

Designation: D6698 – 12

StandardTest Method for On-Line Measurement of Turbidity Below 5 NTU in Water¹

This standard is issued under the fixed designation D6698; 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 is applicable to the on-line measurement of turbidity under 5 nephelometric turbidity units (NTU) in water.

1.2 It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 In this test method calibration standards are defined in NTU values, but other assigned turbidity units are assumed to be equivalent.

1.4 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 NTU formazin standard is also equal to a 1 FNU (formazin nephelometric units) standard, a 1 FNRU (formazin nephelometric ratio units) standard, and so forth.

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

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

2. Referenced Documents

2.1 ASTM Standards:²

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on WaterD3370 Practices for Sampling Water from Closed Conduits

D3864 Guide for On-Line Monitoring Systems for Water Analysis

2.2 Other Standards:

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

GLI Method 2⁴

Hach Method 8195 Determination of Turbidity by Nephelometry EMMC Format⁴

ISO 7027 Determination of Turbidity⁵

Standard Method 2130B⁶

2.3 Other Documents:⁷

U.S. Patent 4,283,143 Patterson, James A. 1981. Optical Characterization of a Suspension. United States Patent 4,283,143, filed November 19, 1979, and issued August 11, 1981.

U.S. Patent 4,291,980 Patterson, James A. 1981. Styrene-Divinylbenzene Copolymer and Method of Manufacturer. United States Patent 4,291,980, filed August 14, 1978, and issued September 29, 1981.

U.S. Patent 5,777,011 Sadar, Michael J. 1998. Stabilized Formazin Composition. United States Patent 5,777,011, filed December 1, 1995, and issued July 7, 1998.

3. Terminology 3-dafe519442e2/astm-d6698-12

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

3.2 Definitions of Terms Specific to This Standard:

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

⁵ Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

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.

Current edition approved June 1, 2012. Published August 2013. Originally approved in 2001. Last previous edition approved in 2007 as D6698 – 07. DOI: 10.1520/D6698-12.

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, http:// www.epa.gov.

⁴ Available from Hach Company, P.O. Box 389, Loveland, CO, 80539-0389, http://www.hach.com.

⁶ Available from Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Public Health Association, Washington, DC, 2005, http://www.standardmethods.org.

⁷ Available from AMCO Clear, P.O. Box 245, Powell, OH, 43065, http:// www.amcoclear.com.

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

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

3.2.2.1 *Discussion*—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. Calibration verification standards may be instrument specific.

3.2.3 *in-situ nephelometer*, *n*—a turbidimeter that determines the turbidity of a sample using a sensor that is placed directly in the sample.

3.2.3.1 *Discussion*—This turbidimeter does not require transport of the sample to or from the sensor.

3.2.4 *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.

3.2.4.1 *Discussion*—Units are NTU (nephelometric turbidity units); when ISO 7027 technology is employed, units are FNU (formazin nephelometric units).

3.2.5 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.

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

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

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

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

3.2.8 *slip stream nephelometer*, *n*—an on-line technique for analysis of a sample as it flows through a measurement chamber of an instrument.

3.2.8.1 *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*.

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

3.2.9.1 *Discussion*—For example: ambient light leakage and divergent light in optical systems.

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

3.2.10.1 *Discussion*—The detected light is quantitatively converted to a numeric value that is traced to a light scatter standard.

3.2.11 *turbidity*, *n*—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.

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

4. Summary of Test Method

4.1 The optical property expressed as turbidity is measured by the scattering effect that suspended solids have on light; the higher the intensity of scattered light, the higher the turbidity. In samples containing particulate matter, the manner in which the particulate matter interacts 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 This test 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 degrees 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 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.

5.2 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.

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

NOTE 1—See 8.2 for discussion on signal spikes resulting from bubbles.

5.4 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.

5.4.1 Table 1 describes 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.

5.4.2 For a specific design that falls outside of the reporting ranges in Table 1, the turbidity should be reported in turbidity units (TU) with a subscripted wavelength value to characterize the light source that was used.

6. Safety

6.1 Wear appropriate personal protection equipment at all times.

6.2 Follow all relevant safety guidelines.



TABLE 1 Technologies and Reporting Resu	TABLE 1	Technologies	and Rep	ortina	Results
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Design and Reporting Unit	Prominent Application	Key Design Features	Typical Instrument Range	Suggested Application
Nephelometric non- ratio	White light turbidimeters	Detector centered at 90°	0.020 to 40	Regulatory reporting
(NTU)	comply with EPA 180.1	relative to the incident		of clean water
	for low-level	light beam. Uses a white		
Ratio White Light	turbidity monitoring. Complies with ISWTR	light spectral source. Used a white light	0.020 to 10 000	Regulatory Reporting
urbidimeters (NTRU)	regulations and Standard	spectral source. Primary	0.020 10 10 000	of clean water
	Method 2130B.	detector centered at 90°.		of clean water
	Can be used for	Other detectors located		
	both low and	at other angles.		
	high-level measurement.	An instrument algorithm		
		uses a combination		
		of detector readings		
		to generate the		
	0 1 10 2027	turbidity reading.	0.010 / 1000	0 10 100 7007
Nephelometric, near-IR	Complies with ISO 7027.	Detector centered at	0.012 to 1000	0–40 ISO 7027
turbidimeters, non-ratiometric (FNU)	The wavelength is less susceptible to	90° relative to the incident light beam.		Regulatory reporting
(FNO)	color interferences.	Uses a near-IR		
	Applicable for samples	(780-900 nm) monochromatic		
	with color and good	light source.		
	for low-level monitoring.	3		
Nephelometric near-IR	Complies with ISO 7027.	Uses a near-IR	0.012 to 10 000	0-40 ISO 7027
urbidimeters, ratio metric	Applicable for samples	monochromatic light source		Regulatory reporting
(FNRU)	with high levels	(780–900 nm).		
	of color and	Primary detector centered		
	for monitoring to	at 90°. Other detectors		
	high turbidity levels.	located at other angles.		
		An instrument algorithm uses a combination of		
		detector readings to		
		generate the turbidity		
		reading.		
Formazin Nephelometric Mul-	Is applicable to	Detectors are geometrically	0.012 to 4000	0–40 Reporting
ibeam	EPA regulatory method GLI	centered at 0° and 90°.		for EPA
Unit (FNMU)	Method 2.	Uses a near-IR light source		and ISO compliance
	Applicable to drinking	(780–900 nm)		
	water and wastewater	monochromatic light source.		
	monitoring applications.	An instrument algorithm		
		uses a combination of		
		detector readings, which		
		turbidities varying magnitude.		
mNTU	Is applicable to	Nephelometric method involving	5 to 5000 mNTU	0–5000 mNTU,
	reporting of clean	a laser-based light source		for EPA compliance
	waters and filter	at 660 nm and 1442 / 152		reporting on drinking
	performance monitoring.	a high sensitivity photo-multplier		water systems
	Very sensitive to	tube (PMT) detector		
	turbidity changes in	for light scattered		
	low turbidity samples.	at 90°.		
		1000 mNTU = 1 NTU		

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

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

7. Interferences

7.1 Bubbles, color, and large suspended particles may result in interferences. Bubbles cause positive interference and color causes negative interference. Dissolved material that imparts a color to the water may cause errors in pure photoelectric nephelometric readings (versus ratio photoelectric nephelometric readings) unless the instrument has special compensating features. Certain turbulent motions also create unstable reading conditions of nephelometers. 7.2 Scratches, finger marks, or dirt on any part of an optical component through which light must travel to reach the sample, or through which scattered light leaves the sample to a detector, may give erroneous readings. Keep these surfaces scrupulously clean and replace damaged (etched or scratched) components.

8. Apparatus

8.1 The sensor used for the on-line monitoring of turbidity is designed for continuous monitoring of the turbidity of the sample stream.

8.2 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, or electronic rejection methods, or a combination thereof.

8.3 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.

8.4 The resolution of the instrument should permit detection of turbidity differences of 0.01 NTU or less in waters having turbidities of less than 1.00 NTU. The instrument should permit detection of turbidity differences of 0.10 NTU or less in waters with turbidity between 1.0 and 5.0 NTU.

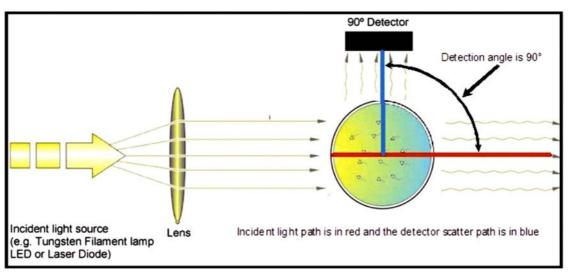
8.5 *Instrument Types*—Two types of instruments are available for the nephelometric turbidity method, the nephelometer and ratio nephelometer.

8.5.1 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 so designed 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 output. The total distance traversed by incident light and scattered light within the sample is not to 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.

8.5.1.1 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. To minimize initial differences, observe the following design criteria:

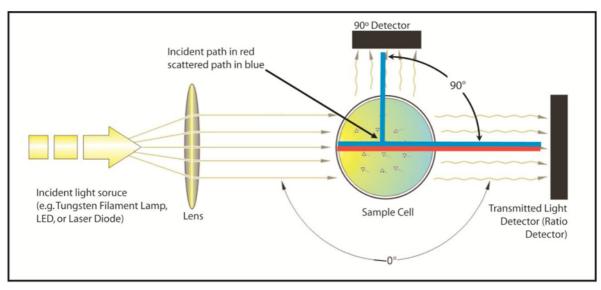
8.5.2 Ratio Photoelectric Nephelometer-(See Fig. 2 for single beam design; see Fig. 3 for multiple beam design.) This instrument uses the measurement derived through the use of a nephelometric detector that serves as the primary detector and one or more other detectors used to compensate for variation 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, or sample color, or combination thereof. The ratio photoelectric nephelometer should be so designed that minimal stray light reaches the detector(s), and should be free from significant drift after a short warm-up period. The light source should be a tungsten lamp, operated at a color temperature between 2200 and 3000 K. 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 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 scalable reading is from the nephelometric detector(s), and other detectors are used to compensate for instrument variation described in 3.2.5.

1(8.5.2.1) 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

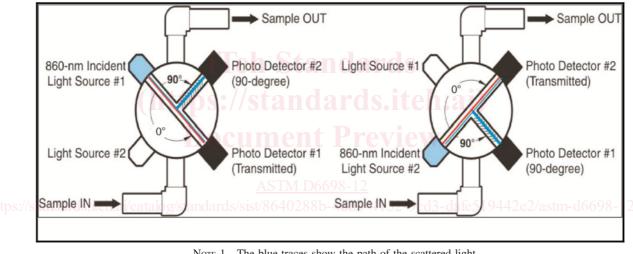


Note 1—The blue traces show the path of the scattered light. FIG. 1 Photoelectric Nephelometer

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Note 1—The blue traces show the path of the scattered light. FIG. 2 Ratio Nephelometer (Single Beam Design)



Note 1—The blue traces show the path of the scattered light. **FIG. 3 Multiple Beam Design**

calibrations. Comparability of measurements made using instruments differing in optical and physical design is not recommended. Examples of ratio nephelometers are shown in Figs. 2 and 3.

8.6 Examples of applicable nephelometers include: photoelectric nephelometer, ratio photoelectric nephelometer with a single beam design, and ratio photoelectric nephelometer in the dual beam design. In these designs, the correlation between detector response and increasing turbidity is positive.

9. Purity of Reagents

9.1 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.

9.2 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 1 hour of use to reduce background turbidity. Reverse osmosis (RO) water is acceptable and preferred in this test method. (See Specification D1193.)

10. Reagents

- 10.1 Reagent, dilution, and final rinsing water, see 9.2.
- 10.2 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 Section 10 is required in order to accurately prepare low-level turbidity standards.

10.2.1 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 9.2. Reference formazin turbidity standard (4000 NTU) is synthesized on the bench.

10.2.1.1 Dissolve 5.000 grams of ACS grade hydrazine sulfate (99.5 % + purity) ($N_2H_4 \cdot H_2SO_4$ into approximately 400 mL of dilution water (see 9.2) contained in a 1-litre Class A volumetric flask.

10.2.1.2 Dissolve 50.000 grams of ACS grade hexamethylenetetramine (99 %+ purity) in approximately 400 mL of dilution water (see 9.2) contained in another flask. Filter this solution through a 0.2-mm filter.

10.2.1.3 Quantitatively pour the filtered hexamethylenetetramine solution into the flask containing the hydrazine sulfate. Dilute this mixture to 1 litre using dilution water (see 9.2). Stopper and mix for at least 5 minutes, and no more than 10 minutes.

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

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

10.2.2 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. (See Hach Method 8195 and U.S. Patent 5,777,011.)

10.2.3 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. (See U.S. Patents 4,283,14 and 4,291,980.)

10.2.4 Formazin Turbidity Suspension, Standard (40 NTU)—All labware shall be seasoned (see Appendix X2). Invert 4000 NTU stock suspension 25 times to mix (1 second inversion cycle); immediately pipette, using a Class A pipette, 10.00 mL of mixed 4000 NTU stock into a 1000-mL Class A volumetric flask and dilute with water to mark. The turbidity of this suspension is defined as 40 NTU. This 40-NTU suspension must be prepared weekly.

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

10.2.5 *Dilute Formazin Turbidity Suspension Standard (1.0 NTU)*—Prepare this standard dilution daily by inverting the 40

NTU stock suspension 25 times to mix (1 second inversion cycle) and immediately pipetting a volume of the 40.0 NTU standard (10.2.4). 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.

10.2.5.1 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.

10.2.5.2 Using the cleaned glassware, pipette 5.00 mL of mixed 40.0 NTU formazin suspension (10.2.4) into the 200 mL flask and dilute to volume with the dilution water. Stopper and invert 25 times to mix (1 second inversion cycle). The turbidity of this prepared standard is 1.0 NTU.

10.2.6 *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 10.2.5.1 and prepare each dilution by pipetting the volume of 40 NTU (10.2.4) into a 100-mL volumetric flask and diluting to mark with dilution water (9.2). 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.

11. Instrument Installation, Sample Lines, and Sampling

Note 6—In principle there are two ways for on-line measurement set ups. The bypass (slip-stream) sample technique and the in-line measurement. For the bypass sample technique a portion of sample is transported out of the process into the measurement apparatus and is then either transported back to the process or to waste. For the in-line measurement the sensor is brought directly into the process (see Fig. 5).

11.1 Bypass Sample Technique:

11.1.1 *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.

11.1.1.1 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.

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

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

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

11.1.2.1 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.