

Designation: D7725 - 17

Standard Test Method for the Continuous Measurement of Turbidity Above 1 Turbidity Unit (TU)¹

This standard is issued under the fixed designation D7725; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the on-line and in-line determination of high-level turbidity in water that is greater than 1.0 turbidity units (TU) in municipal, industrial and environmental usage.

1.2 In principle, there are three basic applications for on-line measurement set ups. This first is the slipstream (bypass) sample technique. For the slipstream sample technique a portion of sample is transported out of the process and through the measurement apparatus. It is then either transported back to the process or to waste. The second is the in-line measurement where the sensor is brought directly into the process (see Fig. 8). The third basic method is for in-situ monitoring of sample waters. This principle is based on the insertion of a sensor into the sample itself as the sample is being processed. The in-situ use in this test method is intended for the monitoring of water during any step within a processing train, including immediately before or after the process itself.

1.3 This test method is applicable to the measurement of turbidities greater than 1.0 TU. The absolute range is dictated by the technology that is employed.

1.4 The upper end of the measurement range is left undefined because different technologies described in this test method can cover very different ranges of turbidity.

1.5 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.

1.5.1 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.

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

1.7 This test method was tested on different waters, 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.

1.8 Those samples with the highest particle densities typically prove to be the most difficult to measure. In these cases, the process monitoring method can be considered with adequate measurement protocols installed.

1.9 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.10 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Refer to the MSDSs for all chemicals used in this procedure.

1.11 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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

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TABLE 1 Technologies for Measuring Turbidity Greater Than 1 TU That Can be Used for In-Line or On-Line Applications

Design and Reporting Unit	Prominent Application	Key Design Features	Typical Instrument Range	Suggested Application
Nephelometric Non-Tatio (NTU)	White light turbidimeters comply with EPA 180.1 for low-level turbidity monitoring.	Detector centered at 90 degrees relative to the incident light beam. Uses a white light spectral source.	0.012 to 40 NTU	Regulatory reporting of clean water
Ratio White Light Turbidi- meters (NTRU)	Complies with Interim Enhanced Surface Water Treatment Rule (ISWTR) regulations and Standard Method 2130B. Can be used for both low- and high-level measure- ment.	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 read- ings to generate the turbidity read- ing.	0.012–10 000 NTRU	Regulatory reporting of clean water
Formazin Nephelometric, Near-IR Turbidimeters, Non-Ratiometric (FNU)	Complies with ISO 7027. The wave- lengthis less susceptible to color in- terferences. Applicable for samples with color and good for low-level monitoring.	Detector centered at 90 degrees relative to the incident light beam. Uses a near-IR (780–900 nm) monochromatic light source.	0.012–1 000 FNU	0-40 FNU ISO 7027 regulatory reporting
Formazin 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 de- tector centered at 90°. Other detec- tors located at other angles. An in- strument algorithm uses a combination of detector readings to generate the turbidity reading.	0.012–1 000 FNU	0-40 FNRU ISO 7027 regulatory reporting
Surface Scatter Turbidi- meters (SSU)	Turbidity is determined through light scatter from a defined volume be- neath the surface of a sample. Appli- cable for reporting for EPA compli- ance monitoring.	Detector centered at 90 degrees relative to the incident light beam. Uses a "white light" spectral source.	0.012–10 000 FNRU	10-10 000 SSU
Formazin Nephelometric Turbidity Multibeam unit (FNMU)	Is applicable to EPA regulatory method GLI Method 2. Applicable to drinking water and wastewater moni- toring applications.	Detectors are geometrically centered at 0° and 90°. An instrument algo- rithm uses a combination of detector readings, which may differ for tur- bidities varying magnitude.	0.012 to 4000 NTMU	0 to 40 NTMU Reporting for EPA and ISO compliane
Formazin Attenuation Unit (FAU)	Compliance Reporting for ISO 7027 for samples that exceed 40 units.	Uses a near-IR light source at 860 ± 30 nm and the detector is 0 degrees relative to the centerline of the incident light beam. The measurement is an attenuation measurement.	10-10 000+ FAU	100–10 000+ FAU Report- ing for ISO 7027 for levels in excess of 40 units
Attenuation Unit (AU)	Not applicable for regulatory pur- poses. Best applied for samples with high-level turbidity.	Uses a white light spectral source (400–680 nm range). Detector ge- ometry is 0° relative to the incident light beam.	a6-fi ad02bd1689/ast 10-10 000+ AU	100–10 000+ AU
Formazin Back Scatter (FBU)	Not applicable for regulatory pur- poses. 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 between 90 and 180° relative to the incident light beam.	10 000+ FBU	10 000 FBU
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-degrees relative to the centerline of the incident light beam.	The technology is sensitive to tur- bidities as low as 1 TU. The ratio technology helps to compensate for color interference and fouling.	The measurement of ambient waters such as streams, lakes, and rivers. The range is typically from about 1–800 FSRU, depending on the manufacturer.	Forward Scatter Ratio Unit (FSRU)

2. Referenced Documents

2.1 ASTM Standards:²

D1129 Terminology Relating to Water

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

- D3370 Practices for Sampling Water from Closed Conduits D3864 Guide for On-Line Monitoring Systems for Water
- Analysis D6698 Test Method for On-Line Measurement of Turbidity
- Below 5 NTU in Water D7315 Test Method for Determination of Turbidity Above 1
- Turbidity Unit (TU) in Static Mode

2.2 Other Publications:

EPA 180.1 Methods for Chemical Analysis of Water and Wastes, Turbidity³

GLI Method 2 Great Lakes Instruments (GLI) — Turbidity⁴

ISO 7027 Water Quality — Determination of Turbidity⁵

Standard Method 2130B Standard Methods for the Examination of Water and Wastewater⁶

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *attenuation-detection angle, n*—the angle that is formed between the incident light source and the primary detector, and that is at exactly 0-degrees.

3.2.1.1 *Discussion*—This is typically a transmission measurement.

3.2.2 backscatter-detection angle, *n*—the angle that is formed between the incident light source and the primary detector, and that is greater than 90-degrees and up to 180-degrees.

3.2.3 *calibration turbidity standard, n*—a turbidity standard that is traceable and equivalent to the reference turbidity standard to within statistical errors; calibration turbidity standards include commercially prepared 4000 NTU formazin, stabilized formazin, and styrenedivinylbenzene (SDVB).

3.2.3.1 *Discussion*—These standards may be used to calibrate the instrument. Calibration turbidity standards may be instrument specific.

3.2.4 *calibration-verification standards*, *n*—defined standards used to verify the accuracy of a calibration in the measurement range of interest.

⁶ Available from American Public Health Association (APHA), 800 I St., NW, Washington, DC 20001, http://www.apha.org.

3.2.4.1 *Discussion*—These standards may not be used to perform calibrations, only calibration verifications. Included verification standards are opto-mechanical lightscatter devices, gel-like standards, or any other type of stable-liquid standard. Calibration verification standards may be instrument specific.

3.2.5 *detection angle, n*—the angle formed with its apex at the center of the analysis volume of the sample, and such that one vector coincides with the centerline of the incident light source's emitted radiation and the second vector projects to the center of the primary detector's view.

3.2.5.1 *Discussion*—This angle is used for the differentiation of turbidity-measurement technologies that are used in this test method.

3.2.6 *forward-scatter-detection angle, n*—the angle that is formed between the incident light source and the primary detector, and that is between 0 and 90-degrees.

3.2.6.1 *Discussion*—Most designs will have an angle between 10 and 45 degrees.

3.2.7 *nephelometric-detection angle, n*—the angle that is formed between the incident light source and the detector, and that is at 90-degrees

3.2.8 *nephelometric-turbidity measurement, n*—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.

3.2.8.1 *Discussion*—Units are NTU (nephelometric turbidity units). When ISO 7027 technology is employed units are FNU (formazin nephelometric units).

3.2.9 ratio-turbidity measurement, n—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.

3.2.10 *reference-turbidity standard*, *n*—a standard that is synthesized reproducibly from traceable raw materials by the user.

3.2.10.1 *Discussion*—All other standards are traced back to this standard. The reference standard for turbidity is formazin.

3.2.11 *seasoning*, *n*—the process of conditioning labware with the standard that will be diluted to a lower value.

3.2.11.1 *Discussion*—The process reduces contamination and dilution errors. See Appendix X2 for suggested procedure.

3.2.12 *slipstream*, *n*—an on-line technique for analysis of a sample as it flows through a measurement chamber of an instrument.

3.2.12.1 *Discussion*—The sample is transported from the source into the instrument (for example a turbidimeter), analyzed, and then transported to drain or back to the process stream. The term is synonymous with the terms "on-line instrument" or "continuous-monitoring instrument."

3.2.13 *stray light*, *n*—all light reaching the detector other than that contributed by the sample.

3.2.14 surface-scatter detection, n—a turbidity measurement that is determined through the detection of light scatter caused by particles within a defined volume beneath the surface of a sample.

3.2.14.1 Discussion-Both the light source and detector are

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

³ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

⁴ Available from Hach Company, P.O. Box 389, Loveland, Colorado 80539, https://www.hach.com.

⁵ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

positioned above the surface of the sample. The angle formed between the centerline of the light source and detector is typically at 90-degrees. Particles at the surface and in a volume below the surface of the sample contribute to the turbidity reading.

3.2.15 *turbidimeter*, n—an instrument that measures light scatter caused by particulates within a sample and converts the measurement to a turbidity value.

3.2.15.1 *Discussion*—The detected light is quantitatively converted to a numeric value that is traced to a light-scatter standard. See Test Method D7315.

3.2.16 *turbidity*, *n*—an expression of the optical properties of a sample that cause light rays to be scattered and absorbed rather than transmitted in straight lines through the sample.

3.2.16.1 *Discussion*—Turbidity of water is caused by the presence of matter such as clay, silt, finely divided organic matter, plankton, other microscopic organisms, organic acids, and dyes.

4. Significance and Use

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

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

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

4.4 When reporting the measured result, appropriate units should also be reported. The units are reflective of the technology used to generate the result, and if necessary, provide more adequate comparison to historical data sets.

4.4.1 Table 1 describing technologies and reporting results. Those technologies listed are appropriate for the range of measurement prescribed in this test method are mentioned, though others may come available. Fig. X3.1 from Appendix X3 contains a flowchart to assist in technology selection.

4.4.2 For a specific design that falls outside of these reporting ranges, the turbidity should be reported in TU with a subscripted wavelength value to characterize the light source that was used.

4.4.3 Ratio white light turbidimeters are common as bench top instruments but not as a typical process instrument. However, if fitted with a flow-cell they meet the criteria of this test method.

5. Safety

5.1 Wear appropriate personal protection equipment at all times.

5.2 Follow all relevant safety guidelines.

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

5.4 Refer to all material safety data sheets (MSDSs) prior to preparing or using standards and before calibrating or performing instrument maintenance.

6. Interferences

6.1 Bubbles, 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.

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 compensation features. Depending on the application color may or may not be considered 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.

6.2.2 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.

6.3 *Absorbing Particles*—Particles such as carbon, anthracite, fire residue will absorb incident light and bias readings to be negative.

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

6.5 Sample cell caps and liners (if applicable for process turbidimeters), and sample chambers must also be scrupulously clean to prevent contamination of the sample. Seasoning of the sample cells or sample chamber should be performed each time a new sample is measured.

6.6 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 14.2 for additional information.

6.6.1 Sample cells that are used in process instruments should be optically matched or a single cell should be used to perform calibrations and measurements.

6.7 Particle size distribution can be considered a interference but is typically considered an inherent part of the sample. The particle-size distribution in a sample, 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.

6.8 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.

6.8.1 Ideally, the same indexed sample cell should be used first for standardization and then for measurement in process instruments. If this is not possible, then sample cells must be matched. Refer to the instrument manual or the instrument manufacturer for instructions regarding the matching of sample cells.

NOTE 1—Indexing of the sample cell to the instrument chamber is accomplished by placing a mark on the top of the sample cell and a similar mark on the upper surface of the sample chamber so that the sample cell can be placed in repeatable position each time.

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

6.10 Fouling of optical elements or windows will cause severe errors in measurement. Inspection of sample chambers for fouling should be conducted in a timely manner.

6.11 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 the measurement it taken.

6.12 Certain turbulent motions also create unstable reading conditions of nephelometers.

7. Apparatus

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

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

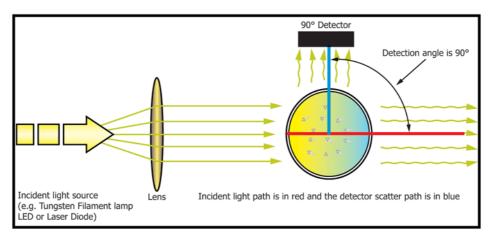
7.3 The instrument design should allow for effective flow passage so that the settling of particulate materials does not occur in the measurement chamber.

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

7.5 The instrument should permit detection of turbidity differences of 0.10 TU or less in waters with turbidity between 1.0 and 5.0 TU (see 13.1).

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

7.6.1 *The Photoelectric Nephelometer*—(see Fig. 1). This instrument uses a light source for illuminating the sample and a single photo-detector with a readout device to indicate the intensity of light scattered at 90° to the centerline of the path of the incident light. The photoelectric nephelometer should be 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 should be a Tungsten lamp operated at a color temperature between 2200 and 3000 K. Light emitting diodes (LEDs) and laser diodes in defined wavelengths ranging from 400–900 nm may also be used. If LEDs or laser diodes are used, then the LED or laser diode should be coupled with a monitor detection device to achieve a consistent energy output. The total distance traversed by incident light and scattered light within the sample is not to



Note 1—Monitor detector is optional (not shown) and its use is typically with LED light sources. FIG. 1 The Photoelectric Nephelometer

exceed 10 cm. Angle of light acceptance to the detector: centered at 90° to the centerline of the incident light path and not to exceed $\pm 10^{\circ}$ from the 90° scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used.

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

7.6.2 Ratio Photoelectric Nephelometer-(see Fig. 2 for single beam design; see Fig. 3 for multiple beam design). This instrument uses the measurement derived through the use of a nephelometric detector that serves as the primary detector and one or more other detectors used to compensate for variation in incident light fluctuation, stray light, instrument noise, or sample color. As needed by the design, additional photodetectors may be used to sense the intensity of light scattered at other angles. The signals from these additional photodetectors may be used to compensate for variations in incident light fluctuation, instrument stray light, instrument noise, sample color, or combination thereof. The ratio photoelectric nephelometer should be designed so that minimal stray light reaches the detector(s), and should be free from significant drift after a short warm-up period. The light source should be a tungsten lamp, operated at a color temperature between 2200 and 3000 K. LEDs and laser diodes in defined wavelengths ranging from 400 to 900 nm may also be used. If an LED or a laser diode is used in the single beam design, then the LED or laser diode should be coupled with a monitor detection device to achieve a consistent energy output. The distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. The angle of light acceptance to the nephelometric detector(s) should be centered at 90° to the centerline of the incident light path and should not exceed $\pm 10^{\circ}$ from the scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used. The instrument calibration (algorithm) must be designed such that the scaleable reading is from the nephelometric detector(s), and other detectors are used to compensate for instrument variation described in 3.2.8.

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

7.7 Surface Scatter Turbidimeters—Surface scatter turbidimeters determine the turbidity through the scatter of light from a defined volume beneath the surface of a flowing sample stream. The incident light strikes the surface of a sample at an angle and the detector of scattered light is also at a different angle but in the same plane with the incident light source. The detection angle is 90 degrees relative to the centerline of the incident light beam, prior to it striking the surface of the sample. Surface scatter turbidimeters have a high operating range and allow for high flow rates (see Fig. 4).

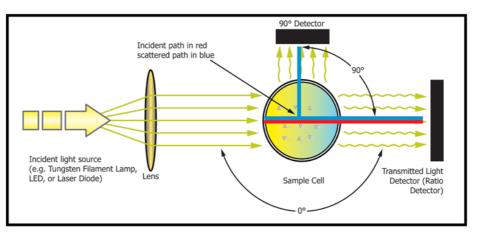
7.8 Formazin Backscatter Turbidimeters—This technology utilizes a near-IR monochromatic light source in the 780-900 nm range. The detector geometry is any angle between 90° and 180° relative to the incident light beam (see Fig. 5).

7.9 Forward Scatter Technologies—This technology encompasses a single, solid-state light source and either a single detector or multiple detectors (ratio). The detection angle for the forward scatter detector is greater than 0-degrees but less than 90-degrees relative to the centerline of the incident light beam. A second ratio gdetector may be incorporated into some designs (see Fig. 6).

8. Purity of Reagents

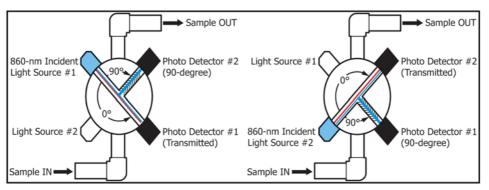
8.1 ACS grade chemicals of high purity (99+ %) shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may 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 2—Refer to product MSDS for possible health exposure concerns.

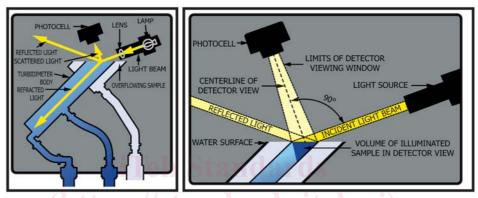


Note 1—The monitor detector (not shown) is optional and it typically used with LED light sources. FIG. 2 The Ratio Photoelectric Turbidimeter

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NOTE 1—Incident light path is in red and scattered light paths are in blue. FIG. 3 Multiple Beam Design Utilizes Two Detectors and Two Light Sources



NOTE 1—Left figure displays the application of the technology and sample flow. Right figure displays the measurement technology for surface scatter detection. (Photos courtesy of Hach Company, Loveland, Colorado.)



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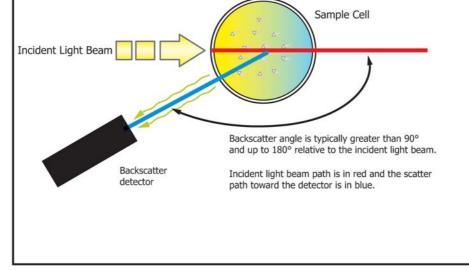


FIG. 5 Backscatter Measurement Design

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

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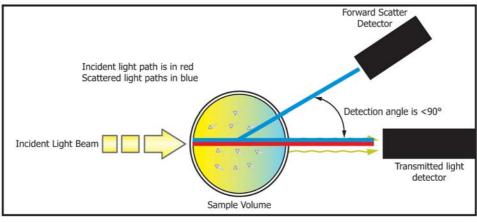


FIG. 6 Forward Scatter Design

9. Reagents

9.1 Reagent, dilution, and final rinsing water, see 8.2.

9.2 Turbidity Standards:

Note 3—A standard with a turbidity of 1.0 NTU is the lowest formazin turbidity standard that should be produced on the bench. Preparation of formazin standards shall be performed by skilled laboratory personnel with experience in quantitative analysis. Close adherence to the instructions within this section is required in order to accurately prepare low-level turbidity standards.

9.2.1 Equivalent, commercially-available, calibration standards may be used. These standards, such as stabilized formazin and SDVB, have a specified turbidity value and accuracy. Such standards must be referenced (traceable) to formazin. Follow specific manufacturer's calibration procedures.

Note 4—All volumetric glassware must be scrupulously clean. The necessary level of cleanliness can be achieved by performing all of the following steps: washing glassware with laboratory detergent followed by 3 tap water rinses; then rinse with portions of 1:4 HCl followed by at least 3 tap water rinses; finally, rinse 3 times with rinse water as defined in 8.2. Reference formazin turbidity standard (4000 NTU) is synthesized on the bench.

9.2.1.1 Dissolve 5.000 g of ACS 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-L Class A volumetric flask.

9.2.1.2 Dissolve 50.000 g of ACS grade hexamethylenetetramine (99 %+ purity) in approximately 400 mL of dilution water (see 8.2) contained in another flask. Filter this solution through a 0.2-mm filter.

9.2.1.3 Quantitatively pour the filtered hexamethylenetetramine solution 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 five minutes, and no more than ten minutes.

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

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

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

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

9.2.4 Formazin Turbidity Suspension, Standard (40 NTU)— All labware shall be seasoned (see Appendix X2). Invert 4000-NTU stock suspension 25 times to mix (one 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.

9.2.5 Dilute Formazin Turbidity Suspension Standard (1.0 NTU)—Prepare this standard dilution daily by inverting the 40-NTU stock suspension 25 times to mix (one second inversion cycle) and immediately pipetting a volume of the 40.0-NTU standard (9.2.4). All labware shall be seasoned (see Appendix X2).

Note 5—The instructions below result in the preparation of 200 mL of 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.

9.2.5.1 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.

9.2.5.2 Using the cleaned glassware, pipette 5.00 mL of mixed 40.0 NTU formazin suspension (9.2.4) into the 200-mL flask and dilute to volume with the dilution water. Stopper and invert 25 times to mix (one second inversion cycle). The turbidity of this prepared standard is 1.0 NTU.

9.2.6 Miscellaneous Dilute Formazin Turbidity Suspension Standard—Prepare all turbidity standards with values below 40.0 NTU daily. All labware shall be seasoned (see Appendix X2). Standards with values above 40.0 NTU have a useful life of one week. Use Class A glassware that has been cleaned in accordance with the instructions in 9.2.5.1 and prepare each dilution by pipetting the volume of 40 NTU (9.2.4) into a 100-mL volumetric flask and diluting to mark with dilution water (8.2). For example, prepare 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.

10. Instrument Installation, Sample Lines and Sampling

Note 6—In principle there are three sampling methods for on-line measurement set ups: slipstream (bypass), in-line, and in-situ. For the slipstream sampling method, a sample is continuously transported out of the process and through the measurement apparatus, and is then either transported back to the process or to waste. For the in-line sampling method, the sensor is brought directly into the process (see Fig. 7). For the in-situ sampling method, the sensor is placed directly into a sample that is in the environment. The in-situ does not measure a sample that is in the process of being transported (that is, such as through a pipe).

10.1 Slipstream Sample Technique:

10.1.1 *Instrument Installation*—Proper location of the sensor and the instrument will help assure accurate results. Assuring that the sensor sees a flowing, bubble free and representative sample is essential for accurate results. Refer to the instrument manufacturer for proper instrument set-up and installation; also see Practices D3370.

10.1.1.1 Locate the sensor as close to the sample location as possible to minimize sample response time. Additionally, locate the instrument for safe, easy access for maintenance and calibration. The location must also provide adequate flow at a rate that particulate settling will not take place in the sample lines, instrument, or drain lines.

10.1.1.2 Locate the instrument so external interferences such as vibration, ambient light, humidity, and extreme conditions are minimized.

10.1.1.3 Position the instrument so it is level and stable to ensure the sample stream is consistent and adequate over long periods of time.

10.1.2 *Sample Lines*—Refer to the instrument manufacturer for recommended sampling procedures for the respective instrument.

10.1.2.1 Sample inlet lines should be a minimum of 4-mm inner diameter, rigid or semi-rigid tubing to allow easy passage of large particles and to minimize the possibility of air lock.

10.1.2.2 Examples of tubing that can be used for sample lines include but are not limited to: polyethylene, nylon, polypropylene, or TFE-fluorocarbon–lined tubing.

10.1.2.3 Soft or porous tubing that could harbor the growth of micro-organisms or contribute turbidity to the sample should not be used.

10.1.3 Sampling:

10.1.3.1 A sample tap should project into the center of the pipe to minimize interference from air bubbles or pipeline bottom sediment. See Fig. 7 for proper sample taps or review instrument manual.

10.1.3.2 Run sample lines directly from the sample point to the turbidimeter sensor to minimize sample flow lag time (response time) or refer to instrument manual.

10.1.3.3 Adjust the flow rate to minimize particle fallout in the sample lines while maximizing bubble removal so bubbles are not carried through the sensor or refer to instrument manual. The best practice is to determine the maximum allowable sample flow in which bubble removal is still effective. This maximum flow will provide the best condition to prevent sample settling.

NOTE 7—There will be some instances where the sample flow rate will be inadequate in the prevention of particle settling. In such cases, a different technique should be used that allows for more rapid passage of sample through the instrument. Or, the user should consider an in-situ measurement technique.

Refer to the instrument installation procedures from the manufacturer for optimization of sample flow rates through the instrument.

10.1.4 The use of either internal or external bubble removal devices (bubble traps) prior to performing measurement of the sample is recommended. Reference Practices D3370 and Guide D3864.

10.1.4.1 When using bubble removal devices, flow must be sufficient to prevent particulate settling within these devices. If particle settling cannot be prevented, then the bubble removal device should be abandoned.

10.2 In-line Measurement Technique: ____7725_1

10.2.1 The principal set up for an in-line turbidity measurement is shown below.

10.2.2 For proper set-up and installation of sensor and transmitter refer to the instrument manufacturer. Some general recommendations for the installation should be followed:

10.2.2.1 The sensor should be mounted into process lines so that the sample flow is consistent and adequate to minimize interference from air bubbles or pipeline bottom sediment.

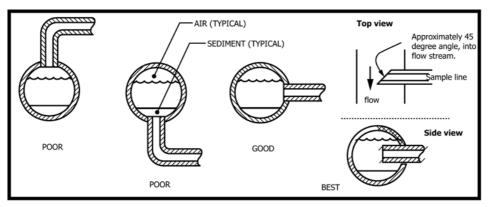


FIG. 7 Illustration of Proper and Improper Sampling Techniques

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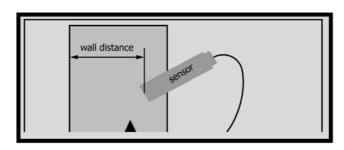


FIG. 8 Principal Set-Up for In-line Turbidity Measurement

Avoid dead spots in flow or areas where the flow through the line slows significantly.

10.2.2.2 Install sensor surface at an angle with respect to medium flow so that flow increases self cleaning effects of optical parts and repels air bubbles.

10.2.2.3 The sensor should be installed with maximized wall distance to reduce backscattered or reflective signal (see Fig. 8).

10.2.2.4 Locate transmitter and sensor so that there is easy access for maintenance or calibration.

10.2.2.5 Adjust the flow rate to minimize particle fallout in the sample lines while maximizing bubble removal.

10.2.2.6 Measurement should be done under pressure to avoid degassing.

10.2.2.7 The location of the in-line sensor should be such that the sensor can be easily removed from the process for servicing.

10.3 The In-Situ Measurement Technique:

10.3.1 Before making an in-situ turbidity determination, ensure that the instrument to be used has been cleaned, verified, and or calibrated properly, if need be, and that the verification/ calibration process has been accurately documented.

10.3.2 Guidelines for long-term instrument deployment fall under the topic of continuous monitors; refer to the manufacturer's instructions and recommendations.

10.3.3 Perform a calibration verification on the instrument in the laboratory or on-site using a calibration solution before inserting it into the process and if verification does not confirm the calibration is still within 5 %, then re-calibrate.

10.3.3.1 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 ± 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 %.

10.3.3.2 Document verification readings and any metadata, including the instrument manufacturer and model. Use reporting units appropriate for the instrument, as described in Table 1.

10.3.3.3 After verification, clean the sensor with a thorough rinse of deionized water prior to installation. Follow the manufacturer's recommendations for storage of sensors.

10.3.4 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 representa-

tive sample and dilute it with one or more equal volumes of turbidity-free water, recording the volume of water used for dilution.

10.4 When taking an aliquot for dilution from an on-line technology, it is important to use the same technology to measure the dilution. If a different technology is used, the measurements may not be comparable.

10.4.1 *Quality Control*—Periodically check instrument performance by placing a primary or secondary calibration solution in the instrument storage vessel and comparing the standard value with the reading displayed. Record in the instrument maintenance logbook all the readings obtained. Re-calibrate if the following criteria are exceeded: ± 0.5 turbidity unit or ± 5 % of the measured value, whichever is greater.

11. Calibration and Calibration Verification

11.1 Calibration of In-line and On-Line Turbidimeters:

11.1.1 Determine if the instrument requires any maintenance such as cleaning the sample chamber, bubble removal devices, or flow-through cell, adjusting sample flow rates, wiper maintenance, and so on. Follow the manufacturer's instructions for any required instrument maintenance prior to calibration.

11.1.2 Follow the manufacturer's instructions for calibration and operation. Calibrate the instrument to assure proper operation for the range of interest with appropriate standards.

NOTE 8—Close adherence to the calibration procedure and to the rinsing/seasoning techniques is very important to ensure the data remains consistent across all locations with all of the turbidimeters.

11.1.3 Formazin-based calibration standards should be resuspended through inversion (one second inversion cycle) 25 times followed by a 2–10 minute wait to allow for bubble removal. Standards of 40 NTU or below will remain suspended for up to 30 minutes; standards greater than 40 NTU may require resuspension more frequently.

11.1.3.1 The relationship between turbidity and nephelometric light scatter is known to be linear only up to 40 NTU. Above 40 NTU, the relationship may become non-linear and additional calibration standards will be necessary. It is imperative that the user of this test method follow the manufacturer's instructions for calibration and calibration standard values. Following these instructions will optimize the measurement performance of the respective turbidimeter. Verify calibrations and performance in the range of interest (or as close to the measurement range of interest as possible) using defined