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## DETERMINATION OF TURBIDITY BY LED NEPHELOMETRY

Mitchell Method M5331

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Revision 1.1

March 5, 2009

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## Mitchell Method M5331

### 1.0 SCOPE AND APPLICATION:

- 1.1 This method covers the determination of turbidity in drinking, ground, surface and any colorless water with a turbidity of less than 40 NTU.
- 1.2 The applicable range is 0 to 40 NTU.
- 1.3 This method can be used for compliance monitoring and reporting under the Safe Drinking Water Act (SDWA).

### 2.0 SUMMARY OF METHOD:

- 2.1 The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions, with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the the higher the turbidity. Readings in NTUs are made using a laser nephelometer designed according to specifications given in section 6.1 and 6.2. Primary standard suspensions are used to calibrate the instrument. A secondary standard is monitored periodically for deterioration using one of the primary standards.
  - 2.1.1 Any approved primary standard may be used in conjunction with this method. These standards include formazin polymer, stabilized formazin polymer and styrene divinylbenzene polymer suspensions.

### 3.0 DEFINITIONS

- 3.1 CALIBRATION BLANK (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.
- 3.2 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) – A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.
- 3.3 LABORATORY REAGENT BLANK (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the

laboratory environment, the reagents, or the apparatus.

- 3.4 LINEAR CALIBRATION RANGE (LCR) – The concentration range over which the instrument response is linear.
- 3.5 MATERIAL SAFETY DATA SHEET (MSDS) – Written information provided by vendors concerning a chemical/s toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
- 3.6 PRIMARY CALIBRATION STANDARD (PCAL) – A suspension prepared from the primary dilution stock standard suspension. The PCAL suspensions are used to calibrate the instrument response with respect to analyte concentration.
- 3.7 QUALITY CONTROL SAMPLE (QCS) - A solution of the method analyte of known concentrations that is used to fortify an aliquot of LBR matrix. The QCS is obtained from a source external to the laboratory, and is used to check laboratory performance.
- 3.8 SECONDARY CALIBRATION STANDARDS (SCAL) – commercially prepared, stabilized sealed liquid or gel turbidity standards, or other apparatus or mechanism calibrated against properly prepared and diluted Formazin or styrene divinylbenzene polymers.
- 3.9 STOCK STANDARD SUSPENSION (SSS) – A concentrated suspension containing the analytic solution prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source. Stock standard suspension is used to prepare calibration suspensions or other needed suspensions.

#### 4.0 INTERFERENCES:

- 4.1 The presence of floating debris and coarse sediments that settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.
- 4.2 The presence of true color, that is the color of water that is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking water.
- 4.3 Light-absorbing materials such as activated carbon in significant concentrations can cause low readings.

#### 5.0 SAFETY:

- 5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable.

- 5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.
- 5.3 Hydrazine sulfate (7.2.1) is highly toxic and may be fatal if inhaled, swallowed or absorbed through the skin. Formazin can contain residual hydrazine sulfate. Proper protection should be employed.

## 6.0 EQUIPMENT AND SUPPLIES:

- 6.1 The turbidimeter shall consist of a nephelometer, with a light source for illuminating the sample, and one or more photo-electric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. The turbidimeter should be designed so that little stray light reaches the detector in the absence of turbidity and should be free from significant drift after a short warm-up period.
- 6.2 Differences in physical design of turbidimeters will cause differences in measured values for turbidity, even though the same suspension is used for calibration. To minimize such differences, the following design criteria should be observed:
  - 6.2.1 LED light source: Monochromatic source operated at a nominal wavelength of  $525 \pm 15\text{nm}$ . The light source shall be used as a directly received reference for the scattered nephelometric signal.
  - 6.2.2 There shall be no divergence from parallelism as the incident radiation and any convergence shall not exceed  $\pm 1 \frac{1}{2}$  degrees in the measurement area.
  - 6.2.3 Distance traversed by incident light and scattered light not to exceed 10 cm.
  - 6.2.4 Detector/Light Receiver: Centered at  $90^\circ \pm 1 \frac{1}{2}$  degrees to the incident light path and the light cone not to exceed  $\pm 30$  degrees from 90 degrees. The receiver, if used, may be coupled to a photo detector using an optical path.
    - 6.2.4.1 The photo detector must have a spectral response that encompasses complete spectral output of the light source. A Mitchell/Mersch light trap shall be incorporated.
  - 6.2.5 An optical device may be used to carry light from the light source to the sample or to carry scattered light from the sample to the photo detector.

- 6.2.6 Instrument shall have incorporated a Mitchell/Mersch bubble trap for on-line use and Mitchell/Mersch anti-fog windows. Sensor shall be horizontal with vertical windows. Both windows shall be immersed in the measurement fluid for on-line use. Instrument shall be capable of being pressurized to 30 psig.
- 6.2.7 Turbidimeter example meeting these specifications is the Mitchell M5331.
- 6.3 The sensitivity of the instrument should permit detection of a turbidity difference of .001 NTU or less in waters having turbidities less than 1 NTU units. The instrument should measure from 0 to 40 NTU turbidity units.
- 6.4 Balance – Analytical, capable of accurately weighing to the nearest 0.0001g.
- 6.5 Glassware – Class A volumetric flasks and pipettes as required.

## 7.0 REAGENTS AND STANDARDS

- 7.1 Reagent water, turbidity-free: Pass deionized distilled water through a 0.45 micron pore-size membrane filter if necessary. Water should have a turbidity  $\leq 0.03$  NTU. This value should be considered when preparing standards.
- 7.2 Stock standard suspension (Formazin)
  - 7.2.1 Dissolve 1.00g hydrazine sulfate (CASRN 10034-93-2) in reagent water and dilute to 100 mL in a volumetric flask.
  - 7.2.2 Dissolve 10.00g hexamethylenetetramine (CASRN 100-97-0) in reagent water and dilute to 100 mL in a volumetric flask. In a 100 mL volumetric flask, mix 5.0 mL of each solution (7.2.1 + 7.2.2). Allow to stand 24 hours at  $25 \pm 3$  degree C, then dilute to the mark with reagent water.
- 7.3 Primary calibration standards: Mix and dilute 10.00 mL of stock standard suspension (7.2) to 100 mL with reagent water. The turbidity of this suspension is defined as 40 NTU. For other values, mix and dilute portions of this suspension as required.
  - 7.3.1 A new stock standard suspension (7.2) should be prepared each month. Primary calibration standards (7.3) should be prepared daily by dilution of the stock standard suspension.
- 7.4 Formazin in commercially prepared, certified, concentrated stock standard suspension (SSS) may be diluted and used as required. Dilute turbidity standards should be prepared daily.

- 7.5 Styrene divinylbenzene polymer in commercially prepared, certified, concentrated stock or ready to use dilutions. Manufacturer's instructions should be followed for choosing the appropriate standard for the instrument.
- 7.6 Secondary standards may be acceptable as a calibration check, but must be monitored on a routine basis for deterioration and replaced as required.

## 8.0 SAMPLE COLLECTION AND INSTRUMENT SETUP

- 8.1 Online instrumentation does not require sample cooling, preservation or storage.
- 8.2 Install and set up the instrument according to the manufacturers instructions.

## 9.0 QUALITY CONTROL

- 9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and analysis of laboratory reagent blanks and other solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated.
- 9.2 INITIAL DEMONSTRATION OF PERFORMANCE
  - 9.2.1 The initial demonstration of performance is used to characterize instrument performance (determined of LCRs and analysis of QCS).
  - 9.2.2 Linear Calibration Range (LCR) – The LCR must be determined initially and verified whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by  $\pm 10\%$ , linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.
  - 9.2.3 Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analysis of a QCS. If the determined concentrations are not within  $\pm 10\%$  of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

## 10.0 CALIBRATION AND STANDARDIZATION

10.1 Turbidity Calibration: The manufacturer's operating instructions should be followed. Measure standards on the turbidimeter covering the range of interest. If the instrument is already calibrated in standard turbidity units, this procedure will check the accuracy of the calibration scales. At least one standard should be run in each instrument range to be used. Some instruments permit adjustments of sensitivity so that scale values will correspond to turbidities. Solid standards, if used, must be protected from surface scratches which may cause potential calibration changes. If a pre-calibrated scale is not supplied, calibration curves should be prepared for each range of the instrument. Calibration must be performed under identical optical conditions as operational conditions. The unit may optionally incorporate a Mitchell/Mersch self-check system for a secondary verification of instrument calibration.

## 11.0 PROCEDURE:

11.1 For on-line use, all instrument parameters should be set to insure constant sample flow is fed to the instrument. This will insure bubble interferences are minimized. Refer to the manufacturer's instrument manual for the set up of measurement parameters and flow rates.

## 12.0 DATA ANALYSIS AND CALCULATIONS:

12.1 Report Results as follows:

NTU	Record to Nearest
0.0 -1.0	.01
1 – 10	0.1
10 – 40	1.
40 – 100	5.
100 – 400	10.
400 – 1,000	50.
≥1,000	100.

## 13.0 METHOD PERFORMANCE:

The following data was collected in using styrene divinylbenzene polymer standards (n=10 for 0.10, 0.30, 0.50 and n=5 for 5.0, 10, 20 and 40 NTU levels):

Theoretical (NTU)	Average (NTU)	Standard Deviation	% RSD	Average % Recovery
0.10	0.100	0.0006	0.6	100
0.30	0.294	0.007	0.2	98
0.50	0.506	0.007	0.1	101
5.0	5.01	0.008	0.1	100
10	9.97	0.005	0.1	100
20	20.0	0.09	0.4	100
40	40.0	0.2	0.4	100

#### 14.0 POLLUTION PREVENTION:

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste as the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution-prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.
- 14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

#### 15.0 WASTE MANAGEMENT:

- 15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect air, water, and land by minimizing and controlling all releases from hoods and bench operations; complying with the letter and spirit of any waste discharge permit and regulations; and by complying with all solid hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

#### 16.0 REFERENCES:

- 16.1 *Annual Book of ASTM Standards*, Volume 11.01 Water (1), Standard D1889-88A, p. 359, (1993).
- 16.2 *Standard Methods for the Examination of Water and Wastewater*, 18<sup>th</sup> Edition, pp.2-9, Method 2130B (1992). APHA, AWWA, and WEF. Port City Press, Baltimore, MD.
- 16.3 McKee, Gerald F., (1985); *Turbidity Standard*, EPA Record.