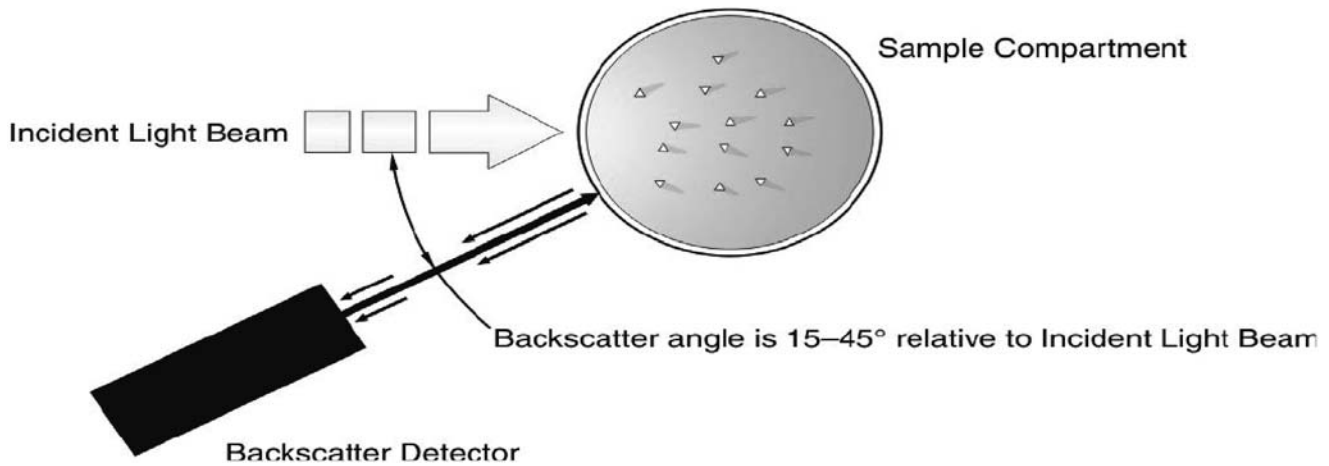


FIG. 4 Geometric Diagram of a Backscatter Measurement

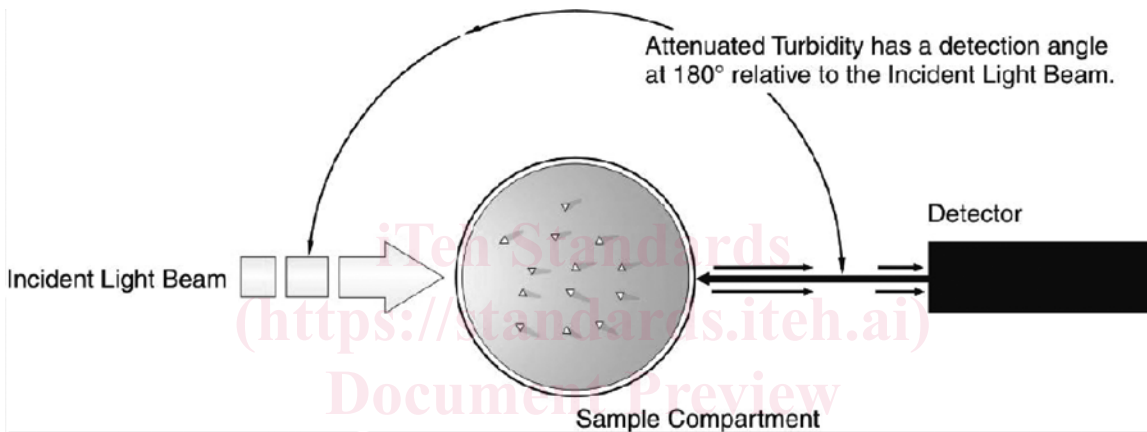


FIG. 5 Geometric Design of an Attenuated Measurement

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 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 7.4.1.

7.5.2 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 following design criteria should be observed (see Figs. 2 and 3).

7.5.3 Report in the appropriate units using Table 1 as guidance.

7.5.3.1 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.

7.6 Backscatter Turbidimeters:

7.6.1 The instrumentation contains a light source that meets or exceeds the criteria specified in 7.4.1 for illumination of the sample.

7.6.2 The response curve of the detector should be such that it overlaps the output of the light source.

7.6.3 The detection angle for backscatter is to be set at  $30 \pm 15^\circ$  relative to the centerline of the incident light beam. See Fig. 4.

7.6.4 When reporting turbidity, report in units that best fit the light source and detector in Table 2. Report in BU (white

TABLE 2 Reporting of Results for High Level Static Turbidity Measurements

NOTE—New developments in technologies may allow instruments to extend beyond range.

Measured Value In Appropriate Units	Report to Nearest
1.0* < 9.9	0.1
10 < 99	1
100–999	5
1000 <	50

light source) or FBU (if a 780–900 nm light source was used).

#### 7.7 Attenuation-Based Turbidimeters:

7.7.1 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.

7.7.2 The detector response curve should overlap the incident light source.

7.7.3 The detection angle for attenuation is to be set at 180° relative to the centerline of the incident light beam. See Fig. 5.

7.7.4 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).

7.8 Sample Cells (if used with typical benchtop or portable instruments):

7.8.1 The sample cells used in calibration and sample measurement must be the following:

7.8.1.1 Clear, colorless glass or optically clear plastic, be kept scrupulously clean, both inside and out, and discarded when it becomes etched or scratched (see non-mandatory Appendix X3 for sample cell cleaning procedure).

7.8.1.2 Index marked so that repeated exact placements into the instrument sample cell compartment for measurement can be made. The location of the index mark should be such that the window of movement is less than 10° of rotation where the measurement is consistent. See 11.4.2.1.

7.8.1.3 Cells should be handled where the light path does not pass during measurement. Provision should be made in design to give the sample cell a proper place in which to handle the cell during calibration or sample measurement procedure.

7.8.1.4 The outside surface of a glass sample cell may be oiled, using silicone oil and a soft cloth, or a lint free laboratory tissue to minimize imperfections that could cause light to scatter off the surface of this sample cell, or wiped with alcohol. See the manufacturer's recommendations for sample cell preparation.

7.8.1.5 Preferably matched sample cells that provide consistent readings to within 10 % on filtered DI water should be used.

#### 7.9 Sample Chambers:

7.9.1 For those instruments not using sample cells, the sample is placed directly into the sample chamber. For those units, the sample chamber must be the following:

7.9.1.1 Sample chambers should be kept scrupulously clean. Scratches, fingerprints and dirt on the walls of the sample chamber may give erroneous results. See the manufacturer's recommendations for sample chamber maintenance.

7.9.1.2 Sample chambers should be designed in such a way as to negate any influence from external light sources, and to minimize stray light interference with readings.

## 8. Purity of Reagents

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 MSDS 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 h of use to reduce background turbidity. Type III water is also acceptable (See Specification D 1193). 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:

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 quantitative 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 (StablCal®) and styrenedivinylbenzene (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.

<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.



9.2.2.1 Quantitatively transfer 5.000 g of reagent grade hydrazine sulfate (99.5%+ purity) ( $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{SO}_4$ ) into approximately 400 mL of dilution water (see 8.2) contained in a 1-L 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 g 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  $\mu\text{m}$  filter into a clean flask.

9.2.2.3 Quantitatively transfer the filtered hexamethylenetetramine 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 min, and no more than 10 min.

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 h at  $25 \pm 1^\circ\text{C}$ . The 4000 NTU Formazin suspension develops during this time.

NOTE 11—This suspension, if stored at  $20\text{--}25^\circ\text{C}$  in amber polyethylene bottles, is stable for 1 year; it is stable for 1 month if stored in glass at  $20\text{--}25^\circ\text{C}$ .

9.2.3 Stabilized formazin turbidity standards (StabCal®) 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.

NOTE 12—Sealed or solid samples should not be used to standardize turbidimeters for the turbidity measurement of water or waste; they may only be used for calibration verification. These two methods (sealed or solid examples) neglect the zeroing out of the sample cell prior to making water measurement in the cell.

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 X3). Invert 4000 NTU stock suspension 25 times to mix (1 s 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 s inversion cycle) and immediately pipet a volume of 40 NTU standard. All glassware shall be seasoned (see Appendix X3).

NOTE 13—The instructions below result in the preparation of 200 mL of a 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 s 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  $\geq 40$  NTU have a useful life of one week. All labware shall be seasoned (See Appendix X3). Use Class A glassware that has been cleaned per 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 styrene-divinylbenzene (SDVB), have a specific turbidity value and accuracy. Such standards must be traceable to the reference turbidity standard.

## 10. Safety

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

## 11. Sampling and Sample Preservation

11.1 *Collection of Sample*—Collect the sample in accordance with the applicable standard, Specification and Practices. See Guide D 4411 for the collection of samples in moving waters.

11.2 *Storage of Sample*—Analyze the sample immediately. Do not store the sample.

11.3 *Sample Handling*—Samples should be measured expeditiously after collection to prevent changes in particle characteristics due to temperature changes and settling. Temperature can effect particles either by changing their behavior or creating new particles, if precipitates are created. Dilution water may dissolve particles or change their characteristics. Operators should draw samples only when turbidimeters are ready for operation. Do not draw a sample and allow it to sit while the turbidimeter is being readied.

#### 11.4 *Other Important Sampling Techniques:*

11.4.1 Minimize agitation of samples as particles can be altered or air may be entrained into the sample. Gentle agitation or swirling is recommended to reduce particle settling. Swirling may not be appropriate for rapidly settling particles.

11.4.2 Sample cells should only be used with the instrumentation for which they were intended.

11.4.2.1 Prior to each measurement inspect the filled sample cell and ensure that there are no bubbles in the sample, and that the cell is free of scratches.

NOTE 14—If degassing is necessary, a non-intrusive procedure for removing bubbles can be used. Examples include the application of a vacuum or the use of an ultra-sonic bath. Caution must be exercised not to alter the composition of the samples.

11.4.2.2 Sample cells should be evaluated with a low turbidity water (after cleaning) to determine if cells remain matched. If the evaluation determines that a cell is corrupted, discard the cell. This check should be performed on a weekly basis.

11.4.2.3 If a sample cell's condition is questionable, discard the cell and replace with a new sample cell.

#### 11.5 *Sample Preparation for Measurement:*

11.5.1 Rinse the clean sample cell or chamber twice with the sample that is to be measured, and discard the rinsings.

11.5.2 Fill the sample cell or chamber to a level at which the top air/liquid interface will not interfere with the subsequent reading. Follow manufacturer recommendations as to sample cell or chamber filling.

11.5.3 After the sample cell is filled, use a lint-free tissue to remove all traces of dirt or fingerprints. Tissue alone does not clean dirty sample cells and one of the common nonabrasive glass cleaners may be necessary.

11.5.4 The cleaned sample cell is handled by its very top and placed in an indexed manner in the instrument.

## 12. Calibration and Calibration Verification

12.1 Determine if the instrument requires any maintenance such as cleaning the sample cell or sample chamber, etc. Follow the manufacturer's instructions for any required instrument maintenance prior to calibration.

### 12.2 *Calibration:*

12.2.1 Follow the manufacturer's instructions for calibration and operation. Calibrate the instrument to assure proper operation for the range of interest with appropriate standards. See standard or instrument manufacturers for the preparation and use of calibration or verification standards.

12.2.2 Formazin-based calibration standards should be re-suspended through inversion (1 s inversion cycle) 25 times followed by a 2 to 10 min wait to allow for bubble removal. Standards of 40 NTU or below will remain suspended for up to 30 min; standards greater than 40 NTU may require more frequent re-suspension.

NOTE 15—A Calibration Turbidity Standard is 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 styrene-divinylbenzene (SDVB). These standards may be used to calibrate the instrument.

12.2.3 Verify instrument calibration accuracy in the expected measurement area using a calibration verification standard. The calibration verification standard used should have a defined value with known accuracy. The calibration verification standard should allow the instrument to perform to within its defined performance specifications. Verification should be conducted at timely intervals between calibrations. (Consult instrument manufacturer's recommendations, standard's manufacturers recommendations, or both for guidance associated with verification methods and devices.)

NOTE 16—Some regulatory agencies may have specific requirements or guidelines regarding the calibration of turbidimeters. Consult those authorities if reporting to specific regulatory agencies.

12.2.4 In case of verification failure clean the instrument to reduce stray light levels or contamination. Follow with a recalibration (instrument adjustment) according to manufacturer's calibration instructions, using a calibration standard (see 3.2.4), or at a minimum on a quarterly basis.

12.2.5 Close adherence to the calibration procedure and to the rinsing/seasoning techniques is very important to ensure data quality.

12.2.6 Proper sample handling and preparation procedures must be followed to assure data quality (see Section 11).

12.2.7 Proper care and maintenance of sample cells and sample chambers must be performed in a timely manner (see 7.7).

## 13. Procedure for the Measurement of High-Level Static Turbidity

13.1 Identify the technology to be used and determine the most appropriate turbidity unit. See Table 1 and Appendix X2 for guidance and application of the technologies offered in this test method.

13.2 *Turbidity Greater than 1.0 NTU*—Gently invert the sample several times (1 s inversion cycle) to thoroughly suspend any solids. Rinse a clean sample cell several times with sample. Fill the sample cell with sample and cap the cell. Clean outside surfaces of the sample cell (see non-mandatory Appendix X3). Invert again and immediately place the sample cell into the instrument at the index mark and measure at a consistent distinct time interval, not to exceed 15 s, after inserting the vial. Repeat a minimum of 2 more times with new sample. Average the values to generate the reported result.

NOTE 17—Care must be taken to ensure the sample does not settle when transferring from the sample container to a sample vial. See 13.5.1.

NOTE 18—If the instrument has a long measurement-averaging interval, the interval should be as short as possible to minimize error due to particle