APPLICATION NOTE

CALIBRATION METHODS FOR LOW-LEVEL TURBIDIMETER MEASUREMENT

Introduction

The process of calibrating and subsequently verifying the calibration of turbidimeters for low-level measurement is very sensitive to user technique and the surrounding environment. As measured turbidity levels drop below 1.0 NTU, interferences caused by bubbles, particulate contamination, and stray light become major factors. To minimize these errors, many approaches can be taken. Two of the most common approaches for low-level calibration are one-point and two-point calibrations.

After identifying and addressing turbidity interferences, the calibration approach that is best suited for a specific low-level measurement can be identified. This article discusses possible errors and the resulting calibration approach Hach uses for its turbidimeters.

Interferences in Low Turbidity Measurement

Hach Company's approach to turbidimeter calibration has been consistent over several generations of turbidimeters. In turbidity science it is well known that the correlation between turbidity and nephelometric light scatter (light scattered by particles at an angle of 90 degrees to the incident light source) is highly linear over the range of 0.012 to 40 NTU. Because of this relationship, an approach can be taken that reduces calibration error while extending measurement accuracy to the lowest turbidity levels.

At very low turbidity levels, measurement errors become very significant. The major source of error is stray light. Stray light is defined as any light reaching the light scatter (90 degree) detector that is not scattered by the sample. Stray light is always a positive interference. Sources of stray light include dust contamination in the instrument optics, low quality or damaged sample cells and instrument electronics. The best way to reduce stray light is through the design of robust instruments with high-quality optics combined with good laboratory and measurement technique. Hach leads the market in the production of instruments with extremely low stray light levels.

Another significant error can be attributed to bubbles in the sample. Bubbles in most samples are easily eliminated by allowing the sample to stand for a short time—2 to 5 minutes—before measurement. For process instruments, bubble removal is best accomplished through the use of bubble traps. At low turbidity levels, a wait of 2-5 minutes will not result in particulate settling.

The third error source that must be considered arises through the preparation of the calibration standards. Calibration standards can be easily and accurately prepared at higher values (greater than 1 NTU) if close attention is paid to cleanliness, dilution water of high quality (low turbidity) is available, and good analytical technique is applied. By addressing these issues, an analyst can prepare standards to within 2 percent at turbidity levels down to 10 NTU and to within 3 percent at turbidity levels between 10 NTU and 1 NTU. Calibration standards below 1.0 NTU can be prepared if dilution water of the highest quality (ultra-filtered or reverse-osmosis) is used to prepare all standards and to clean sample chambers, cells, and glassware. Excellent laboratory technique is necessary to reduce any source of contamination. Unfortunately, high purity water is often difficult to obtain and even when all cleanliness issues are addressed, a 0.30 NTU standard typically has, at best, a 10 percent error.

Hach designs its instruments for higher-level calibration because the accuracy of a prepared 20 NTU standard is typically better than 2 percent. Preparation of standards below 1 NTU will result in much greater error mostly due to residual turbidity in the dilution water. Filtration will greatly lower this interference. If possible, use either distilled or deionized water that has been passed through an ultra-filtration process such as reverse-osmosis.

The One-point Calibration Algorithm

The calibration of Hach turbidimeters was designed to reduce or eliminate the interferences presented previously. The calibration algorithm is very simple and is based on two readings: a calibration standard and a zero point, which requires no standard. The basis for this algorithm approach is discussed below.

- The nephelometric detector response to turbidity is highly linear in the range of 0.012 to 40 NTU. This linearity allows a calibration to be performed using a single standard at any point in this range. For Hach turbidimeters, the 20 NTU calibration point is preferred due to ease of preparation and high accuracy. Even if the dilution water has some turbidity (less than 0.5 NTU), the error contributed to the standard is low. In addition, errors due to instrument stray light are negligible at 20 NTU. Since a calibration performed at 20 NTU has a very low error, theoretically, measurement error will also be low when a turbidimeter is calibrated at this level. If the sum of the errors between the standard and the instrument equal 2 percent at 20 NTU, the same accuracy can be extrapolated to low-level measurements.

- The zero point is performed with the instrument light source turned off, but with all other measurement parameters in place. During this measurement, any residual light in the optics or environment that could result in interference is effectively removed from all subsequent measurements. This is often referred to as the blank or dark values. Typically, the zero point is determined when the instrument power is cycled.

- After the zero measurement and standard measurement have been taken, the instrument algorithm draws a straight line between these two points, constructing the linear calibration curve. The only remaining error is from stray light contributed by the instrument lamp. This error becomes a factor at the lowest measurements, typically below 0.05 NTU.

- Most Hach turbidimeters require the measurement of the dilution water (DW) so the turbidity contribution of the dilution water used to prepare the high-end calibration standard(s) can be compensated. Once measured, the value of the DW standard is stored in the turbidimeter's software. The value is then subtracted from the measured value of the other calibration standards. By subtracting this value from the measured value of the calibration standards, the absolute value of each calibration standard is calculated and the accuracy of the high-end calibration points is maximized.

The use of the one-point calibration algorithm deals with all the interferences noted above except for stray light observed when the instrument light source is on. This interference is mediated by designing the instrument to produce high collimated light and eliminate light exposure to all surfaces inside the instrument optics except the sample cell. This is exactly what Hach does. With high quality components coupled with strict design standards, Hach turbidimeters are able to bring incident lamp stray light to very low levels (<0.025 NTU). It is only at the lowest turbidity levels that a minute portion of stray light may be observed and it is always a positive interference.

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