



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

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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 *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:

D 1129 Terminology Relating to Water²

D 1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits²

D 1193 Specification for Reagent Water²

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D19 on Water²

D 3370 Practices for Sampling Water from Closed Conduits²

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

2.2 Other Referenced Standards:

USEPA Method 180.1 Methods for Chemical Analysis of Water and Wastes, Turbidity⁵

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 *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from United States Environmental Protection Association (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460.

ISO 7027 (The International Organization for Standardization) Water Quality—for the Determination of Turbidity⁶

3. Terminology

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

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 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

⁶ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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.

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.

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 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

Figure 1 Photoelectric Nephelometer

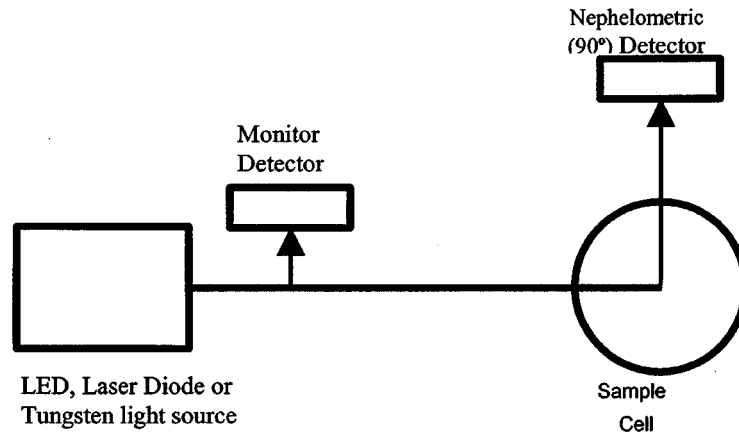


FIG. 1 Photoelectric Nephelometer

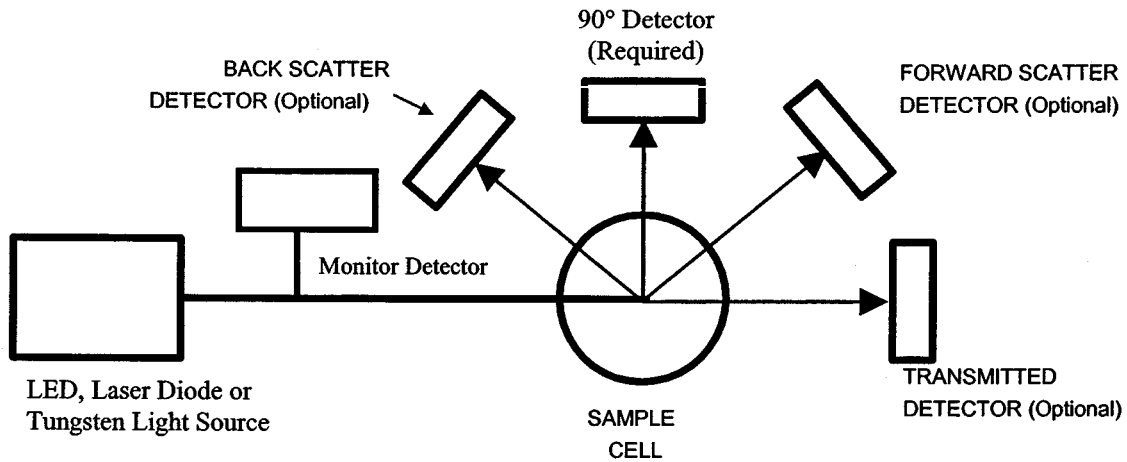


FIG. 2 Ratio Photoelectric Nephelometer (Single Beam Design)

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 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

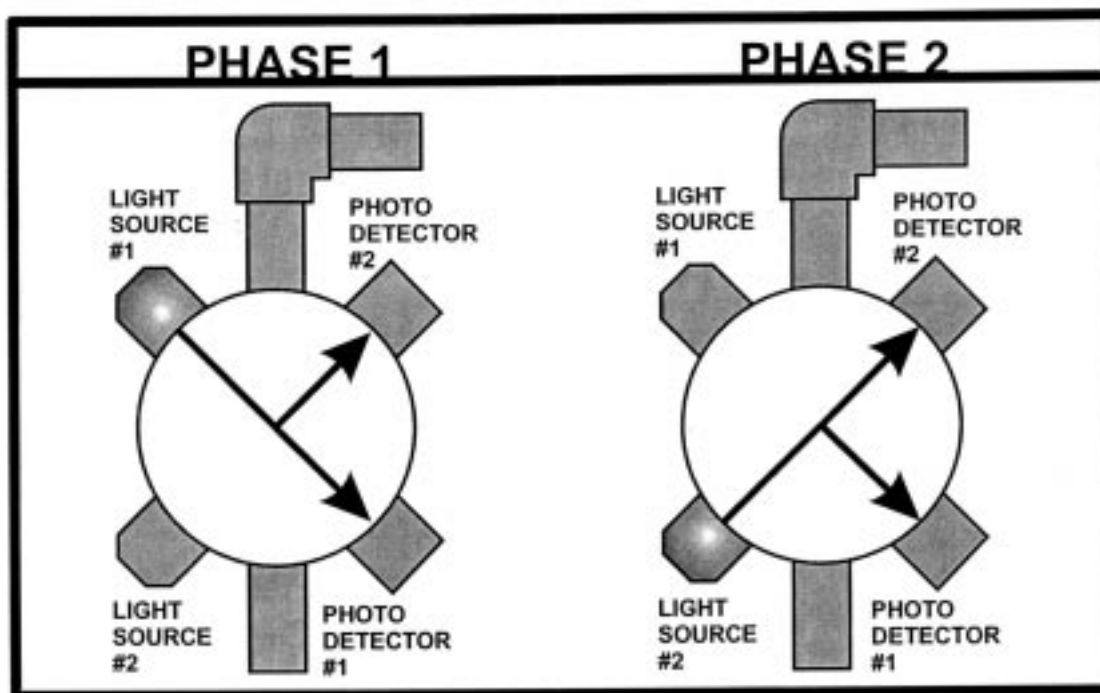


FIG. 3 Ratio Photoelectric Nephelometer (Multiple Beam Design)

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).

7.5 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 11.4.2.1.

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 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.⁷

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

⁷ 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 *Anal. 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.

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 D 1193) 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 styrenedi-vinylbenzene (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) ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{SO}_4$) 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 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 $25 \pm 1^\circ\text{C}$. 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))⁸ 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 (2,3)) 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 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.

9.2.5.1 This suspension serves as the highest calibration standard that may be used with this method.

9.2.6 *Dilute Formazin Turbidity Suspension Standard (1.0 NTU)*—Prepare this standard daily by inverting the 40 NTU (see 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 X2).

NOTE 13—The instructions below result in the preparation of 200 mL of a formazin standard. Users of this method will need different volumes of the standard to meet their instrument's individual needs; glassware and reagent volumes shall be adjusted accordingly.

9.2.6.1 Within one day of use, rinse both a glass Class A 5.0 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.

9.2.6.2 Using the cleaned glassware, pipet 5.0 mL of well-mixed 40.0 NTU formazin suspension (see 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.

9.2.7 *Miscellaneous Dilute Formazin Turbidity Suspension Standard*—Prepare all turbidity standards with values below 40 NTU daily. Standards ≥ 40 NTU have a useful life of one week. All labware shall be seasoned (See Appendix X2). Use

⁸ The boldface numbers in parentheses refer to the list of references at the end of this standard.

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 (see 9.2.5) into a 100-mL volumetric flask and diluting to mark with dilution water (see 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 4.0 NTU.

NOTE 14—Refer to Appendix X3 for stability information of formazin standards.

9.2.8 Stable low-level turbidity standards are commercially available. These standards, such as stabilized formazin and styrenedivinylbenzene (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 D 1192 and Practices D 3370.

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 affect particles, 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.

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 15—If degassing is necessary an un-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 very 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.

12.2.2 The relationship between turbidity and nephelometric light scatter is known to be linear up to 40 NTU; therefore, calibration standards ranging up to 40 NTU may be used for this method. Verify linearity in the range of interest (or as close to the measurement range of interest as possible) using defined calibration or calibration verification standards with a known accuracy. (Consult manufacturer's recommendations for guidance associated with verification methods and devices.)

12.2.3 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 16—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 styrenedivinylbenzene (SDVB). These standards may be used to calibrate the instrument.

12.2.4 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 manufacturer's recommendations for guidance associated with verification methods and devices.)

12.2.5 In case of verification failure, clean the instrument to reduce stray light levels or contamination. Follow with a

recalibration according to manufacturer's calibration instructions, or at a minimum on a quarterly basis.

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

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

12.2.8 Proper care and maintenance of sample cells and sample chambers must be applied (see 7.5).

13. Procedure for the Measurement of Water Turbidity

13.1 *Turbidity Less Than 5.0 NTU*—Gently invert the sample several times (1 s inversion cycle) to thoroughly disperse any solids. Rinse a clean sample cell several times with sample. Slowly fill the sample cell with sample and cap the cell. Clean outside surfaces of the sample cell (see non-mandatory Appendix X1). Place the sample cell into the instrument at index and wait for 2 to 10 min for measurement to become stable. Record the stable reading.

14. Report

14.1 Report results as follows:

NTU	Report to Nearest (NTU)
0.02* < NTU ≤ 1.00	0.01
1.00 < NTU < 5.00	0.05

NOTE 17—New developments may allow instruments to extend this range.

15. Quality Control/Quality Assurance

15.1 In order to be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when running the test.

15.2 *Calibration and Calibration Verification:*

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

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

15.2.3 Verify instrument calibration by analyzing calibration or calibration verification standards that are within the range of interest. These standards must be run prior to and after any sample determinations. The recommended frequency for determining calibration verification is more than or equal to 5 % of all samples determined for each batch of samples.

NOTE 18—Consult instrument or Standards manufacturers for recommended and available sources for verification standards.

15.2.4 The values obtained upon analysis of calibration or calibration verification standards must fall within the acceptance limits presented in the following table as determined from single operator standard deviation (S_o) presented in Table 1 of this standard.

15.2.5 Alternative to 15.2.4:

15.2.5.1 The values obtained upon analysis of calibration or calibration verification standards must fall within the acceptance criteria generated after sufficient data is generated for each of the standards, typically 20 to 30 results. Control charts must be developed from the mean recovery (\bar{x}) and the standard deviation (S) of the percent recovery for the standards. These data are used to establish upper and lower control limits as follows:

$$\begin{aligned} \text{Upper control limit} &= \bar{x} \pm 3S \\ \text{Lower control limit} &= \bar{x} \pm 3S \end{aligned}$$

15.2.5.2 After each five to ten new recovery measurements, new control limits must be calculated using the most recent 20 to 30 data points. If these calculated control limits exceed those established in the method, corrective action must be taken. If calibration cannot be verified because determinations of standards are determined to be outside acceptance limits, recalibrate the instrument.

15.3 *Duplicate:*

TABLE 1 Final Statistical Summary for Turbidity in Standard Sample Surrogates

Sample ID	0.9 NTU Formazin A	0.625 NTU Formazin B	0.122 NTU StablCal A	0.262 NTU StablCal B	4.09 NTU StablCal C	0.50 NTU SDVB A	0.20 NTU SDVB B	2.00 NTU SDVB C
True Conc, NTU	0.900	0.625	0.122	0.262	4.090	0.500	0.200	2.000
# of Retained Values	6	6	6	6	6	6	6	6
Mean recovery (\bar{XBAR}), NTU	1.08367	0.67783	0.11878	0.23422	3.99900	0.57176	0.22594	2.11233
Recovery, %	120.41 %	108.45 %	97.36 %	89.40 %	97.78 %	114.35 %	112.97 %	105.62 %
Overall Standard Deviation (S_L) ^A	0.01688	0.07285	0.01756	0.02443	0.08256	0.04759	0.02433	0.13128
Standard Deviation of Cell Averages ($S_{\bar{XBAR}}$) ^A								
Most Extreme Value	1.10833	0.80333	0.14333	0.26667	4.15333	0.63133	0.25667	2.28667
Single Operator Standard Deviation (S_o) ^B	0.1547	0.0174	0.0089	0.0055	0.0477	0.0088	0.0053	0.0217
Analyst Relative Deviation, % ^C	14.328 %	2.657 %	7.549 %	2.435 %	1.219 %	1.54 %	2.435 %	1.03 %
Laboratory Standard Deviation (S_L) ^D	0.1274	0.0742	0.0190	0.0248	0.0913	0.0481	0.0247	0.1325
Laboratory Relative Deviation, % ^C	11.8 %	10.9 %	16.0 %	10.6 %	2.3 %	8.4 %	10.9 %	6.3 %

^A The S_L is the laboratory standard deviation and was calculated using the Practice E 691 statistical package. This is the standard deviation of the means from triplicate values. While not required by Practice D 2777, this information may still be useful to the users of this method. This was the standard deviation of the means of triplicate observations among six laboratories.

^B S_o was calculated as the pooled within-laboratory standard deviation from triplicate observations among six laboratories, calculated in accordance with the "repeatability" measure of Practice E 691. This is the exact equivalent of single operator precision as defined in Practice D 2777 when Youden pairs are not utilized.

^C Standard deviation divided by mean recovery (S/\bar{XBAR}).

^D S_L was calculated in accordance with the "reproducibility" measure of Practice E 691. This is the equivalent of the overall standard deviation of Practice D 2777, which describes the standard deviation of a single measurement among laboratories. Where $S_L < S_o$, use S_o as the reproducibility.

TABLE 2 Final Statistical Summary for Turbidity in Real World Water Samples

Sample ID	Combined Filter Effluent	South Applied to Filters	South Applied to Filters #2	Raw Water Deleware	Raw Water Schuykill	Filtered Tap
True Conc, NTU	N/A	N/A	N/A	N/A	N/A	N/A
# of Retained Values	6	6	6	6	6	6
Mean Recovery (X_{BAR}), NTU	0.0444	0.2581	1.0139	3.5497	3.7807	0.0662
Recovery, %	N/A	N/A	N/A	N/A	N/A	N/A
Overall Standard Deviation (S_L) ^A	0.00625	0.02480	0.04170	0.26323	0.19192	0.00927
Standard Deviation of Cell Averages (S_{XBAR}) ^A						
Most Extreme Value	0.03333	0.29000	1.06667	3.22333	3.53333	0.04900
Single Operator Standard Deviation (S_o) ^B	0.0050	0.0092	0.0163	0.0541	0.0804	0.0055
Analyst Relative Deviation, % ^C	11.219 %	3.657 %	1.61 %	1.52 %	2.13 %	8.327 %
Laboratory Standard Deviation (S_L) ^D	0.0075	0.0259	0.0438	0.2669	0.2028	0.0103
Laboratory Relative Deviation, % ^C	16.8 %	10.0 %	4.3 %	7.5 %	5.4 %	15.5 %

^A The S_L is the laboratory standard deviation and was calculated using the Practice E 691 statistical package. This is the standard deviation of the means from triplicate values. While not required by Practice D 2777, this information may still be useful to the users of this method. This was the standard deviation of the means of triplicate observations among six laboratories.

^B S_o was calculated as the pooled within-laboratory standard deviation from triplicate observations among six laboratories, calculated in accordance with the "repeatability" measure of Practice E 691. This is the exact equivalent of single operator precision as defined in Practice D 2777 when Youden pairs are not utilized.

^C Standard deviation divided by mean recovery (S/X_{BAR}).

^D S_L was calculated in accordance with the "reproducibility" measure of Practice E 691. This is the equivalent of the overall standard deviation of Practice D 2777, which describes the standard deviation of a single measurement among laboratories. Where $S_L < S_o$, use S_o as the reproducibility.

15.3.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch of samples. The recommended frequency for determining precision is more than or equal to 5 % of all samples determined for each batch of samples.

15.3.2 Calculate the standard deviation of the duplicate values and compare it to the single operator precision data found in Table 1 of this procedure by using a one-sided F test at the $\alpha = 0.01$ significance level.

15.3.2.1 Appendix X4 is the Table X2.1 from Practice D 5847 for the critical values of F at the 1 % significance (99 % confidence) level (one-sided). Also, reference Practice D 5847 if necessary to see an example of how the calculation utilizes both the single operator standard deviation (S_o) and the standard deviation of the duplicate sample analysis values (S_A).

15.3.2.2 The result from $(S_A)^2/(S_o)^2 = (\text{standard deviation of sample reps})^2/(\text{single operator standard deviation})^2 =$ calculated value versus ($>$ or $<$) critical value from table. The result will determine if the duplicate values are acceptable.

15.3.3 If the result exceeds the precision limit as derived from the F test, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test procedure.

15.4 *Independent Reference Material (IRM)*—In order to verify the quantitative value produced by the test procedure, analyze an IRM submitted as a regular sample to the laboratory at least once per quarter. The value of the IRM should be in the range of the determinations that the lab normally determines during the analyses of samples. The value obtained must fall within the control limits specified by the provider of the IRM.

16. Precision and Bias ⁹

16.1 This Test method was tested on 7 laboratories that were assembled at a common site. Each laboratory consisted of an

operator and an instrument (turbidimeter). Testing was conducted over a two-day period.

16.1.1 The collaborative Test Data included two groups of samples: Standard turbidity sample surrogates and real world turbidity samples. Artificial Turbidity sample surrogates included: Formazin (freshly prepared) at 0.90 and 0.625 NTU; Stabilized Formazin at 0.122, 0.262, and 4.09 NTU; and Styrenedivinylbenzene (SDVB) of 0.20, 0.50, and 2.0 NTU. Real world turbidity samples included: Two river water samples denoted Raw Water Deleware and Raw Water Schuykill; two settled water samples from a drinking water plant (DWP); one combined filter effluent water sample from a DWP; and one tap water sample. The real-world samples have no assigned values. The true values for these samples are unknown and therefore an estimate of bias cannot be made for these samples.

16.1.2 Each sample was measured in triplicate over the same period of time across all laboratories in an effort to minimize sample changed due to their potential lack of stability.

16.2 Results of this collaborative study may not be typical of results for matrices other than those studied.

16.3 Precision and bias was determined in accordance to Practice D 2777 whenever possible. Final statistics for the two groups of samples can be found in Tables 1 and 2.

16.4 Because Youden Pair samples were not feasible in this study, S_o was calculated in accordance with the repeatability measure of Practice E 691. This is the exact equivalent of single operator precision as defined in Practice D 2777 when Youden pairs are not utilized.

16.4.1 Only a statement of single operator precision is required. The modification of this study was allowed according to section 1.3.2 in Practice D 2777.

16.4.2 The final statistical package utilized Practice D 2777 for all applicable calculation with one exception, which was Stathe Single Operator Standard Deviation, S_o .

⁹ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D19-1172.

17. Keywords

17.1 calibration; calibration turbidity standard; calibration verification; measurement; nephelometric turbidity; ratio turbidity; turbidimeter; turbidity; turbidity standards

APPENDIXES

(Nonmandatory Information)

X1. CLEANING AND MAINTENANCE OF TURBIDITY SAMPLE CELLS AND CAPS

X1.1 Introduction

X1.1.1 Sample cells and caps must be kept scrupulously clean both inside and outside. This level of cleanliness must be established prior to cell matching process and maintained throughout the cell's working lifetime.

X1.2 Cleaning

X1.2.1 Sample cells and caps must be examined prior to cleaning. All scratched, blemished, and scuffed cells should be discarded. All caps with liners that show signs of deterioration also must be discarded. Caps with teflon liners are recommended.

X1.2.2 Prepare a 1 % solution of a liquid detergent such as Liqui-Nox. The quantity prepared should be sufficient to fill all cells twice with this solution.

X1.2.3 Fill each cell to half of its capacity with the solution, cap each cell, and shake each cell vigorously for at least 1 min. Flush each cell and cap with tap water 5 times.

X1.2.4 Refill each cell to capacity with the solution, cap each cell, and allow to stand for 2 h.

NOTE X1.1—Plastic test tube racks should be used for storage of cells.

X1.2.5 Uncap each cell, empty each, and flush each cell and cap with tap water 5 times making sure to rid each cell and cap of any residual detergent solution.

X1.2.6 Fill each cell with deionized (DI) water that has been filtered through a 0.2 μm filter, and cap. Allow to stand for 1 h.

X1.2.7 Uncap each cell and empty.

X1.2.8 Prepare a 1:4 hydrochloric acid (HCl) solution.

X1.2.9 Fill each cell to half of its capacity. Replace the cap and invert the cell 5 times (1 s inversion cycle). Uncap the cell, and pour off the HCl solution to the next cell to be rinsed.

X1.2.9.1 Repeat X1.2.9 for up to 10 cells, after which the HCl solution is discarded. The process is repeated for every 10 cells. Each sample cell should be rinsed at least 3 times with the HCl solution.

NOTE X1.2—Caps with Teflon liners are not damaged by the HCl solution. If less chemical resistant liners are present on caps, this procedure can be completed by using several caps that are dedicated for acid rinsing.

X1.2.10 After acid rinsing each cell 3 times, rinse the cells and caps 5 times with tap water and 5 times with 0.22 μm filtered DI water.

X1.2.11 Fill the cell with DI water, and place into a plastic test tube rack.

X1.2.12 Sample cells now are ready for matching, and/or sample analyses.

X1.3 Maintenance

X1.3.1 Sample cells must be evaluated frequently (weekly evaluations are recommended) to determine if they remain matched. Prior to the evaluation, all cells must be prepared as per X1.2 of this appendix.

X1.3.2 Fill a thoroughly clean container with sufficient 0.22 μm filtered DI water so that each cell can be filled 4 times. Acclimate the water so that it is at 20 to 25°C.

X1.3.3 Fill each cell with the water, degas the water by placing the cell into an ultrasonic bath for no more than 2 s. Cap the cell, and wipe the outside of the cell dry with a lint free tissue to eliminate any moisture.

X1.3.4 Place the water filled clean and dry cell in an indexed manner into the instrument and record the stable value.

X1.3.5 Repeat this process for each cell. If any cell is more than 0.010 NTU from any other cell, replace it with a cell that is.

X1.3.6 Continue with the evaluation of the cells until sufficient cells are obtained that meet the 0.010 NTU criteria and fulfill the analytical needs of the user.

X1.3.7 The cells passing the evaluation criteria now can be used for sample analysis.

X1.3.8 After sample determinations cells must be well rinsed with 0.22 μm filtered DI water and stored filled.

X2. PROCEDURE FOR SEASONING GLASSWARE WHEN PREPARING CALIBRATION STANDARDS

X2.1 Introduction

X2.1.1 Seasoning is a procedure in which glassware is conditioned immediately prior use in the preparation of turbidity standards. Seasoning will reduce contamination and volumetric dilution errors and is a common practice in volumetric quantitative analysis. The process involves rinsing the glassware twice with the specific standard that will be diluted to prepare a standard of lower value. Seasoning should be used when preparing any standard from the Stock 4000 NTU Formazin Standard. It is of primary importance to season pipets used to prepare low-level turbidity standards. Seasoning should be performed immediately before performing the actual volumetric dilution. Below is the general procedure that should be used for seasoning a pipet. A similar practice should be applied when filling sample cells with sample immediately before analysis.

X2.2 Procedure

X2.2.1 Prepare the solution that is to be diluted. For formazin, this involves mixing the standard immediately prior to use.

X2.2.2 Rinse a small beaker with a small portion of the standard. Discard the rinsing to waste. Repeat this a second time.

X2.2.3 Fill the beaker with enough standard to accommodate at least three times the volume required to prepare the dilution. For example, if a 10 mL dilution volume is to be used, then at least 30 mL of standard should be placed in the beaker.

X2.2.4 Draw a small amount of the standard from the beaker into the pipet. Swirl the standard around the pipet, making sure it contacts all internal surfaces up to the draw line. Then, discard this to waste.

X2.2.5 Draw up a second amount of standard from the beaker up slightly past the fill line. Immediately discard to waste.

X2.2.6 The pipet is now ready for volumetric draw of the standard. There should be enough standard left in the beaker to use. This volumetric draw of the standard should take place immediately after the seasoning.

X3. STABILITY OF FORMAZIN

X3.1 Stability studies of low level and high level formazin standards were conducted by ASTM members to support the formazin preparation instructions set forth in this document.

X3.1.1 Table X3.1 summarizes the stability data collected for low level formazin standards.¹⁰

X3.1.2 Table X3.2 summarizes the stability data collected for high level formazin standards.¹¹

¹⁰ ASTM Low-Level Formazin Stability Study. ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

¹¹ ASTM High-Level Formazin Stability Study. ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

TABLE X3.2 Summary of High Level Formazin Stability

Formazin 20 NTU						
	Day 0.08	Day 1.00	Day 1.92	Day 6.92	Day 13.92	Day 28.79
Average	19.67	19.470	19.315	19.125	18.805	18.125
Std Dev	0.5630	0.52273	0.524982	9.521167	0.589096	0.60339
% Error versus Theoretical	-1.65	-2.65	-3.425	-4.375	-5.975	-9.375
Formazin 0.60 NTU						
	Day 0.08	Day 1.00	Day 1.92	Day 6.92	Day 13.92	Day 28.79
Average	0.61	0.592	0.591	0.586	0.569	0.533
Std Dev	0.0176	0.017515	0.019028	0.018961	0.017041	0.022104
% Error versus Theoretical	1.091037	-1.26667	-1.52273	-2.41667	-5.18333	-11.2333

TABLE X3.1 Summary of Low Level Formazin Stability

Change in the Measured Value versus Time Since Preparation										
Standard	0.1 Days	1 Day	2.2 days	7.3 Days	13.1 Days	21 Days	28 Days	47 Days	61 Days	81.3 Days
0.1	-0.92	-1.61	0.0	-2.99	-5.06	-6.7	-8.05	-14.02	-20	-32.41
0.3	-0.74	0.0	3.31	3.23	-3.23	-5.38	-6.45	-14.81	-22.91	-44.5
0.5	-1.7	-1.7	-0.94	-2.21	-6.97	-5.53	-6.38	-8.5	-11.35	-11.44
20	0.00	-0.77	-0.51	-2.05	-4.6	-3.07	-3.07	-4.6	-4.86	-6.39

X4. CRITICAL VALUES OF *F* TABLE

X4.1 See Fig. X4.1.

		Degrees of Freedom for Numerator (df _N)											
		1	2	3	4	5	6	7	8	9	10	12	
Degrees of Freedom for Denominator (df _D)	1	4052.	4999.	5403.	5625.	5764.	5859.	5928.	5981.	6022.	6056.	6106.	
	2	98.50	99.00	99.17	99.25	99.30	99.33	99.36	99.37	99.39	99.40	99.42	
	3	34.12	30.82	29.46	28.71	28.24	27.91	27.67	27.49	27.34	27.23	27.05	
	4	21.20	18.00	16.69	15.98	15.52	15.21	14.98	14.80	14.65	14.54	14.37	
	5	16.26	13.27	12.06	11.39	10.97	10.67	10.46	10.29	10.16	10.05	9.89	
	6	13.74	10.92	9.78	9.15	8.75	8.47	8.26	8.10	7.98	7.87	7.72	
	7	12.25	9.55	8.45	7.85	7.46	7.19	6.99	6.84	6.72	6.62	6.47	
	8	11.26	8.65	7.59	7.01	6.63	6.37	6.18	6.03	5.91	5.81	5.67	
	9	10.56	8.02	6.99	6.42	6.06	5.80	5.61	5.47	5.35	5.26	5.11	
	10	10.04	7.56	6.55	5.99	5.64	5.39	5.20	5.06	4.94	4.85	4.71	
	11	9.65	7.21	6.22	5.67	5.32	5.07	4.89	4.74	4.63	4.54	4.40	
	12	9.33	6.93	5.95	5.41	5.06	4.82	4.64	4.50	4.39	4.30	4.16	
	13	9.07	6.70	5.74	5.20	4.86	4.62	4.44	4.30	4.19	4.10	3.96	
	14	8.86	6.51	5.56	5.04	4.69	4.46	4.28	4.14	4.03	3.94	3.80	
	15	8.68	6.36	5.42	4.89	4.56	4.32	4.14	4.00	3.89	3.80	3.67	
	16	8.53	6.23	5.29	4.77	4.44	4.20	4.03	3.89	3.78	3.69	3.55	
	17	8.40	6.11	5.18	4.67	4.34	4.10	3.93	3.79	3.68	3.59	3.46	
	18	8.28	6.01	5.09	4.58	4.25	4.01	3.84	3.71	3.60	3.51	3.37	
	19	8.18	5.93	5.01	4.50	4.17	3.94	3.77	3.63	3.52	3.43	3.30	
	20	8.10	5.85	4.94	4.43	4.10	3.87	3.70	3.56	3.46	3.37	3.23	
	21	8.02	5.78	4.87	4.37	4.04	3.81	3.64	3.51	3.40	3.31	3.17	
	22	7.95	5.72	4.82	4.31	3.99	3.76	3.59	3.45	3.35	3.26	3.12	
	23	7.88	5.66	4.76	4.26	3.94	3.71	3.54	3.41	3.30	3.21	3.07	
	24	7.82	5.61	4.72	4.22	3.90	3.67	3.50	3.36	3.26	3.17	3.03	
	25	7.77	5.57	4.68	4.18	3.85	3.63	3.46	3.32	3.22	3.13	2.99	
	26	7.72	5.53	4.64	4.14	3.82	3.59	3.42	3.29	3.18	3.09	2.96	
	27	7.68	5.49	4.60	4.11	3.78	3.56	3.39	3.26	3.15	3.06	2.93	
	28	7.64	5.45	4.57	4.07	3.75	3.53	3.36	3.23	3.12	3.03	2.90	
	29	7.60	5.42	4.54	4.04	3.73	3.50	3.33	3.20	3.09	3.00	2.87	
	30	7.56	5.39	4.51	4.02	3.70	3.47	3.30	3.17	3.07	2.98	2.84	
40	7.31	5.18	4.31	3.83	3.51	3.29	3.12	2.99	2.89	2.80	2.66		
60	7.08	4.98	4.13	3.65	3.34	3.12	2.95	2.82	2.72	2.63	2.50		
120	6.85	4.79	3.95	3.48	3.17	2.96	2.79	2.66	2.56	2.47	2.34		
∞	6.63	4.61	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32	2.18		

FIG. X4.1 Critical Values of F at 1 % Significance (99 % Confidence) Level (One-Sided)

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