



Designation: ~~D6855-03~~ Designation: D6855 - 10

Standard Test Method for Determination of Turbidity Below 5 NTU in Static Mode¹

This standard is issued under the fixed designation D6855; 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 static determination of turbidity in water (see 4.1).

1.2 This test method is applicable to the measurement of turbidities under 5.0 nephelometric turbidity units (NTU).

1.3 This test method was tested on municipal drinking water, ultra-pure water and low turbidity samples. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.4

1.4 This test method uses calibration standards as defined in NTU values, but other assigned turbidity units are assumed to be equivalent.

1.5 This test method assigns traceable reporting units to the type of respective technology that was used to perform the measurement. Units are numerically equivalent with respect to the calibration standard. For example, a 1.0 NTU formazin standard is also equal to a 1.0 FNU standard, a 1.0 FNRU standard and so forth.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Refer to the MSDSs for all chemicals used in this procedure.

2. Referenced Documents

2.1 *ASTM Standards*:²

D1129 Terminology Relating to Water

D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits

D1193 Specification for Reagent 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

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

E691 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, Turbidity³

ISO 7027 (The International Organization for Standardization) Water Quality—for the Determination of Turbidity⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method refer to Terminology D1129.

3.2 *Definitions*:

3.2.1 *calibration turbidity standard*—a turbidity standard that is traceable and equivalent to the reference turbidity standard to within statistical errors, including commercially prepared 4000 NTU Formazin, stabilized formazin (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 specific.

3.2.2 *calibration verification standards*—defined standards used to verify the accuracy of a calibration in the measurement range of interest. These standards may not be used to perform calibrations, only calibration verifications. Included standards are

¹ This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.07 on Sediments, Geomorphology, and Open-Channel Flow.

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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 Association (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

*A Summary of Changes section appears at the end of this standard.

opto-mechanical light scatter devices, gel-like standards, or any other type of stable liquid standard.

NOTE 2—Calibration verification standards may be instrument specific.

3.2.3 *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).

3.2.4 *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.

3.2.5 *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).

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

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

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

3.2.9 *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.

4. Summary of Test Method

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

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

5. Significance and Use

5.1 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 is often accomplished by coagulation, settling, and filtration. Measurement of turbidity provides a rapid means of process control for when, how, and to what extent the water must be treated to meet specifications.

5.2 This test method is suitable to turbidity such as that found in drinking water, process water, and high purity industrial water.

5.3 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.

5.3.1 Table 1 describes technologies and reporting results (see also Refs (1),(2),(3)).⁵ Those technologies listed are appropriate for the range of measurement prescribed in this method. Others may come available in the future. Fig. X5.1 provides a flow chart to aid in selection of the appropriate technology for low-level static turbidity applications.

5.3.2 If a design that falls outside of the criteria listed in Table 1 is used, the turbidity should be reported in turbidity units (TU) with a subscripted wavelength value to characterize the light source that was used.

6. Interferences

6.1 For this application, bubbles, color and large particles, although they cause turbidity, may result in interferences in measured turbidity as determined by this 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.

6.3 Scratches, finger marks, or dirt on the walls of the sample cell may give erroneous readings. 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.

⁵ 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 Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁵ The boldface numbers in parentheses refer to the list of references at the end of this standard.

TABLE 1 Applicable Technologies Available for Performing Static Turbidity Measurements Below 5 NTU

Design and Reporting Unit	Prominent Application	Key Design Features	Typical Instrument Range	Suggested Application
Nephelometric non-ratio (NTU)	White light turbidimeters. Comply with USEPA Method 180.1 (1) for low level turbidity monitoring.	Detector centered at 90° relative to the incident light beam. Uses a white light spectral source.	0.020 to 40	Regulatory reporting of clean water
Ratio White Light turbidimeters (NTRU)	Complies with ISWTR regulations and Standard Method 2130B. (2) 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.	0.020 to 10 000	Regulatory Reporting of clean water
Nephelometric, near-IR turbidimeters, non-ratiometric (FNU)	Complies with ISO 7027. The wavelength is less susceptible to color interferences. Applicable for samples with color and good for low level monitoring.	Detector centered at 90° relative to the incident light beam. Uses a near-IR (780-900 nm) monochromatic light source.	0.012 to 1000	0 - 40 ISO 7027 Regulatory reporting
Nephelometric near-IR turbidimeters, ratio metric (FNRU)	Complies with ISO 7027. Applicable for samples with high levels of color and for monitoring to high turbidity levels.	Uses a near-IR monochromatic light source (780-900 nm). Primary detector centered at 90°. Other detectors located at other angles. An instrument algorithm uses a combination of detector readings to generate the turbidity reading.	0.012 to 10 000	0 - 40 ISO 7027 Regulatory reporting
Nephelometric Turbidity Multibeam Unit (NTMU)	Is applicable to EPA regulatory method G1 Method 2. (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.	0.012 to 4000	0 to 40 Reporting for EPA and ISO compliance
mNTU	Is applicable to reporting of clean waters and filter performance monitoring. Very sensitive to turbidity changes in low turbidity samples. (3)	Nephelometric method involving a laser-based light source at 660-nm and a high sensitivity photo-multiplier tube (PMT) detector for light scattered at 90°. 1000 mNTU = 1 NTU	5 to 5000 mNTU or 0.005 to 5.000 NTU	0-5000 mNTU, for EPA compliance reporting on drinking water systems

6.3.1 Sample cell caps and liners must also be scrupulously clean to prevent contamination of the sample.

6.4 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 for instructions on matching sample cells.

NOTE 3—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.

NOTE 4—Sample cells can be matched by first filling with dilution water (see 8.2). Allow the sample cell to stand for 5 to 10 min to allow for bubbles to vacate the sample. This is followed by cleaning and polishing the outside of the cell. Cells are then measured on the same turbidimeter and should read no different than 0.01 NTU.

6.5 Condensation of optical elements or sample cells can lead to severe errors in measurement.

7. Apparatus

7.1 Two types of instruments are available for the nephelometric method, the nephelometer and ratio nephelometer (see Figs. 1 and 2).

7.2 The resolution of the instruments should permit detection of differences of 0.01 NTU or less in waters having turbidities of less than 5.0 NTU. The instrument must measure the range from ≤ 0.02 to 5.0 NTU. See 12.1 for calibration of instruments. Calibration verification in the immediate range of interest must be performed using acceptable, defined verification standards (see 12.2).

NOTE 5—Consult manufacturer's instructions for guidance associated with verification methods and verification devices.

7.2.1 Consult the manufacturer to ensure that your instrument meets or exceeds the specifications of this method.

7.3 Photoelectric Nephelometer :

7.3.1 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 to 900 nm may also be used if accurately characterized to be equivalent in performance to tungsten using calibration and

Figure 1 Photoelectric Nephelometer

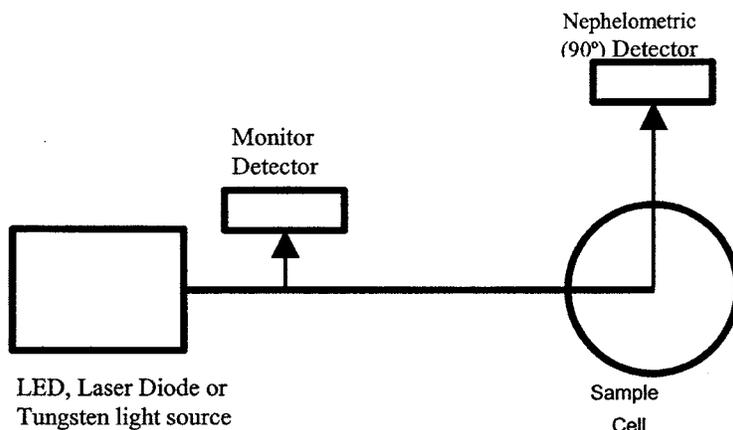


FIG. 1 Photoelectric Nephelometer

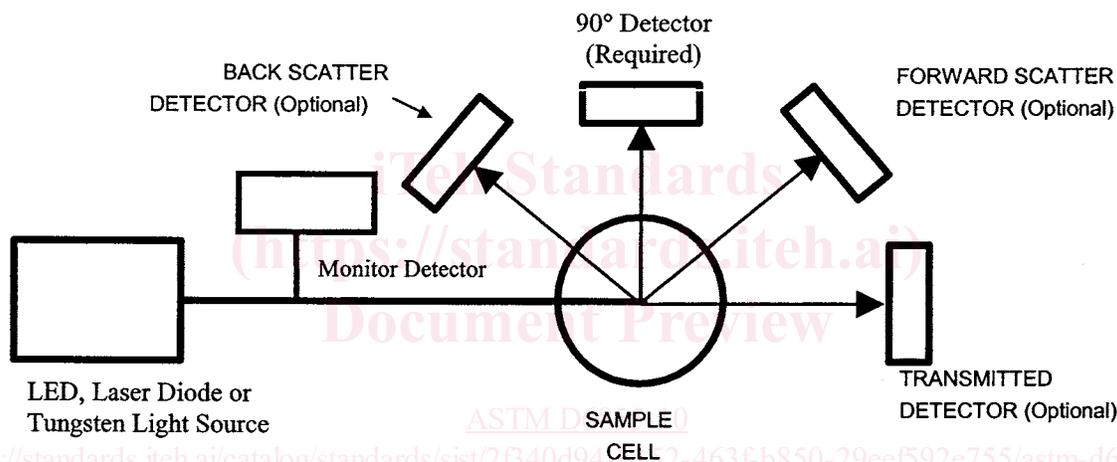


FIG. 2 Ratio Photoelectric Nephelometer (Single Beam Design)

calibration verification standards. If LEDs or laser diodes are used, then the LED or Laser diode should be coupled with a monitor detection device to achieve a constant output. LEDs and laser diodes should be characterized by a wavelength of between 400 and 900 nm with a bandwidth of less than 60 nm. (Examples of LEDs include: White light with a defined bandwidth and 860 ± 30 nm per ISO 7027.) The total distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. The angle of light acceptance to the detector shall be centered at 90° to the centerline of the incident light path and shall not exceed $\pm 10^\circ$ from the 90° scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used.

7.3.2 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. To minimize initial differences, the following design criteria should be observed (see Fig. 1).

7.4 Ratio Photoelectric Nephelometer:

7.4.1 Ratio Photoelectric Nephelometer—(see Fig. 2 for single beam design; see Fig. 3 for multiple beam design.) This instrument uses the measurement derived through the use of a nephelometric detector that serves as the primary detector and one or more other detectors used to compensate for variation in incident light fluctuation, stray light, instrument noise, or sample color. As needed by the design, additional photodetectors may be used to sense the intensity of light scattered at other angles. The signals from these additional photodetectors may be used to compensate for variations in incident light fluctuation, instrument stray light, instrument noise and/or sample color. The ratio photoelectric nephelometer should be so designed that minimal stray light reaches the detector(s), and should be free from significant drift after a short warm-up period. The light source should be a tungsten lamp, operated at a color temperature between 2200 and 3000 K (USEPA Method 180.1). LEDs and laser diodes in defined wavelengths ranging from 400 to 900 nm may also be used. If an LED or a laser diode is used in the single beam design, then the LED or laser

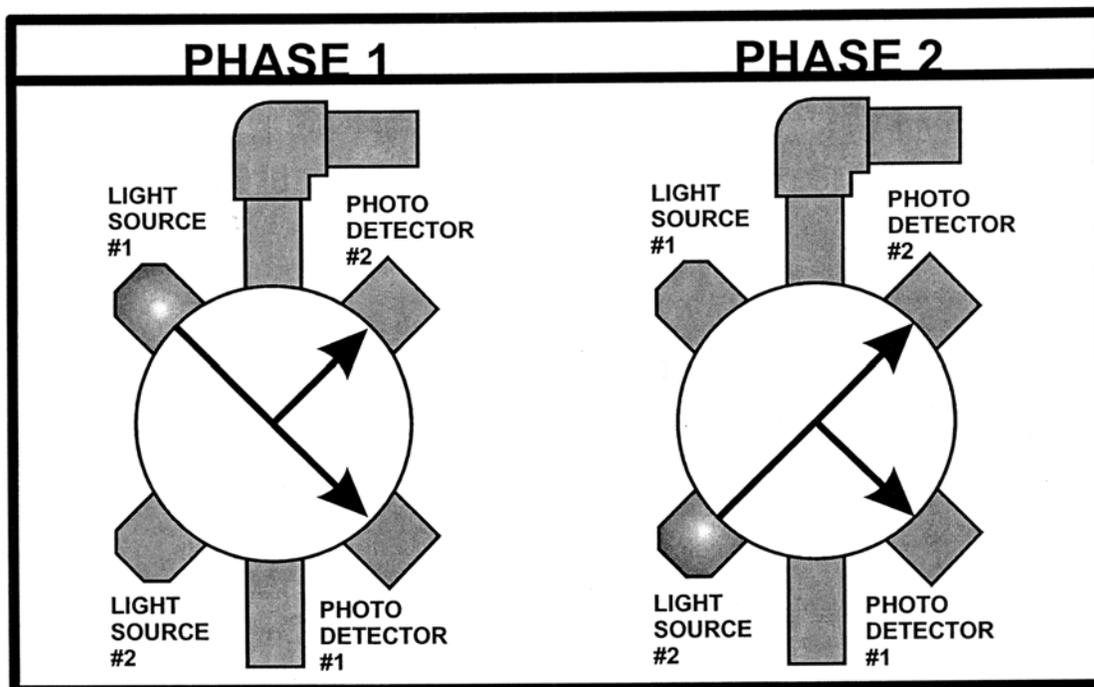


FIG. 3 Ratio Photoelectric Nephelometer (Multiple Beam Design)

diode should be coupled with a monitor detection device to achieve a consistent output. The distance traversed by incident light and scattered light within the sample is not to exceed 10 cm. The angle of light acceptance to the nephelometric detector(s) should be centered at 90° to the centerline of the incident light path and should not exceed $\pm 10^\circ$ from the scatter path center line. The detector must have a spectral response that is sensitive to the spectral output of the incident light used. The instrument calibration (algorithm) must be designed such that the scaleable reading is from the nephelometric detector(s), and other detectors are used to compensate for instrument variation described in 7.3.1.

7.4.2 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. To minimize initial differences, the following design criteria should be observed (see Figs. 2 and 3).

7.4.3 Examples of applicable nephelometers include: Photoelectric Nephelometer, Ratio Photoelectric Nephelometer with a single beam design, laser-based ratio photoelectric nephelometer, and laser-based photoelectric nephelometer and Ratio photoelectric nephelometer in the dual beam design. In these designs, the correlation between detector response and increasing turbidity is positive.

7.5 Sample Cells:

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

7.5.1.1 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 X1 for sample cell cleaning procedure).

7.5.1.2 Index marked so that repeated exact placements into the instrument sample cell compartment for measurement can be made. See H.4.2:H1.4.2.

7.5.1.3 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. Instrument and sample cell design criteria are given in 7.3.1.

7.5.1.4 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.

7.6 Sample Chambers:

7.6.1 For those units not using highly transparent sample cells, the sample is placed directly into the sample chamber. For those units, the sample chamber must be the following:

7.6.1.1 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.

7.6.1.2 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.⁶

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.

NOTE 7—(This is the ASTM Standard Footnote on Purity).

8.2 Reverse osmosis (RO) water is acceptable and preferred in this method. Standard dilution waters and rinse waters shall be prepared by filtration of Type III water (See Specification D1193) through a 0.22 µm or smaller membrane or other suitable filter within 1 h of use to reduce background turbidity.

9. Reagents

9.1 Dilution and final rinsing water, see 8.2.

9.2 *Turbidity Standards*—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.

NOTE 8—Equivalent, commercially-available, calibration standards may be used. These standards, such as stabilized formazin and styrenedivinylbenzene (SDVB), have a specified turbidity value and accuracy. Such standards must be referenced (traceable) to formazin. 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.

9.2.2.1 Quantitatively transfer 5.0 g of reagent grade hydrazine sulfate (99.5 %+ purity) ($N_2H_4 \cdot H_2SO_4$) into approximately 400 mL of dilution water (see 8.2) contained in a 1-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 rinsings to the flask.

9.2.2.2 Quantitatively transfer 50.0 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 µm 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 $25 \pm 1^\circ C$. The 4000 NTU Formazin suspension develops during this time.

NOTE 11—This suspension, if stored at 20 to 25°C in amber polyethylene bottles, is stable for 1 year; it is stable for 1 month if stored in glass at 20 to 25°C.

9.2.3 ~~Stabilized formazin turbidity standards (see Ref (1))~~ Stabilized formazin turbidity standards (see Refs (4, 5)) 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 (see Refs (2,6,37)) 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)*—All labware shall be seasoned (see Appendix X2). Invert 4000 NTU stock suspension 25 times to mix (1 s inversion cycle); immediately pipette, using a Class A pipette, 10.0 mL of mixed 4000 NTU

⁶The boldface numbers in parentheses refer to the list of references at the end of this standard.

⁶ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.