FIAlab®
Leaders in Flow Injection Technology

Early Detection of Issues:
Save Time and Headache by Following These Easy Steps

Any flow analyzer using absorbance-based measurements is susceptible to contamination and other chemical issues. Of course, if proper lab protocol is followed, these are very rare occurrences. However, it is very important to routinely check for these types issues in order to address them in a timely manner. This diligence can mean the difference between 3 hours vs. 30 minutes of time spent. Early detection of issues will save time and headache. With this in mind, we have compiled a list of simple checks to catch any issues before an entire sample rack must be rerun.

Check 1: Calibration Verification

Checking that the calibration curve yields a linear fit of the data is the first consideration. Remember that the R^2 value must be as close to 1 as possible. One commonly used cutoff point is 0.995 or greater. If a calibration point is off of the line, fix it! Ignoring the calibration point may yield a better R^2 value, but may be masking a larger issue. Re-pouring the calibration standard is a quick way to determine whether the standard was off, or if further investigation is necessary.

Check 2: Sample Contamination in the Blank

It is easy to quickly “write off” blank response as carry over from a previous run. If this is thought to be the case, the operator is better off rinsing the instrument and rerunning the blank. Neglecting blank response may be neglecting contamination, especially if the calibration curve with the blank is linear. This positively biased calibration curve not only yields inaccurate results, it can yield negative results. Consider the following calibration run:

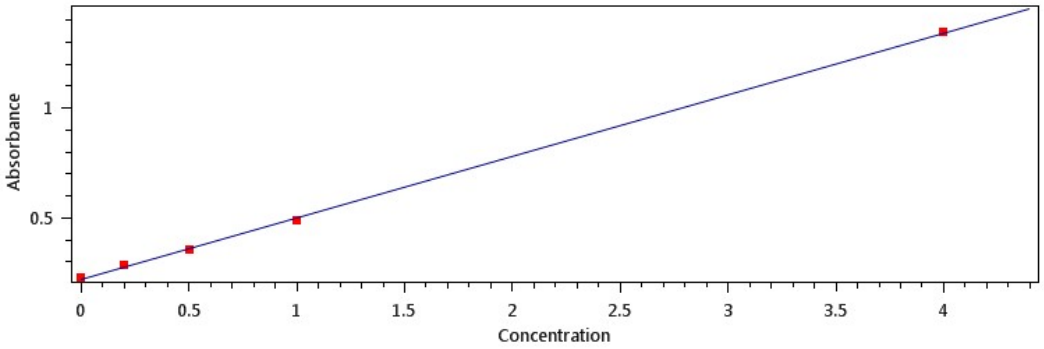

Figure 1: Calibration Run

The calibration curve appears to be very linear, no doubt passing the R^2 cutoff of 0.995. However, upon inspection of the data, the extreme positive bias of the standards puts them much higher in response than the data. This in turn yields negative results.

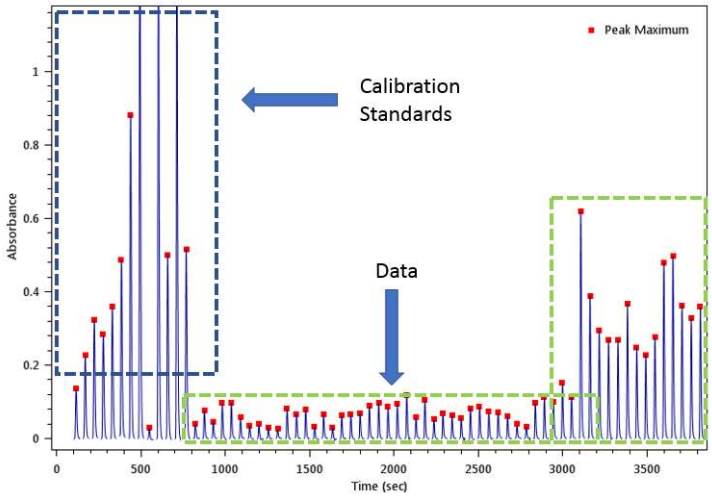

Figure 2: Positively Biased Cal Curve

You can see from the figures that a nice calibration curve does not always ensure quality data. It becomes clear when comparing the responses of the data to the calibration standards. Many of the responses are less than the blank. What's more is that these data points have a discernable response.

Check 3: Drastic Changes in the Absorbance Scale

Always check to see that the absorbance scale is typical. If the response of the calibration curve is much less than before, there could be an issue. For example, in nitrate measurements, if the response starts to decrease, it is likely that the cadmium column is starting to foul, yielding a lower reduction efficiency. At a low enough efficiency, the signal to noise becomes less and less favorable to the point where the peak cannot be differentiated. A lower absorbance scale means less sensitivity at the low end. A snapshot of this occurrence can be seen below:

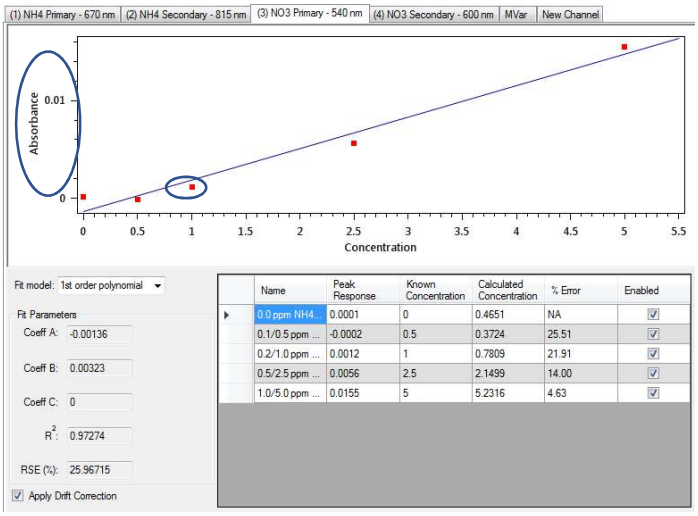

Figure 3: Low Absorbance Calibration

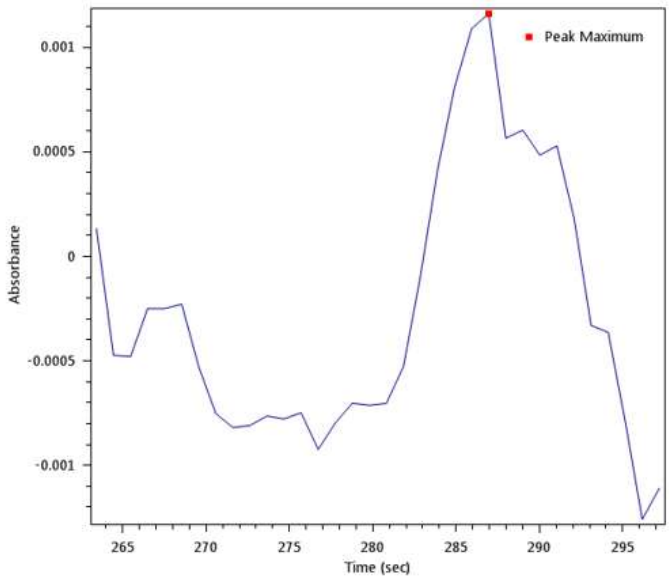

Figure 4: 1 ppm Injection

The calibration curve in Figure 3 is clearly unfavorable, since the points do not fall on the line. The reason becomes much clearer when viewing the circled calibration point's absorbance vs time in Figure 4. There you can see that the response is jagged and close to becoming indistinguishable as a peak. The absorbance scale in the calibration curve ties it all together. Make sure the absorbance scale is as expected. If not, solving the problem will ensure you produce quality data.

As stated above, these issues are rare. However, these checks are quick to do and can save a whole sample rack worth of analysis time if there was a problem that went undetected. Checking R^2, positive bias, and the absorbance scale helps ensure everything is running properly. If issues like this persist, call FIAlab. We have a lot of experience with common customer issues and are efficient at diagnosing and solving the problem.