6.7.2 CALIBRATION

Follow the manufacturer's instructions for instrument calibration and record calibration readings and adjustments in the instrument log book.

- Calibration of turbidity instruments against a Formazin or other approved primary standard usually is done in the laboratory, with instrument checks performed in the field. **Use standards that bracket the range of turbidity anticipated in environmental samples, if possible.**
- For instruments that are factory calibrated in standard turbidity units, the calibration procedure checks the accuracy of calibration scales provided by the manufacturer.
- Periodically check the accuracy and precision of your instrument against that of another instrument.
- Consult the manufacturer if the precision of your instrument deviates 5 percent or more from the manufacturer's specifications.

The USEPA specifies that the turbidimeter must be calibrated with a primary standard (a Formazin or a styrene divinylbenzine polymer standard such as Amco AEPA-1 Polymer™) (U.S. Environmental Protection Agency, 1994). A solid scattering standard provided by the manufacturer for setting overall instrument sensitivity for all ranges should not be relied on unless the turbidimeter is demonstrated to be free of drift on all ranges (U.S. Environmental Protection Agency, 1979).

Temperature changes affect Formazin turbidity standards and the performance of the turbidity instrument.

- Turbidity instruments are not currently available with an automatic temperature-compensating function.
- Standards and instruments should be at the same and constant temperature during calibration to achieve stable and accurate results.
- To avoid the effects of thermal fluctuations on the calibration, perform the Formazin calibration and calibration of the secondary standard (for example, Gelex™) against the primary standard in the office laboratory at room temperature instead of at the field site. At the field site, check instrument calibration using a secondary standard.

Preparation of the stock turbidity suspension and standard dilutions

Prepare the stock turbidity suspension monthly and standard dilutions on the day of instrument calibration. To prepare and dilute a 400 NTU Formazin stock suspension[4]:

1. Dissolve 1.000 g hydrazine sulfate [$(NH_2)_2 \cdot H_2SO_4$] in filtered water and dilute to 100 mL in a volumetric flask.

2. Dissolve 10.00 g hexamethylenetetramine [$(CH_2)_6N_4$] in filtered water and dilute to 100 mL in a volumetric flask.

3. Mix 5.0 mL of hydrazine sulfate and 5.0 mL of hexamethylenetetramine solutions in a 100-mL volumetric flask and let stand 24 hours at 25 ± 3°C; dilute to the mark and mix. To prepare 500 mL of 400 NTU standard, mix 25 mL of the reagent solutions in a 500-mL flask, dilute to the mark, and mix.

4. For a 40 NTU standard, dilute 10.00 mL of the 400 NTU stock suspension to 100 mL with turbidity-free water (sample or deionized water passed through a filter media of ≤ 0.2 µm).
   - Dilute stock suspension on the day the standard is needed, use it immediately after preparation, and discard unused standard.
   - Inconsistent techniques used to dilute standards can add as much as 5 percent measurement error.

TURBIDIMETER CALIBRATION 6.7.2.A

The calibration instructions and procedures that follow are general and should be modified to apply to the instrument being used—check manufacturer's instructions:

1. Prepare Formazin suspensions as described above.
   - Calibrate each instrument range using at least three standard concentrations. Use standards that bracket the range of turbidity anticipated in the sample solution.
   - Prepare dilute standards fresh from the stock at the time of use—after dilution, the stock suspension is stable only for 4 to 6 hours.
   - For turbidity greater than 40 NTU, use undiluted stock solution.
   - **Do not use standards with flocculated suspensions.**

[4] Refer to American Public Health Association and others (1992) for detailed instructions.

2. Switch the turbidimeter on and allow it to warm up. Put on disposable gloves.

3. Check instrument focus: insert template in the cell holder. The lamp image should just fill the inside circle. Adjustment is required if the image is off center, too large, or too small.

4. Field rinse a clean, dry, scratch-free, index-marked cell with the highest concentration of the standard for the instrument range setting or range of interest.
   a. Hold the sample cell by the rim (top lip), not beneath the lip.
   b. Pour standard into the sample cell to the fill mark.
   c. Wipe the exterior of the cell using a soft, lint-free cloth or tissue to remove moisture (condensation) on cell walls.
   d. Apply a thin layer of silicon oil (table 6.7–1) onto the exterior of the cell to reduce condensation on the cell and mask slight scratches and nicks. Apply silicon oil uniformly onto the blank cell if it will be used on the cell filled with standard (check manufacturer's recommendations).

5. Select the desired NTU range.
   • Set the calibration adjustment to equal the high value of standard for the range of interest.
   • Before inserting the standard, ensure that no air bubbles are present.

6. Orient the standard cell in the cell holder—the calibration cell and sample cell must have identical orientation when in the instrument measurement chamber.

7. In the instrument log book, record and graph the instrument value for each standard (instrument reading versus standard value—see fig. 6.7–1).

8. Adjust standardization control until the value on the meter equals the NTU value of the standard used.

9. Remove the sample cell and discard the first turbidity standard.
   a. Rinse and fill a clean cell with the second turbidity standard and orient the cell in the instrument.
   b. Take a reading without adjusting the calibration.
   c. Plot this instrument NTU reading against the NTU value of the turbidity standard (fig. 6.7–1).

10. Repeat step (9) for at least one more turbidity standard with NTU value to cover the turbidity range of interest. The greater the number of turbidity standard values used, the greater the reliability of the calibration.

11. Prepare a calibration curve for each range of values to be used if a precalibrated scale is not supplied by the manufacturer. (The accuracy of calibration scales provided with the instrument must be verified by using a precalibrated instrument and appropriate standards.)
    - The plot of instrument reading versus turbidity standard value is a range calibration curve.
    - Verify that any instrument reading (dial setting) within the range calibrated is correct and agrees with correlative points on the calibration curve.

12. Calculate the NTU of a diluted sample:

$$NTU = A \times (B+C) / C$$

where
A = NTU found in diluted sample,
B = volume of dilution water, in milliliters, and
C = sample volume taken for dilution, in milliliters.

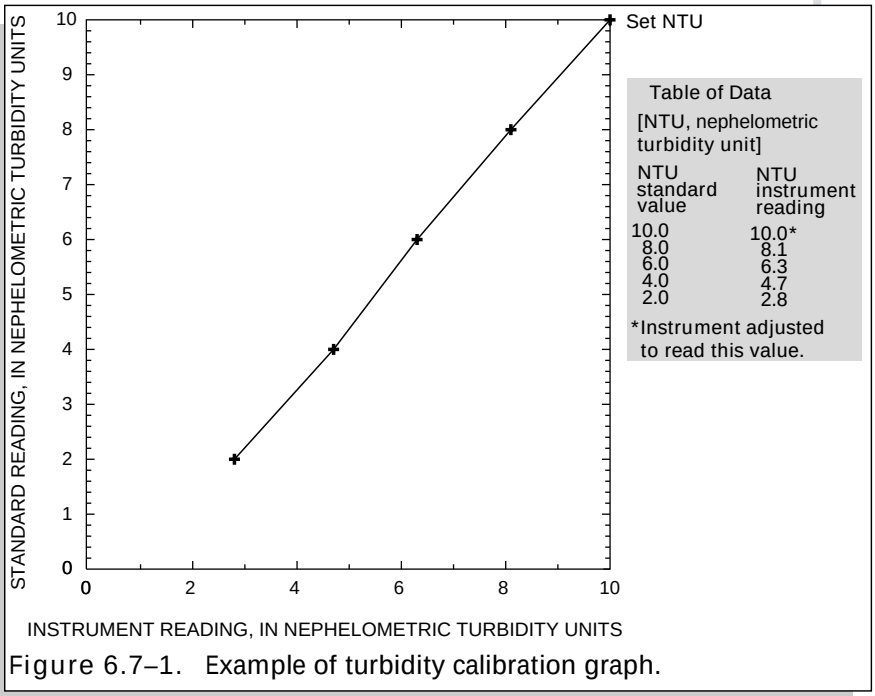

Figure 6.7–1. Example of turbidity calibration graph.

6.7.2.B SUBMERSIBLE TURBIDITY SENSOR CALIBRATION

Most multiparameter instruments with turbidity probe capability are microprocessor-based, with the calibration parameters stored in instrument memory. Turbidity values of the standards are user-selectable in some instruments, but some instruments have internally established standard values that cannot be changed. Low-level check standards in the 1–5 NTU range will allow the user to assess the actual performance of the instrument near the detection limit; **instrument reliability generally decreases at NTU less than 5**—consult manufacturer's specification for the expected accuracy of the measurement.

Monitor digital output carefully to assure that turbidity readings are stable before confirming the calibration. Note that if the instrument uses signal averaging to smooth instrument output, output response to changes in turbidity readings can be slowed.

Calibrate the instrument before leaving for the field site. While in the field, check instrument performance periodically using turbidity standard and turbidity-free water. The optical surface of the probe must be clean before beginning the calibration procedure. Modify the general instructions that follow as necessary so that they are compatible with the manufacturer's instructions:

1. Prepare a sufficient volume of the Formazin standard, as described previously. Volume of standard required could be 500 mL for some instruments, particularly if the entire sonde bundle instead of just the turbidity probe will be immersed.

2. **Select Procedure (A) or (B).** The same procedure, once tested and selected, also should be used in future studies.

   Procedure A. Immersion of entire sonde (bundle of field-measurement sensors, including the turbidity sensor)—requires larger volumes of standard; standard is vulnerable to contamination and dilution. The sonde sensor guard may need to be removed.

   Procedure B. Immersion of turbidity probe only—depending on sonde configuration, isolation of the turbidity probe and achieving a bubble-free optical surface could be difficult. This technique minimizes the volume of standard required for calibration.

3. Using a zero NTU standard (turbidity-free water):
   a. Rinse sonde/probe with deionized water, followed by a portion of turbidity-free water.
   b. Immerse entire surface of sonde/probe in turbidity-free water.
   c. Agitate the sonde/probe repeatedly to remove bubbles from the optical surface (activate mechanical wiper, if present).
   d. Monitor turbidity readings for 1 to 2 minutes or longer to ensure that readings are stable (consult manufacturer's recommendations and signal-averaging information).
   e. Confirm the zero NTU calibration point using manufacturer's instructions.
   f. Remove sonde/probe and dry thoroughly to minimize dilution or contamination of the next standard.
   g. Discard first standard (turbidity-free water).

4. Using the second standard (Formazin suspension):
   a. Rinse sonde/probe surfaces with a portion of standard. Discard rinsate.
   b. Immerse entire surface of sonde/probe in a container filled with standard.
   c. Agitate the sonde/probe repeatedly to remove bubbles from the optical surface (activate mechanical wiper, if present).
   d. Monitor turbidity readings for 1 to 2 minutes or longer to ensure that readings are stable (consult manufacturer's recommendations and signal-averaging information).
   e. Confirm the NTU calibration point for the standard used, according to manufacturer's instructions.
   f. Remove sonde/probe and rinse surfaces thoroughly with deionized water followed by turbidity-free water. Dry sonde/probe thoroughly.
   g. Discard used standard.

5. Repeat steps 4(a–g) using a different Formazin suspension standard if increased accuracy is desired and instrument software permits use of a third calibration point.

6. By diluting the existing standards, prepare a standard with turbidity, either approximately midway between the calibration points and (or) close to the estimated turbidity of the water to be measured.
   a. Measure the turbidity of this suspension, making certain that it is within the accuracy specification of the instrument with regard to the true value.
   b. Repeat the calibration procedure if the measurement is not within the specification.

Once the instrument is calibrated, the accuracy of the recorded measurements can be increased by preparing a calibration graph using dilutions of the Formazin standards, as described previously for calibration of turbidimeters (6.7.2.A).

> TECHNICAL NOTE: Multiparameter instruments with turbidity-probe capability use a light-emitting diode in the range of near-infrared wavelength as the radiation source and usually are microprocessor-based. The USEPA has not approved instruments using this method as of this writing, and the accuracy attainable with probe-based instrumentation is substantially less than that of USEPA-approved instruments. ISO turbidity-measurement criteria were developed to improve measurement consistency of instruments using the near infrared technology, and some of the field instruments available meet ISO 7027 recommendations (table 6.7–2).

SPECTROPHOTOMETER CALIBRATION 6.7.2.C

Spectrophotometric turbidity measurements are useful to indicate relative values or to monitor changes in turbidity with time. Spectrophotometers are inaccurate for absolute turbidity values, and the instrument sensitivity is unrated.

Spectrophotometers commonly have a stored program for turbidity that has been factory-calibrated. This can be verified but not adjusted. Check the instrument output against that of a different instrument every few weeks while the instrument is in use. Check the relative accuracy of the turbidity measurement before leaving for the field by inserting Formazin standards covering the FTU range needed.

1. Use freshly prepared standards.
   - Be accurate in your dilution of the stock suspension.
   - Prepare standards daily and discard any unused portion after each use.
2. **Wear disposable powderless (vinyl or latex) gloves**—fingerprints or smudges on cuvettes cause false turbidity readings; oils from skin can etch the cuvette glass.
3. Hold the sample cell (cuvette) at the rim (on the top lip), not beneath the lip. Pour standard into sample cell to the fill line.
4. Wipe the exterior of the sample cell with a clean, soft, lint-free cloth or tissue after filling to remove moisture and condensation from cell walls.
   - Check periodically for condensation on the sample cell and wipe it dry.
   - After wiping condensation from cell walls, apply a light coating (two drops) of silicon oil (optical grade) using a lint-free cloth—check recommendations from the instrument manufacturer.
5. Eliminate gas bubbles from standards.
6. Check that the calibration cell and sample cell have the same orientation when placed into the instrument measurement chamber.